

# Resistive Detectors with Diamond-Like Carbon(DLC)

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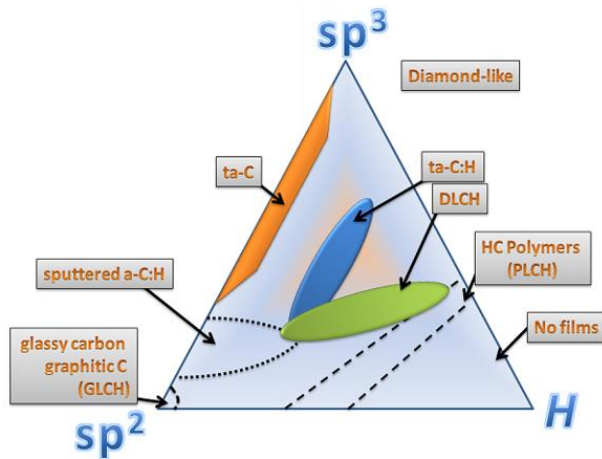
The 10th Symposium on Large TPCs for Low-Energy Rare Events Detection  
17-12-2021

# Outline

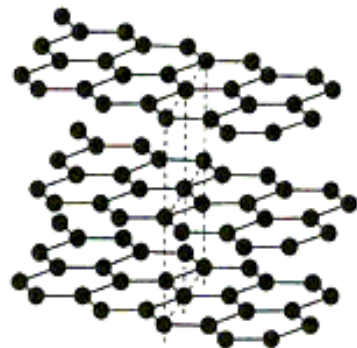
- 1. Introduction of the DLC**
2. Applications of resistive DLC in gaseous detectors
3. Summary

# Introduction of DLC

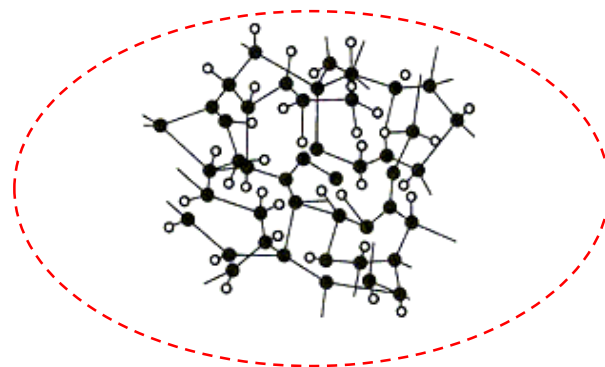
DLC is a type of amorphous carbon which contains both graphite structure ( $sp^2$ ) and diamond structure ( $sp^3$ )



- ✓ *Robust and stable, excellent chemical and physical inertness*
- ✓ *Sub-micrometer thickness, easy to make fine resistive structures;*
- ✓ *Surface resistivity can be precisely adjusted by changing the  $sp^2/sp^3$  ratio or element doping;*
- ✓ *Allow to make precise routes by using photolithography*
- ✓ *Easily extend to large area;*
- ✓ *.....*



Graphite structure

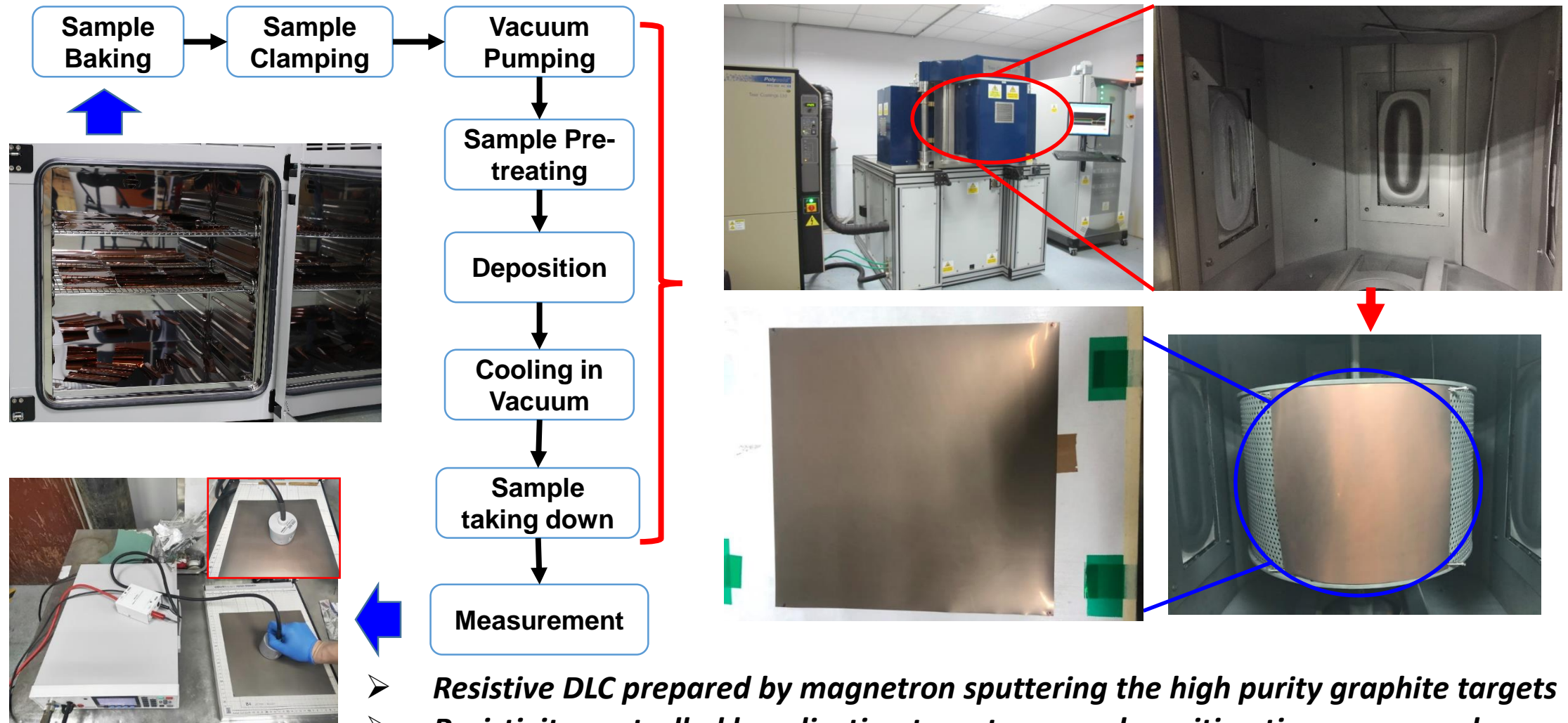


DLC structure



Diamond structure

# Preparation of DLC by Magnetron Sputtering Method



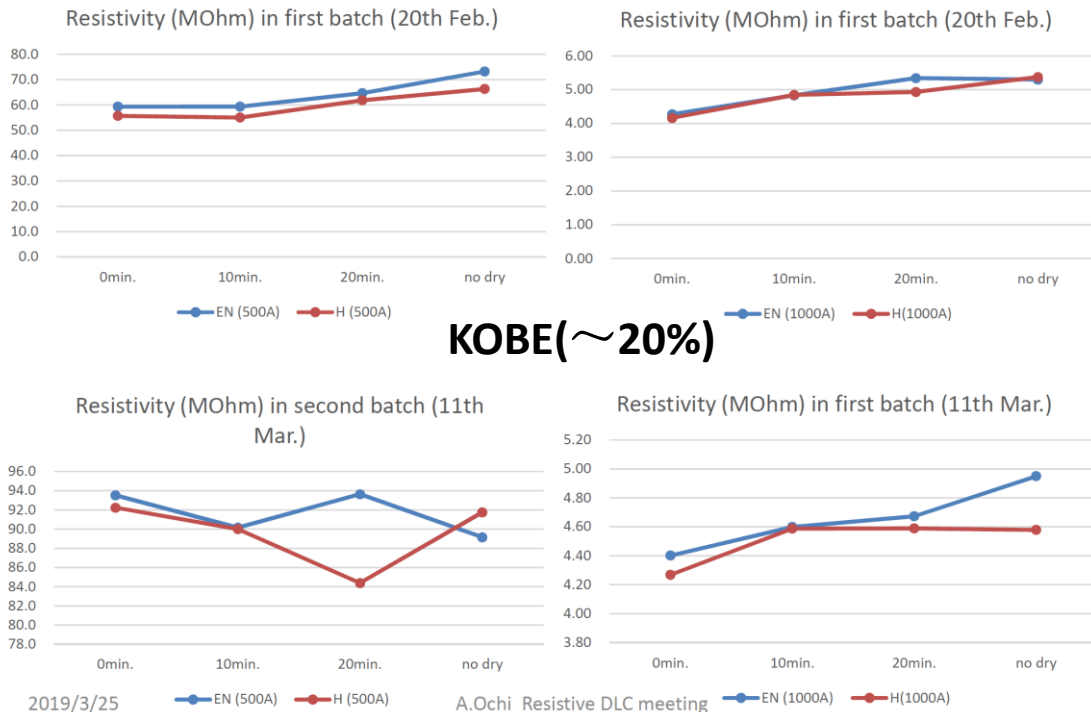
- *Resistive DLC prepared by magnetron sputtering the high purity graphite targets*
- *Resistivity controlled by adjusting target power, deposition time, vacuum degree, and so on.*

# Passivation in air after sputtering

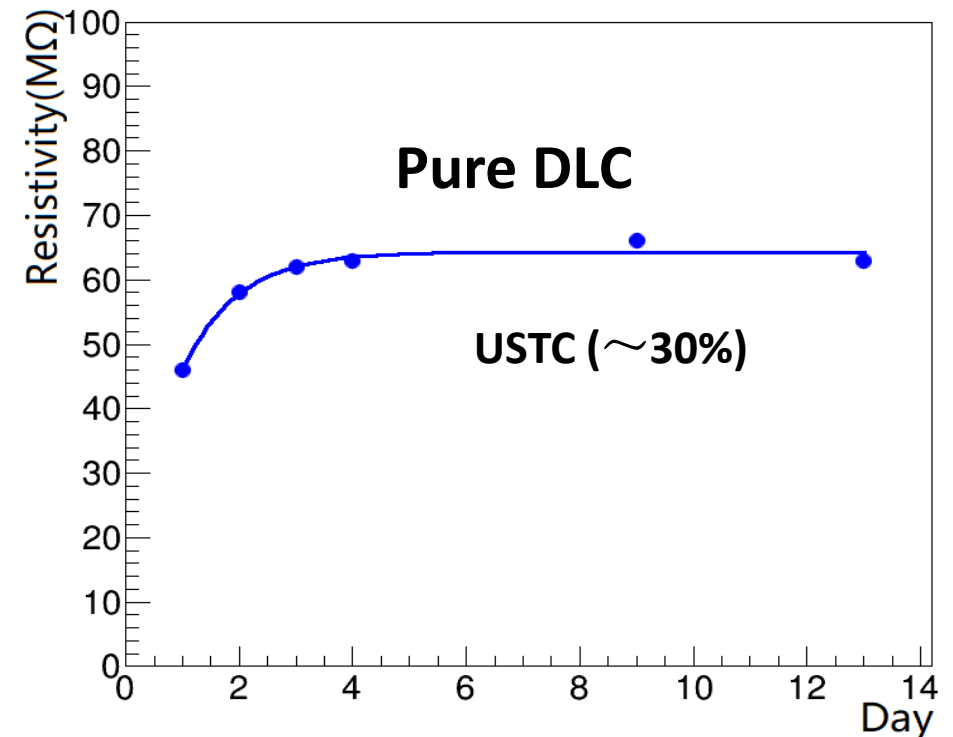
Data on this page comes from Resistive DLC Collaboration

- To estimate the resistivity change before sputtering

## Resistivity vs exposure time (very preliminary)



The resistivity of the a-C increased about 30% in three days, then became stable.



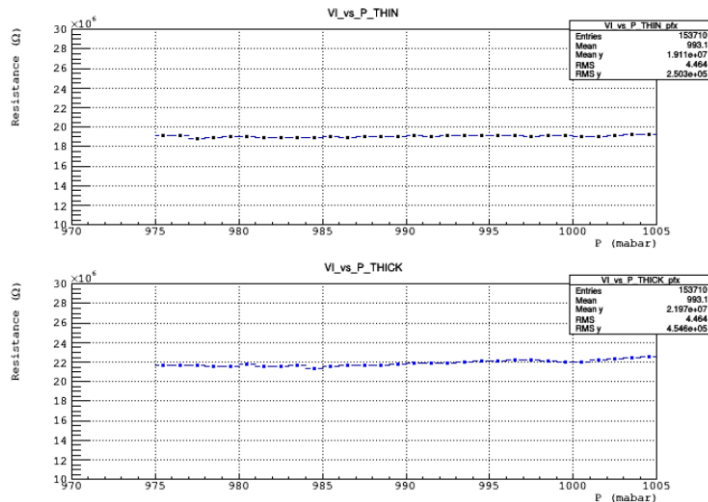
- DLC resistivity will increase in air after it was take out from the sputtering chamber;
- The test results from Kobe are more or less compatible with USTC results;

# Stability in air

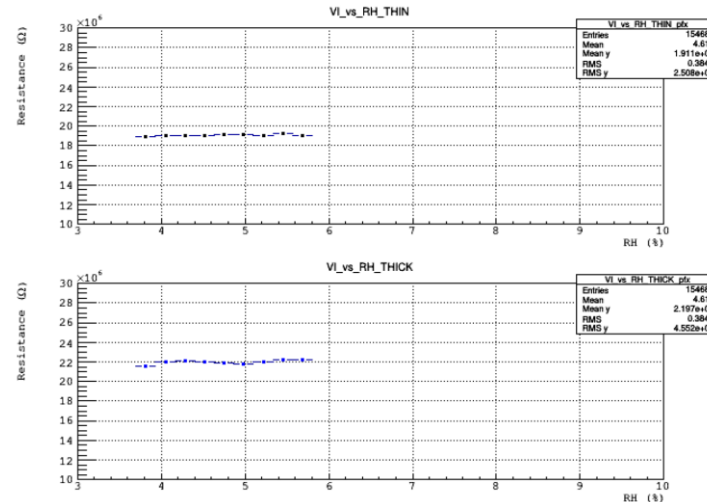
Data on this page comes from Resistive DLC Collaboration

- To make sure the DLC has long term stability in air

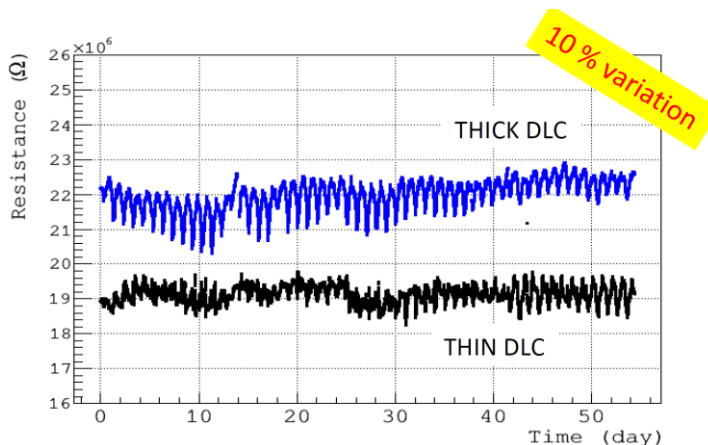
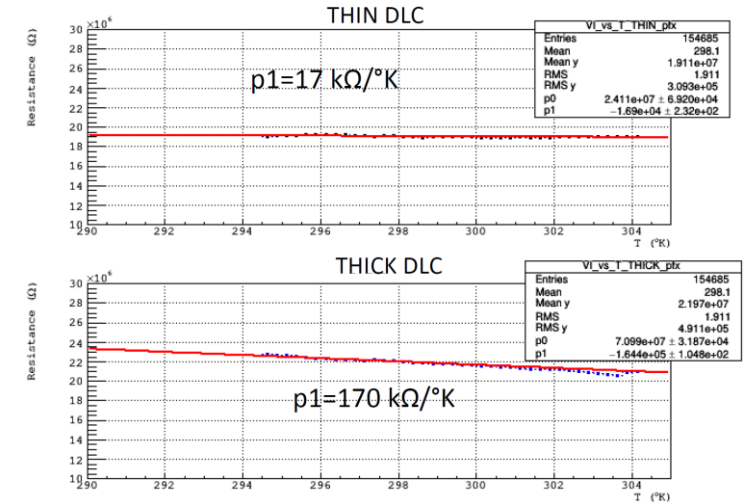
## Resistivity .VS. Pressure



## Resistivity .VS. Humidity



## Resistivity .VS. Temperature



## Conclusions:

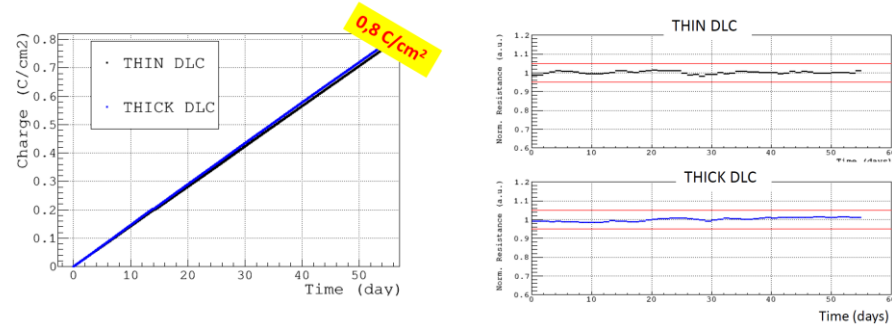
- The DLC resistivity is not sensitive to the pressure and humidity;
- The DLC resistivity will decrease when the temperature increase, more systematically measurement should be done in future;
- DLC has a very good long term stability in air;

# The aging measurement

Data on this page comes from Resistive DLC Collaboration

➤ To make sure the DLC is radiation hard

DLC stability under current drawing

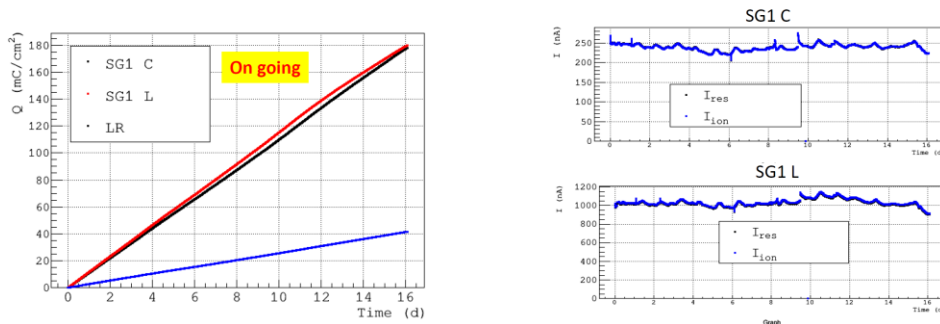


No effects observed after an integrate charge of  $\sim 1 \text{ C/cm}^2$

Detector aging: X-Ray LNF



X-Ray gun- spot 50 cm<sup>2</sup> - Flux up to  $\sim 1,2 \text{ MHz/cm}^2$

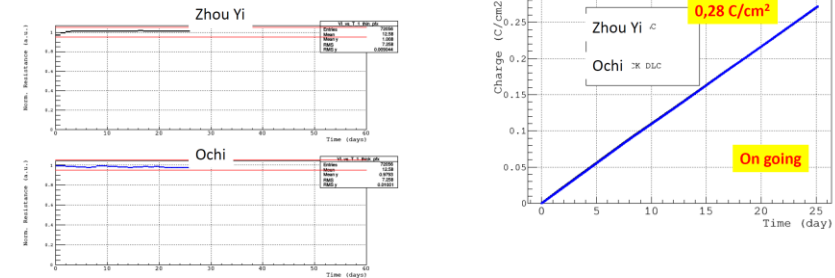


DLC stability under current & X-ray



Similar setup of the previous measurement + X-ray gun irradiating the DLC surface

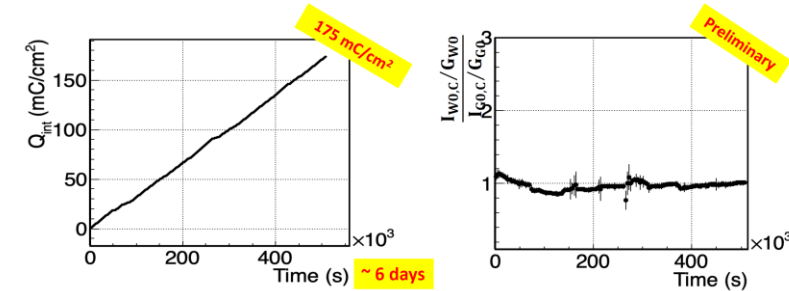
DLC foil (in black) produced by Zhou Yi  
DLC foil (in blue) produced by Be- Sputter - Ochi



Detector aging: PSI



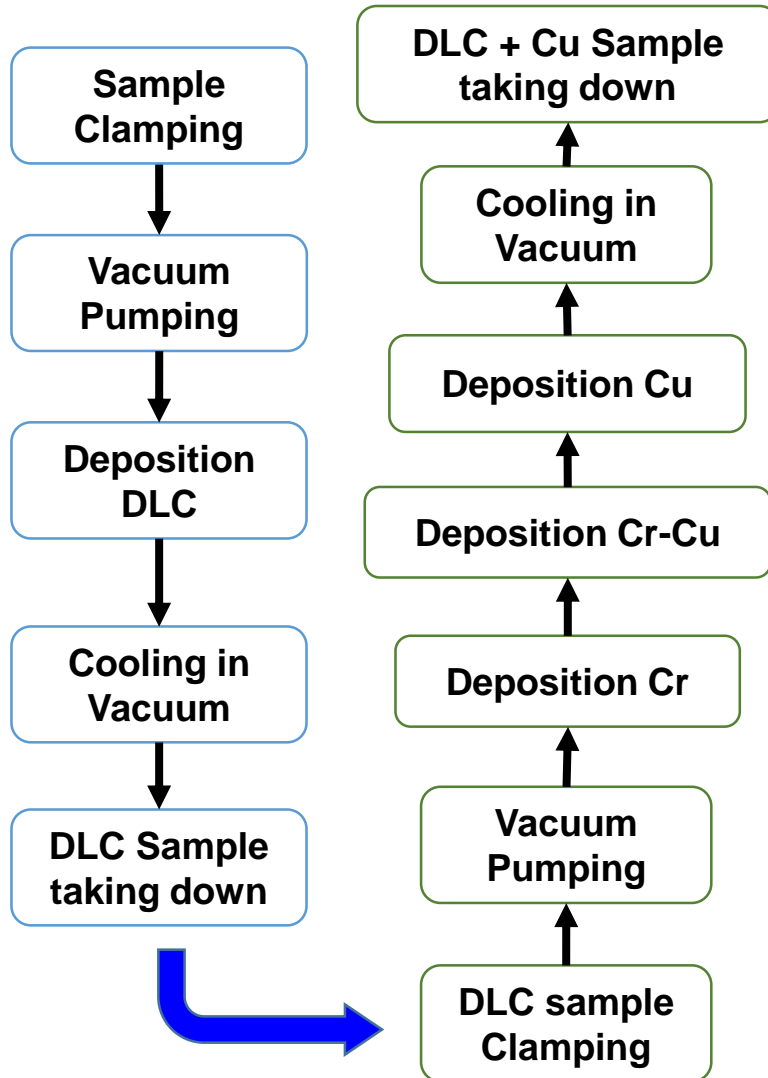
PSI TB, beam spot  $\sim 10 \text{ cm}^2$   $\Phi \sim 7 \text{ MHz/cm}^2$



This result suggests the possibility to easily integrate in several (4-6) months a dose equivalent to 10 years of operation at the HI-Lumi LHCb.

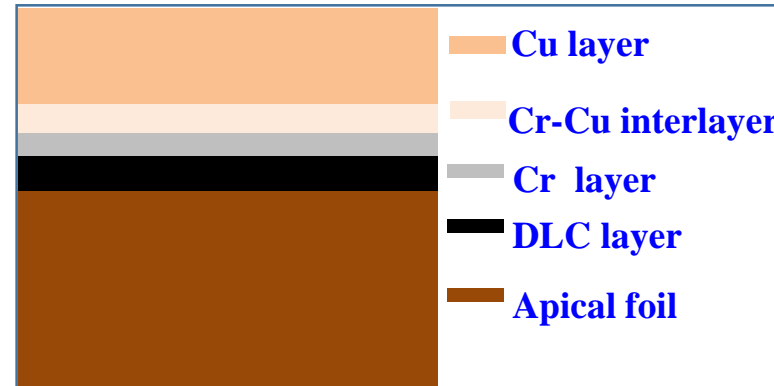
Conclusion: Currently looks good but we need longer time aging test to make sure it is good enough!

# Preparation of DLC + Cu



## Advantages of "DLC + Cu"

- Simplifying the manufacture process and improving the quality of resistive MPGDs
- Allowing precise printed circuit layouts on DLC resistive electrode thus realizing complex functions
- Expanding the capacity and applications of the MPGDs and opening the way for new MPGD architectures

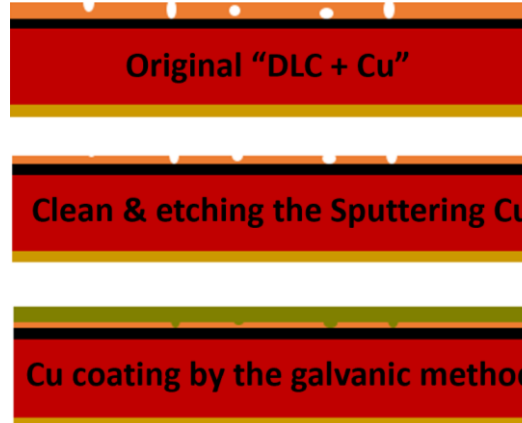
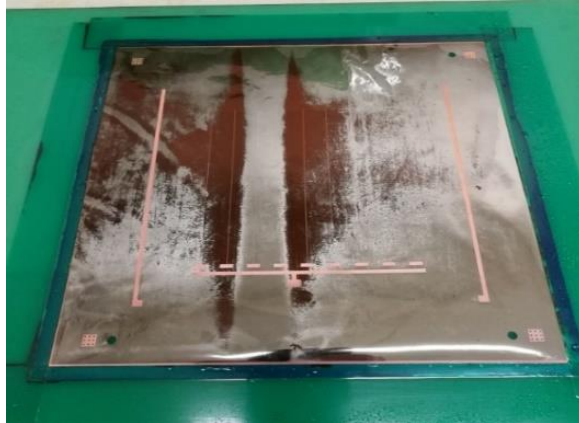




# Problems found in DLC + Cu and the Solutions

## Problems

1. Adhesion between Cr/DLC;
2. Etching of the Cr layer and Cr/Cu co-deposition layer;
3. Cavities inside the copper;
4. Resistivity decreasing after copper coating



## Solutions

1. Increasing the roughness of the substrate and add DLC-Cr co-deposition layer;
2. Optimize the thickness of Cr and Cr-Cu co-deposition layer;
3. Extra Cu coating by galvanic method;
4. Calibrate the decreasing factor by covering the sample with aluminum foil;



1. Introduction of the DLC

**2. Applications of resistive DLC in gaseous detectors**

3. Summary

# The First application of DLC in MPGD

In 1996, high resistivity DLC ( $R \sim 10^{14} \Omega/\square$ ) was firstly used in MSGC to remove the charging-up effect



ELSEVIER

Nuclear Instruments and Methods in Physics Research A 369 (1996) 328–331

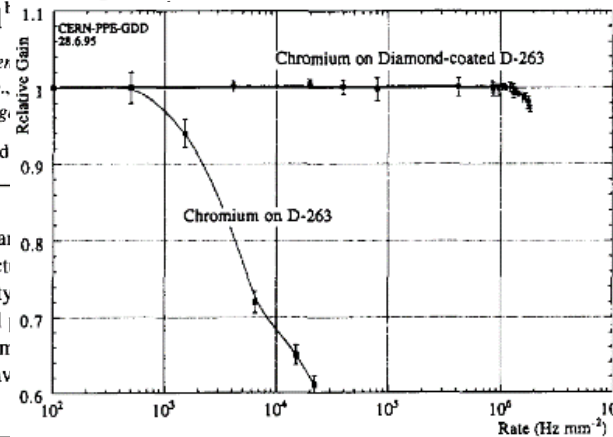
NUCLEAR  
INSTRUMENTS  
& METHODS  
IN PHYSICS  
RESEARCH  
Section A

Letter to the Editor

## High rate operation of micro-strip gas chambers on diamond-coated glass

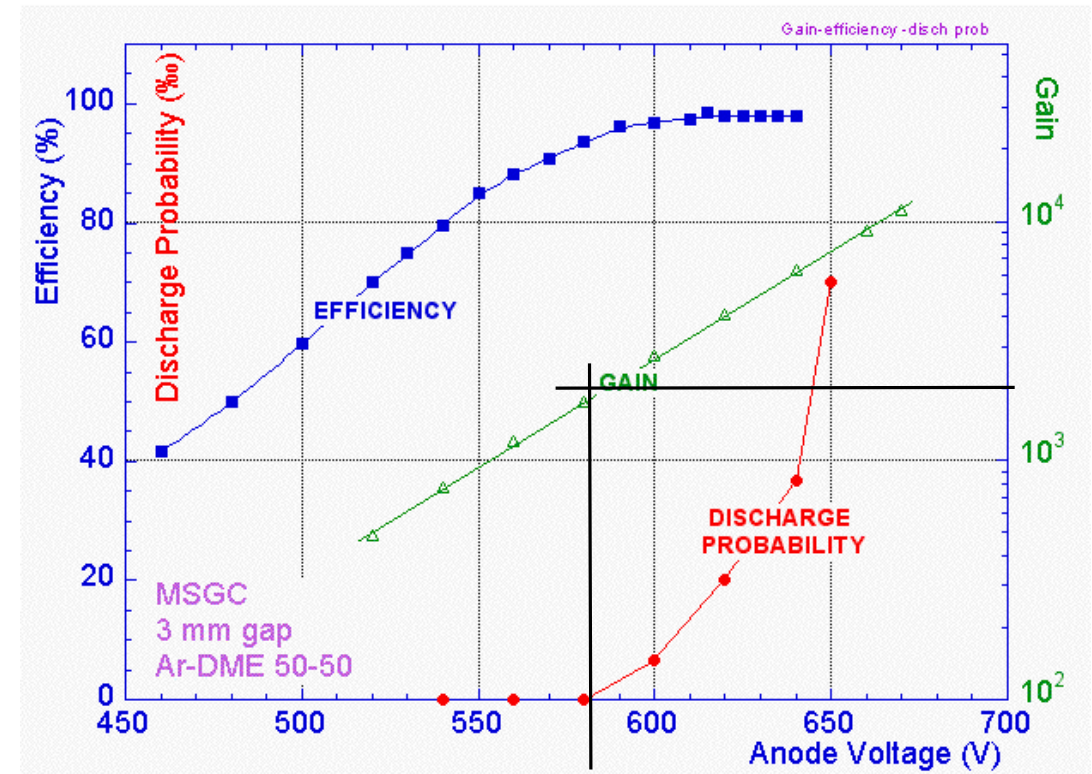
R. Bouclier<sup>a</sup>, M. Capeáns<sup>a</sup>, G. Million<sup>a</sup>, L. Ropelewski<sup>a</sup>, F. Sauli<sup>a,\*</sup>, T. Temmel<sup>a</sup>,  
R.A. Cooke<sup>b</sup>, S. Donnel<sup>c</sup>

<sup>a</sup>CERN, Geneva  
<sup>b</sup>SURMET Corp.,  
<sup>c</sup>IMT Masken und Teilung



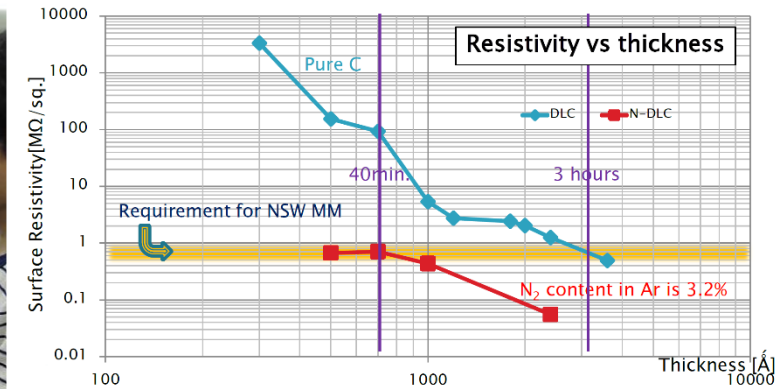
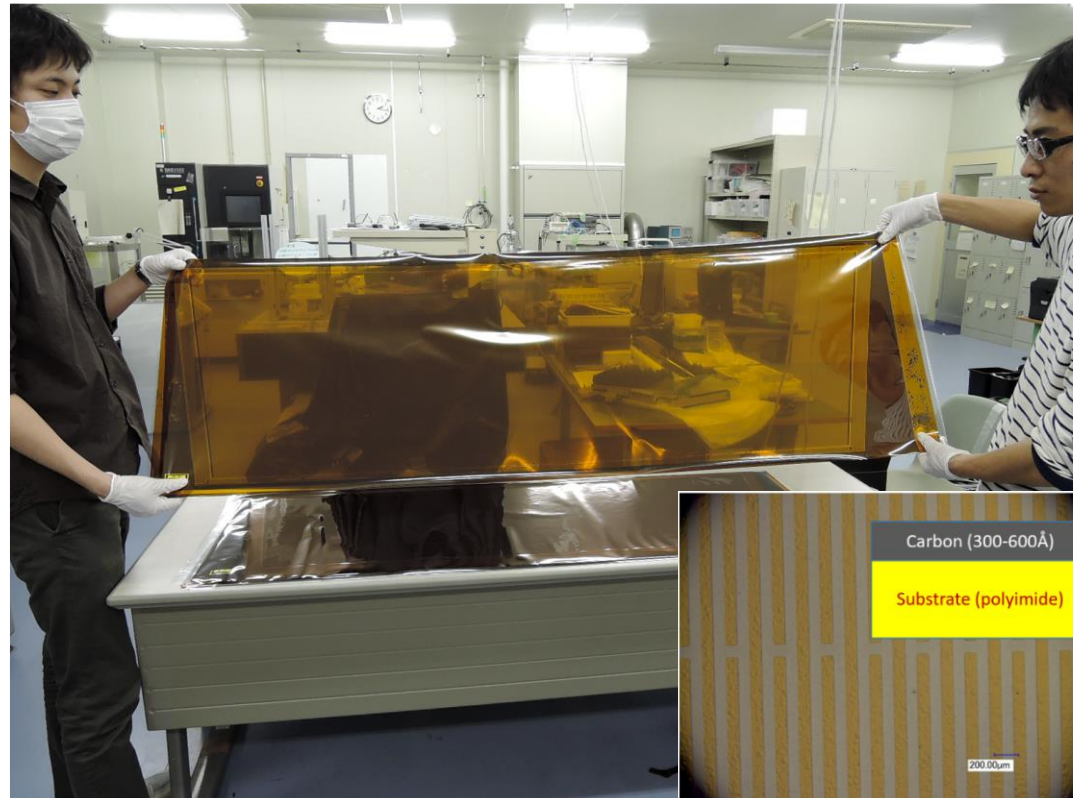
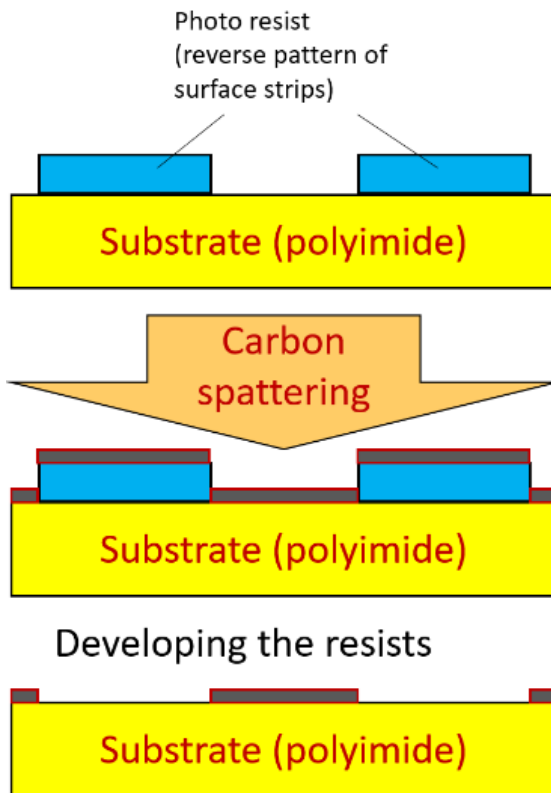
### Abstract

Very high rate operation of micro-strip gas chambers can be achieved by preliminary measurements realized with detectors manufactured with a diamond layer having a surface resistivity capability largely exceeding the one obtained with identical results are confirmed by long-term measurements, the diamond with a moderate cost overhead, to use thin, commercially available production of gas micro-strip detectors.



- × The resistivity of this DLC is too large, it can not be used as a resistive electrode;
- × This DLC is made by the low pressure plasma assisted CVD(LPCVD), it is very difficult to precisely control the resistivity by using LPCVD.

# The First Application of Resistive DLC in MPGD

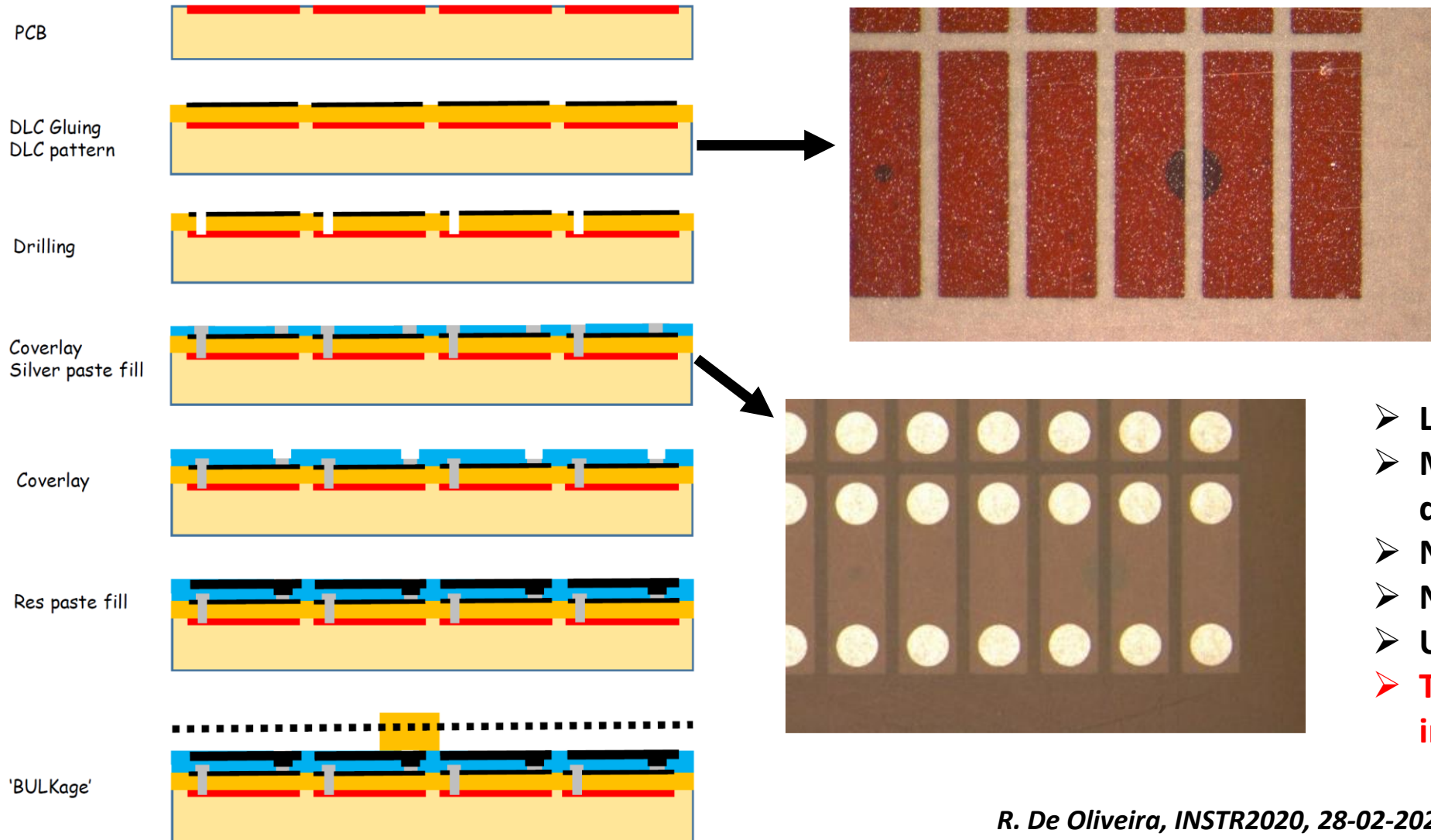


In 2013, Atsuhiko Ochi (collaborated with Be-Sputter) successfully made DLC strips with  $\sim 1\text{M}\Omega/\square$  resistivity by using the magnetron sputtering method and lift-off technique.

*A.Ochi, New Small Wheel MicroMegas Mechanics and layout Workshop, 25-June-013*

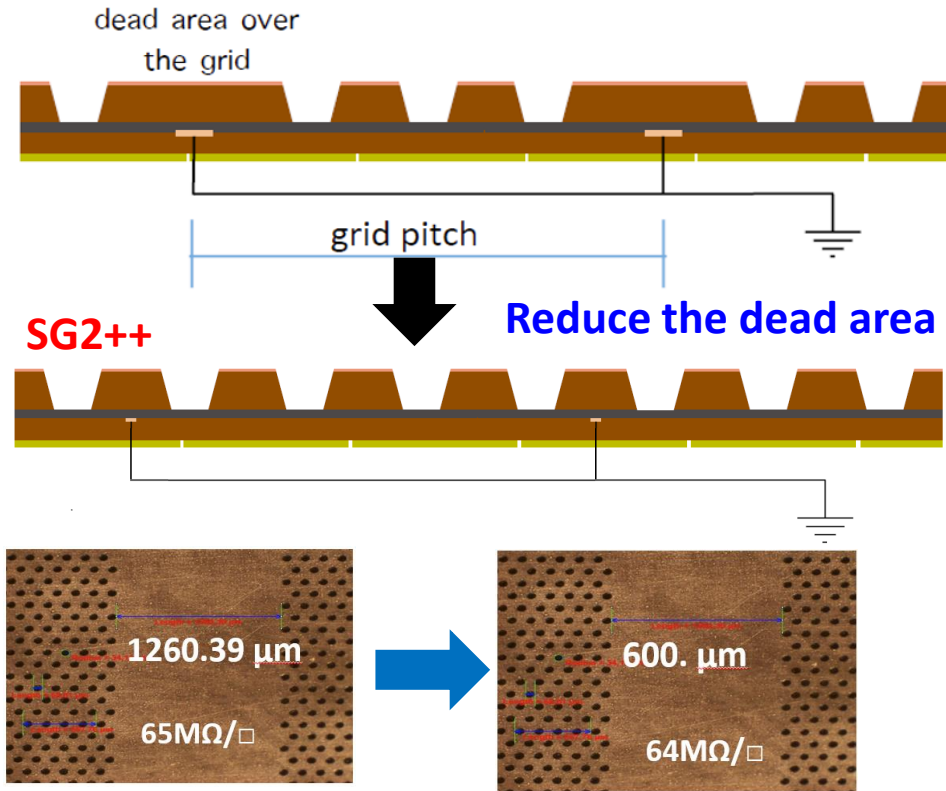
*A.Ochi, RD51 mini week, 08-June-2015*

# Micromegas Made by MIX (DLC + Screen Printing) Method

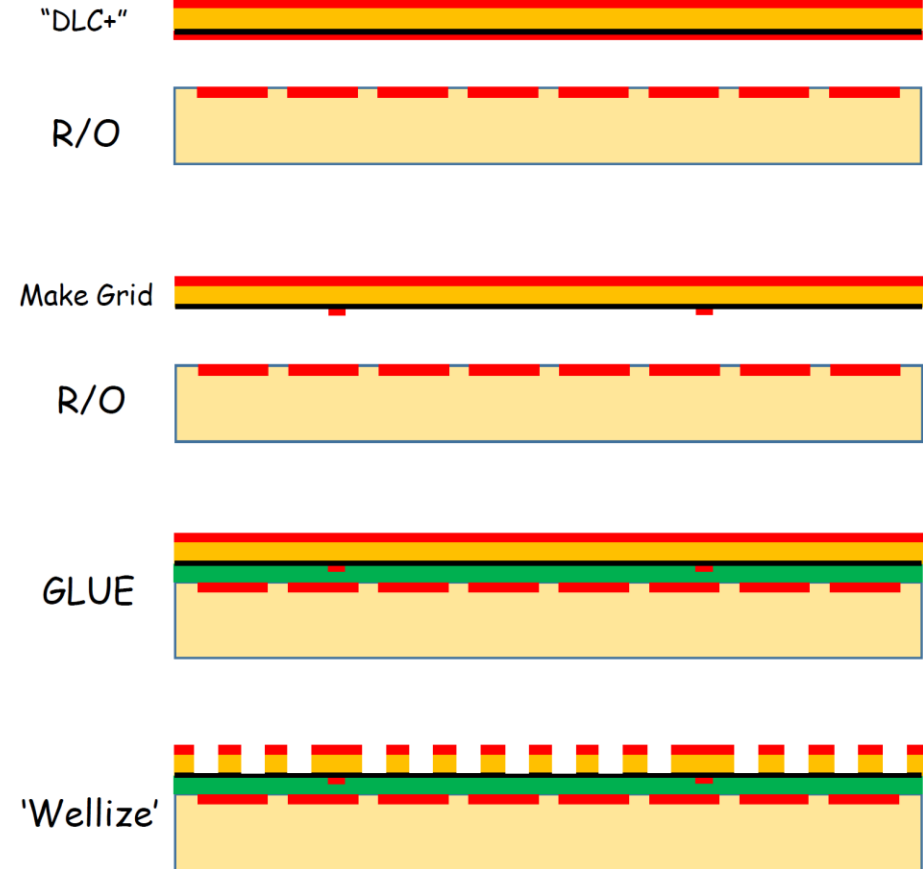


- Large DOCA (2mm)
- Maximized evacuation density points
- Needs simple DLC foils
- No problem with large size
- Ultra high rate detectors
- **The filling technic is not STD in PCB world**

# $\mu$ RWELL with Fast Grounding Lines under the DLC



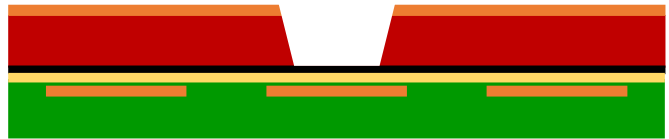
- ✓ *Photolithography is applied on the copper on DLC to make precise grounding lines*
- ✓ *Detection efficiency of SG2++ is better than 97%*
- ✓ *Gain drop of SG2++ <10% @  $\sim 10$  MHz/cm<sup>2</sup>*



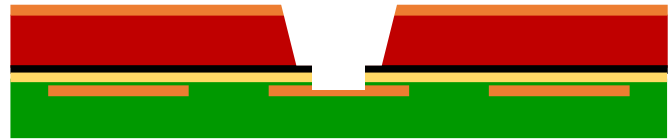
**There are alignment problems when it goes to large area**

# $\mu$ RWELL with GND Patterns on Top Surface

## PEDF: Patterning , Etching , Drilling & Filling



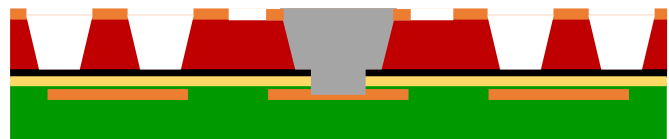
Step1: Copper & APICAL etching, to make a big hole, with DLC on bottom.



Step2: Drill a small hole, the copper of the readout pad expose to air.



Step3: Use silver glue to connect the DLC to readout pad.



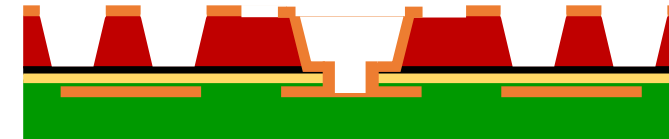
Step4: Make  $\mu$ RWELL structure and remove the copper around silver glue.

### Advantages:

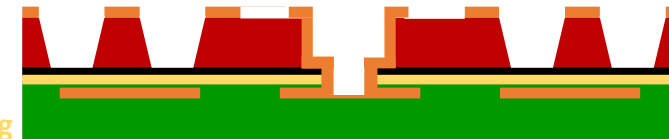
1. No copper-coated DLC needed, better resistivity control;
2. No alignment problems even goes to large area;
3. Larger contact area between DLC and silver glue, improving the connection.

## Different options of high-rate $\mu$ RWELL

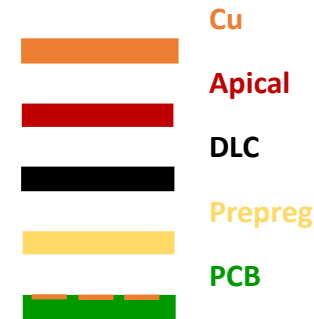
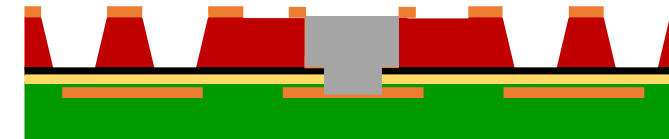
### PEDP: Patterning , Etching , Drilling & Plating (The Best One)



DEP: Drilling , Etching & Plating



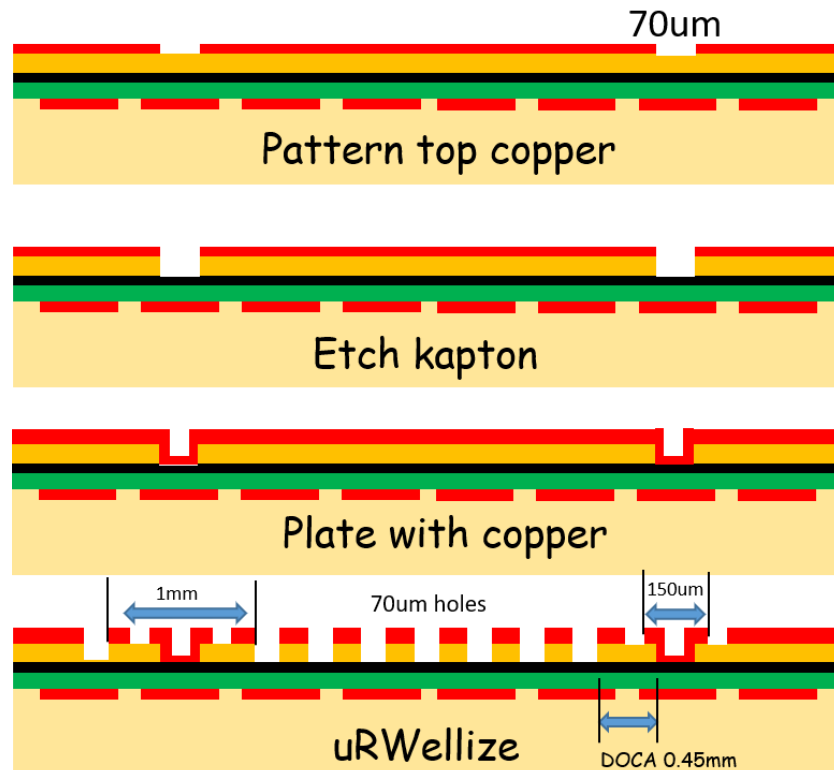
DEF: Drilling , Etching & Filling



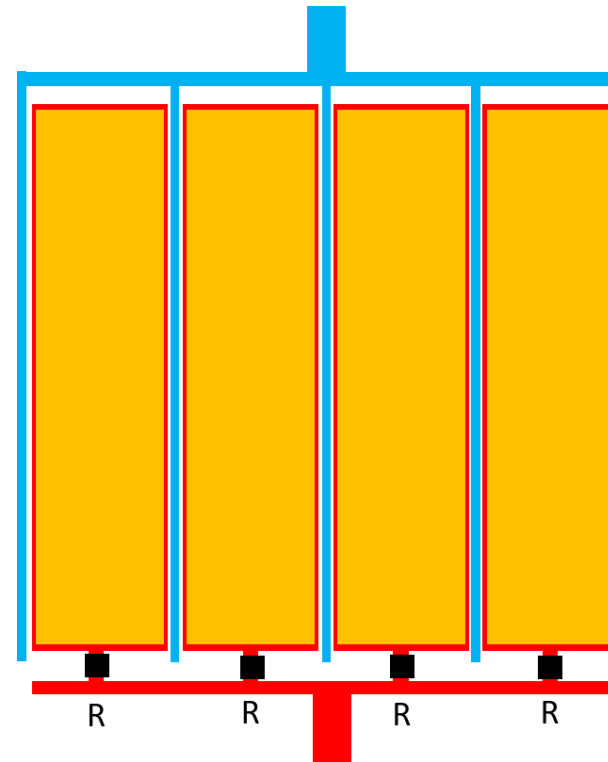
Four different types (PEDF , DEF , PEDP , DEP) of  $\mu$ RWELL PCB have been produced at CERN and transferred to USTC .

# $\mu$ RWELL Made by the PEP Technique

## PEP cross section



## PEP top view



-Cu Connection to DLC through a 70um groove or a row of 70 um vias  
-Top strip 150um  
-Grounded electrode

-Amplification Holes  
-DOCA 0.45mm  
-1mm from hole to hole

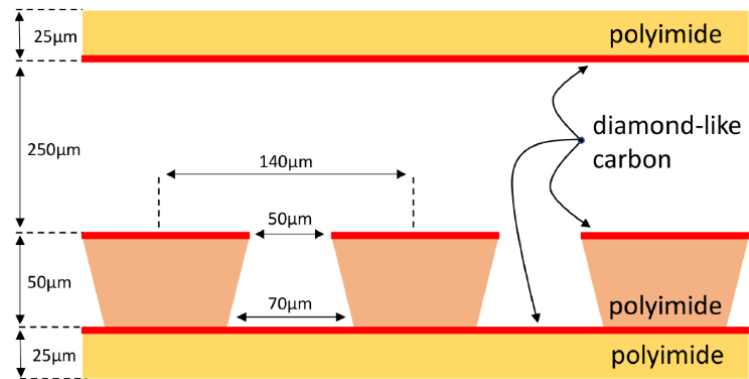
Top electrode connected to -HV  
With or W/O Resistors

Any pattern can be created to connect the DLC , row of dots but also line.  
No alignment problem for large size.  
Do not need drilling with Z axis control (simpler than previous structures).

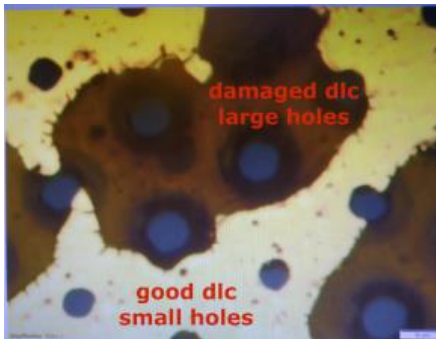
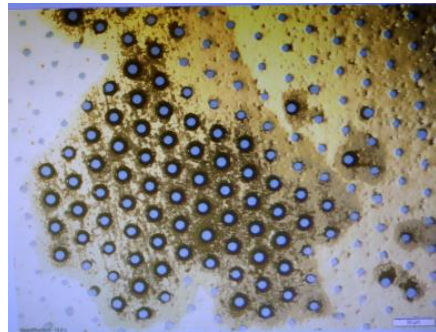
R. De Oliveira, DLC CP Meeting, 24-06-2021



# μRWELL-type FTM



## Problems



## Solution



STEP1



STEP2



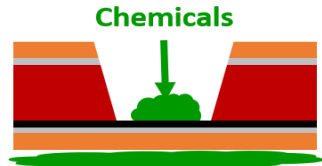
STEP3



STEP4



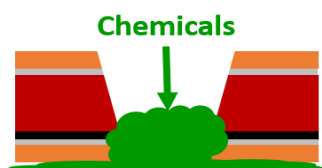
Ideal STEP3



STEP5



Real STEP3



Real STEP4



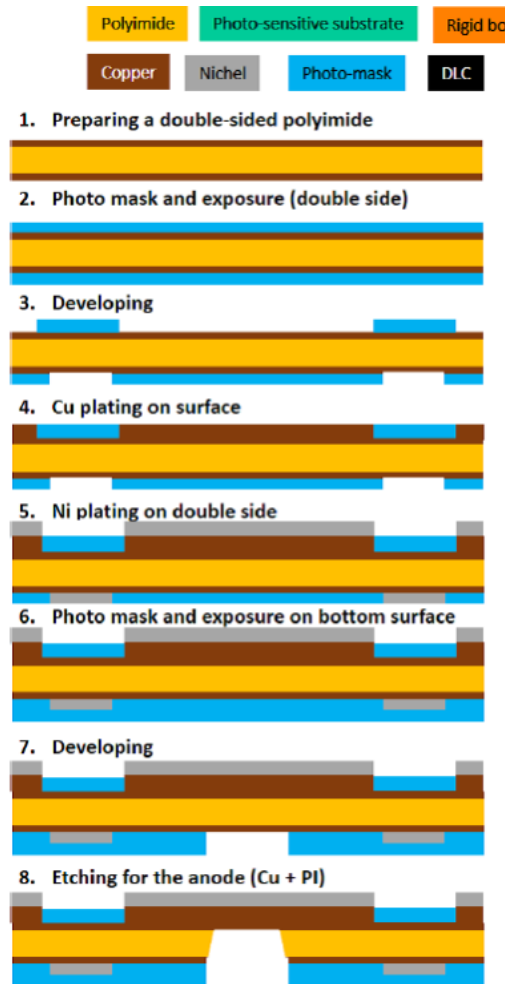
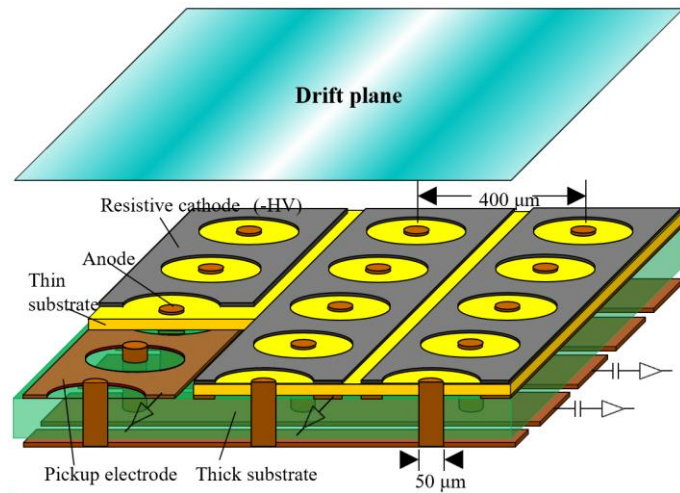
Real STEP5



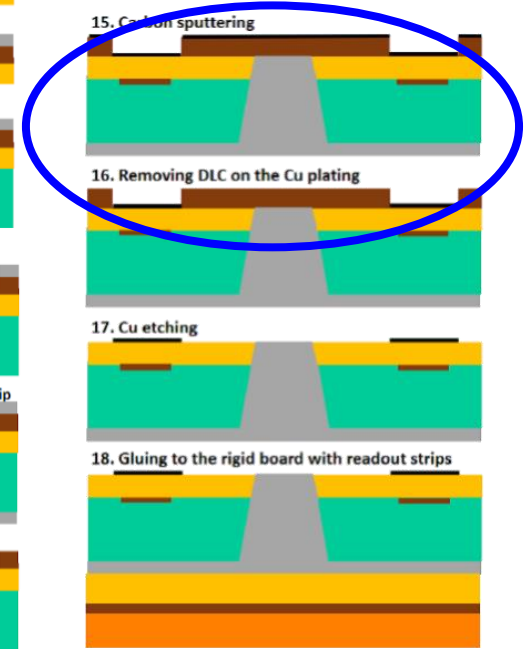
- Galvanized Cu
- Sputtering Cu
- Apical
- DLC
- Original Cu

- Use sandblasting to increase the roughness of APICAL, then increase the adhesion of APICAL and DLC;
- Using galvanized Cu to seal the cavities inside the Sputtering Cu;

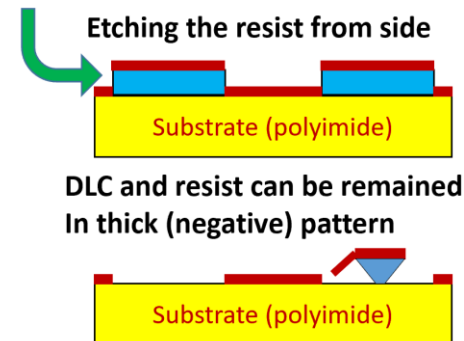
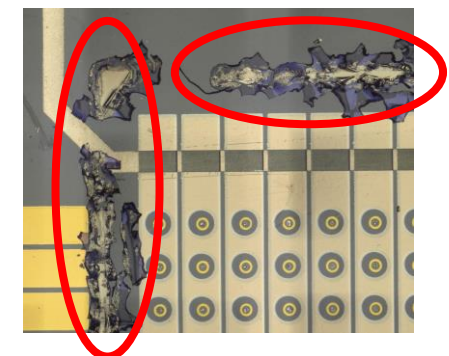
# $\mu$ -PIC with resistive DLC Cathode



## Lift-off using metal

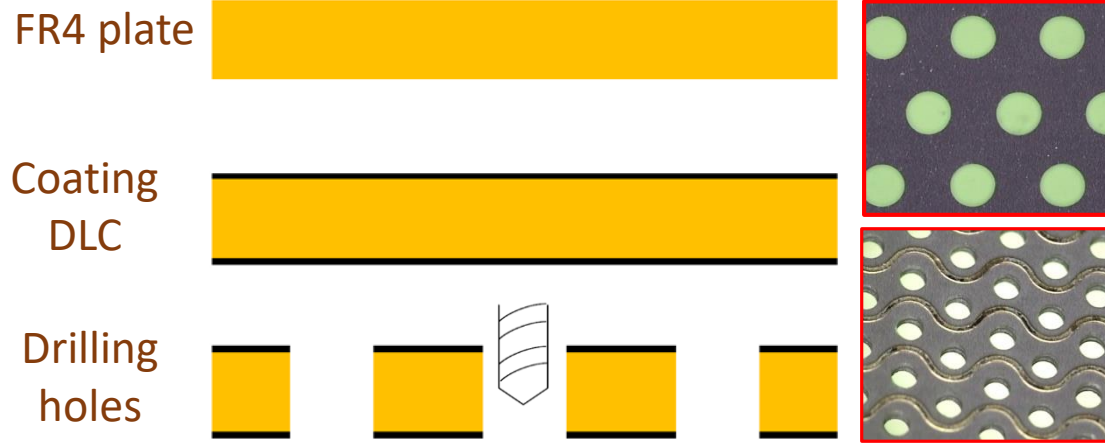


## Photoresist with DLC

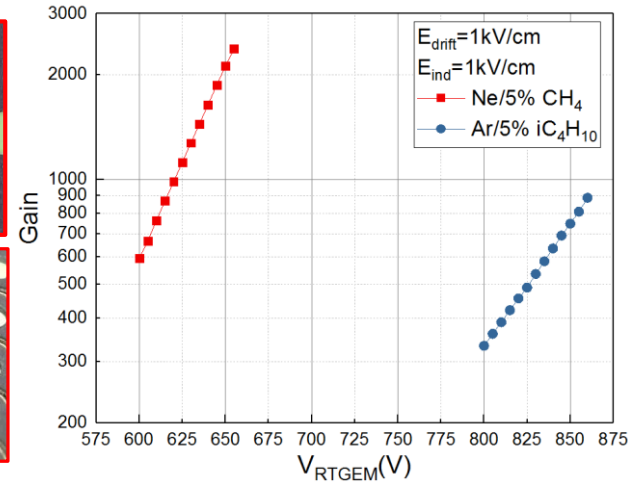


# Resistive THGEM

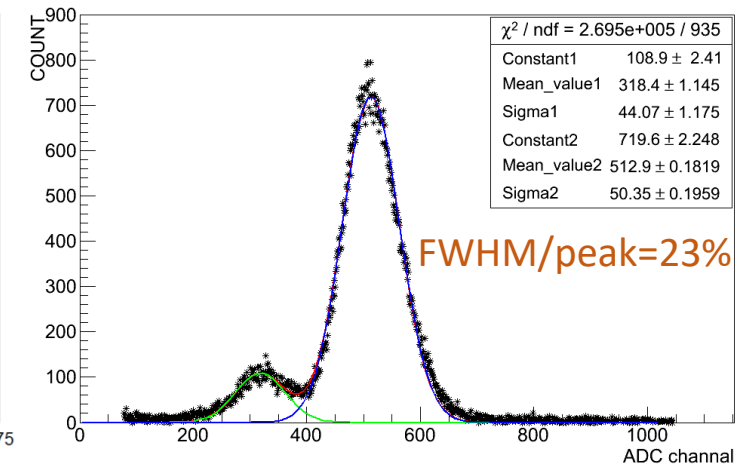
## RTGEM production process



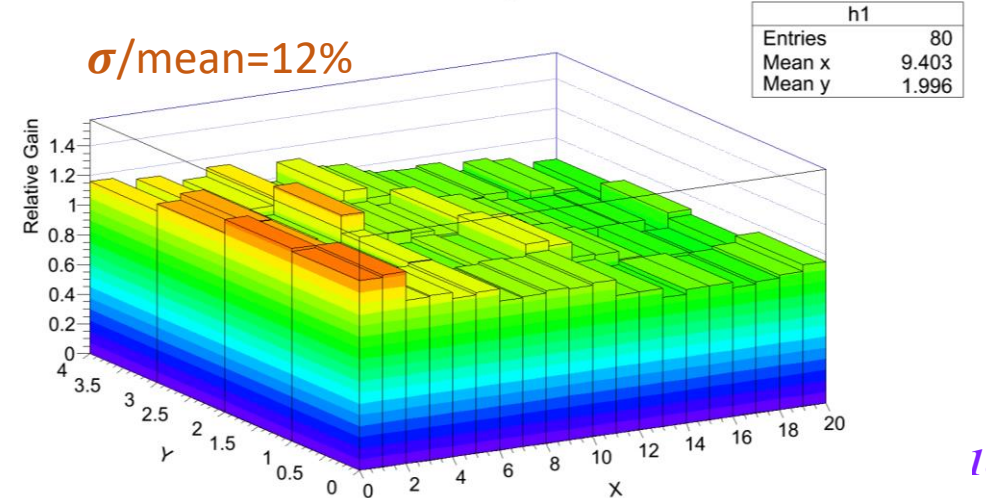
## Gain vs. HV



## Spectrum of 8keV X-ray

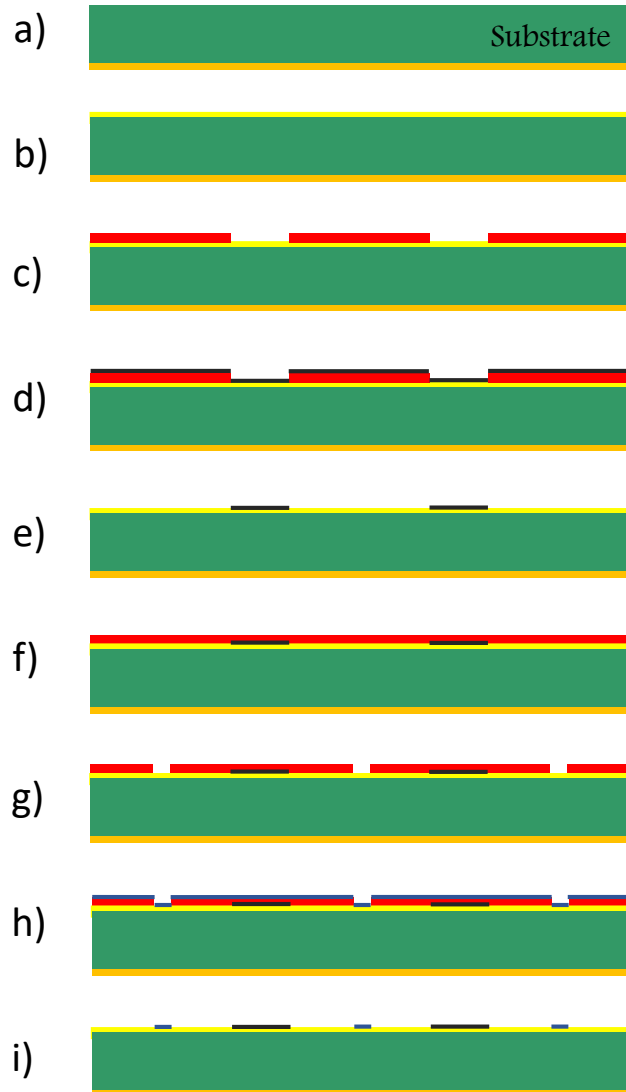


## Uniformity

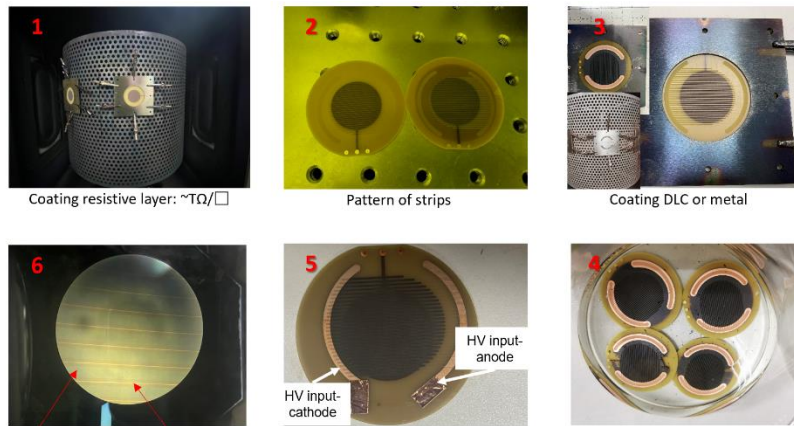
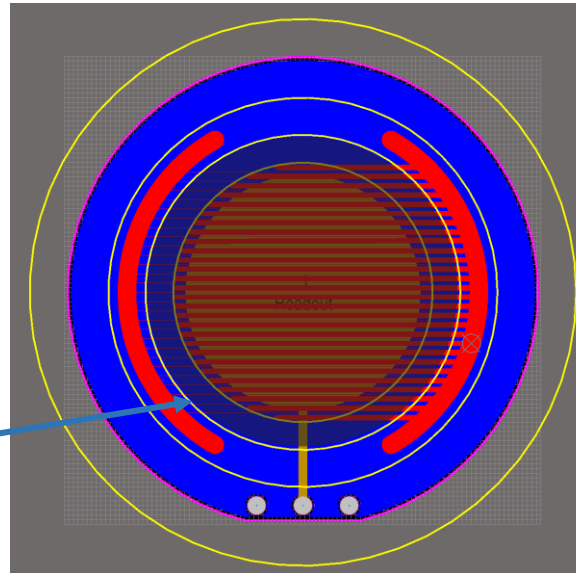


# RMSGC Made by Lift-off Method

*This project is still ongoing*



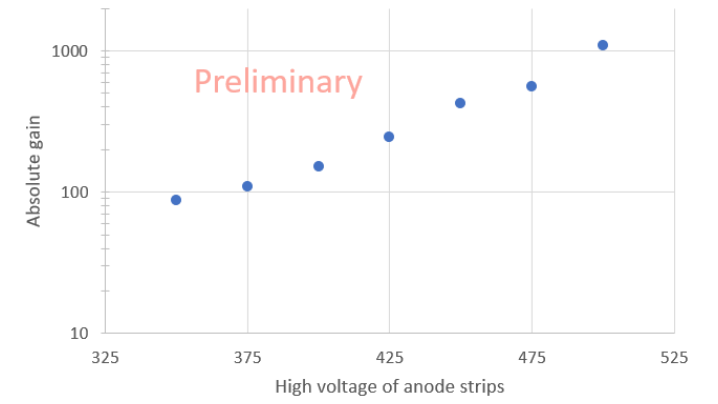
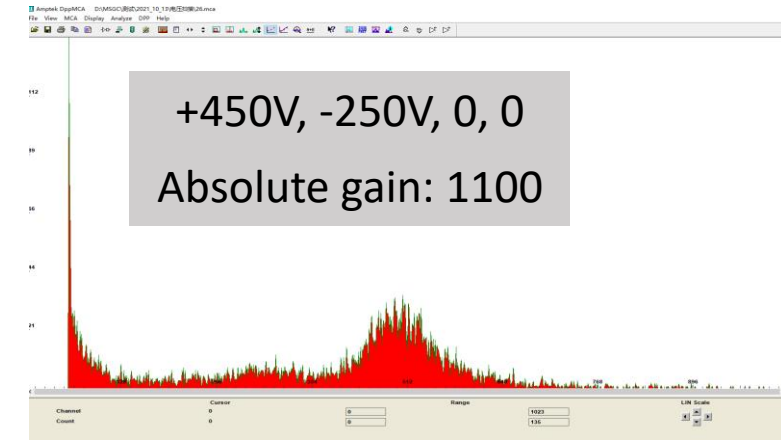
substrate  
back electrode  
photoresist  
DLC electrode  
metal electrode  
DLC film



DLC strips  
600 $\mu$ m width 50nm thick  
 $\sim$ 130M $\Omega$ /sq,  $\sim$ 400M $\Omega$ .cm

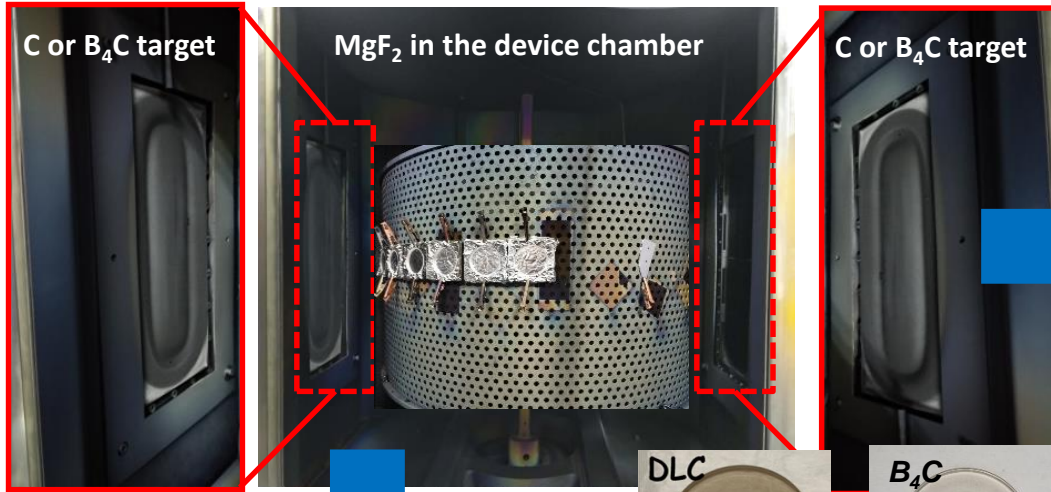
Cu strips: 20 $\mu$ m width, 80nm thick

Anode strips: 20 $\mu$ m  
Cathode strips: 600 $\mu$ m  
Pitch: 1mm

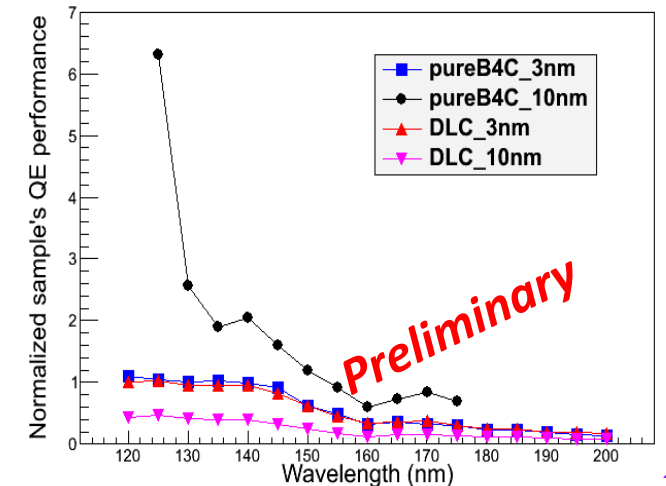
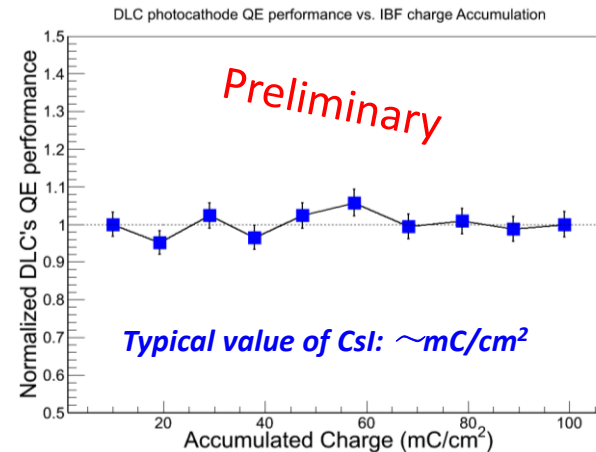
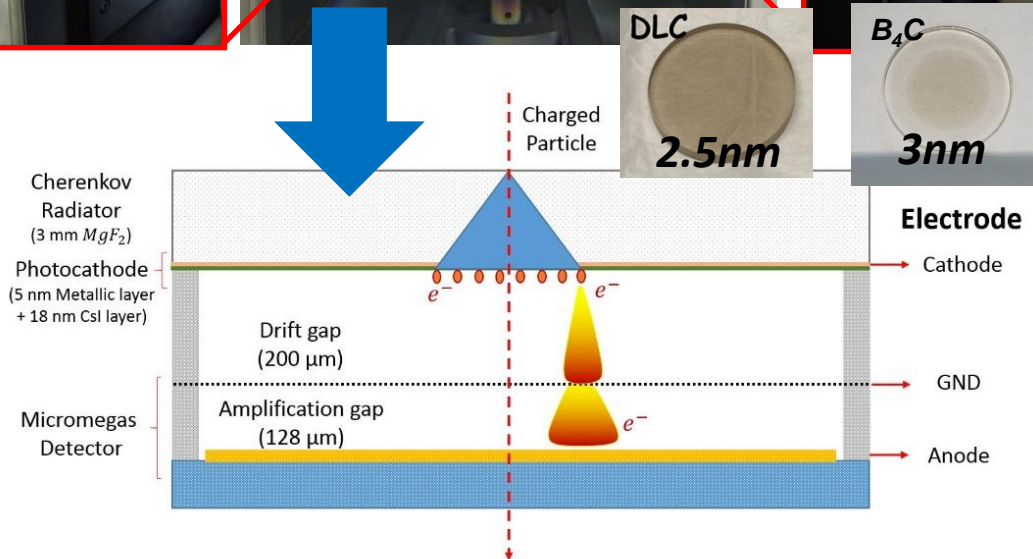
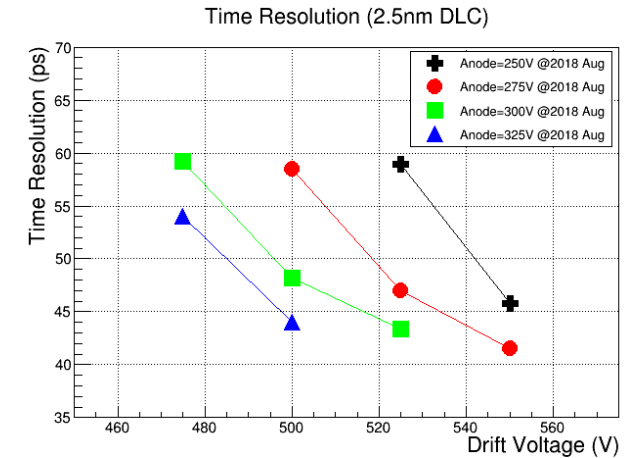


# DLC on MgF<sub>2</sub>—Robust photocathode for Picosec-MM

## DLC & B<sub>4</sub>C/a-C:B photocathode for PICOSEC-MICROMEAS

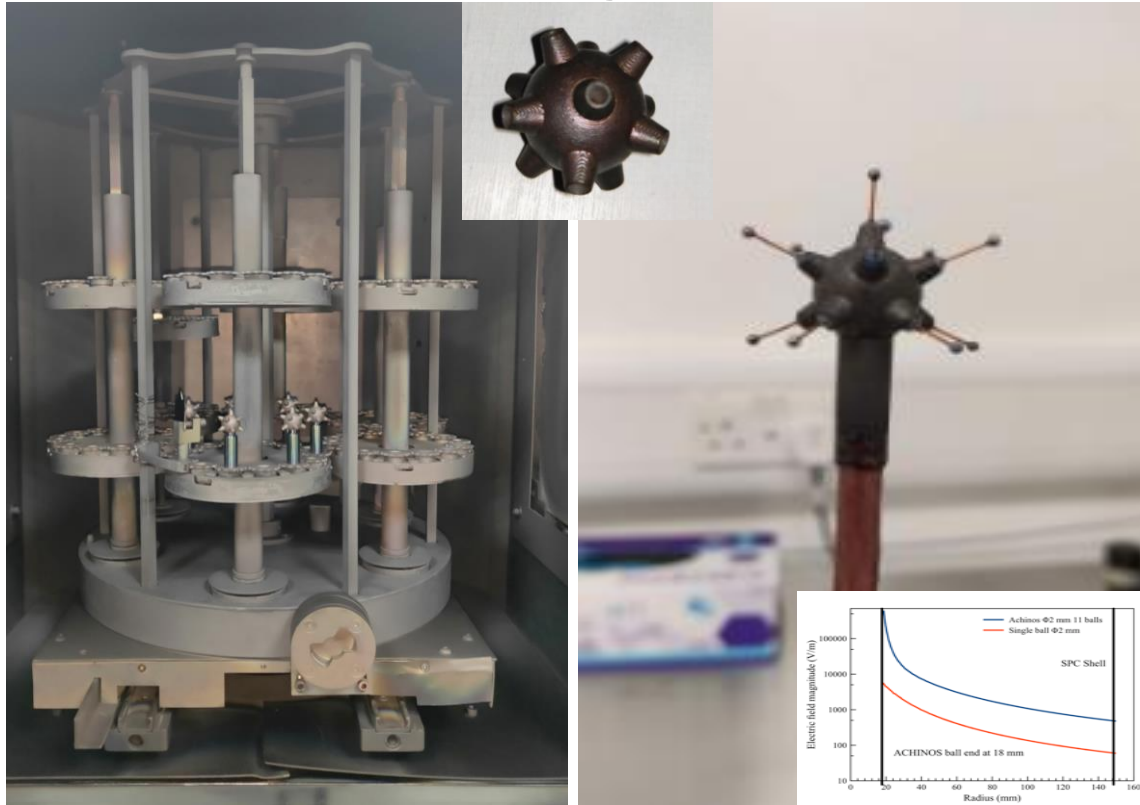


Anode/Drift Voltage (V)	Time resolution (ps)	
	Aug.	Oct.
250/-550	45	37
275/-525	47	38
275/-550	42 Preliminary	34
300/-500	48	39
300/-525	43	34



# DLC on spherical resin—Resistive ACHINO

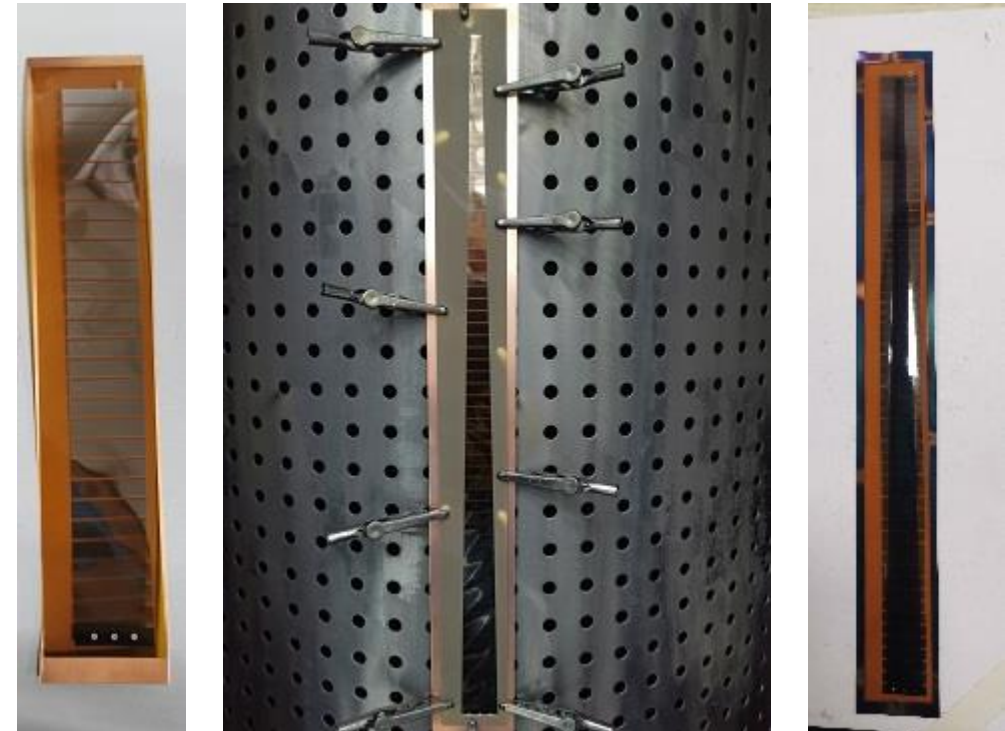
## 3-axis DLC coating for ACHINO



The electric field is stronger (7x) with multi ball compared to a single read-out

*I. Giomataris, RD51 Collaboration Meeting, 22-10-2019*

## DLC coating for Degradator



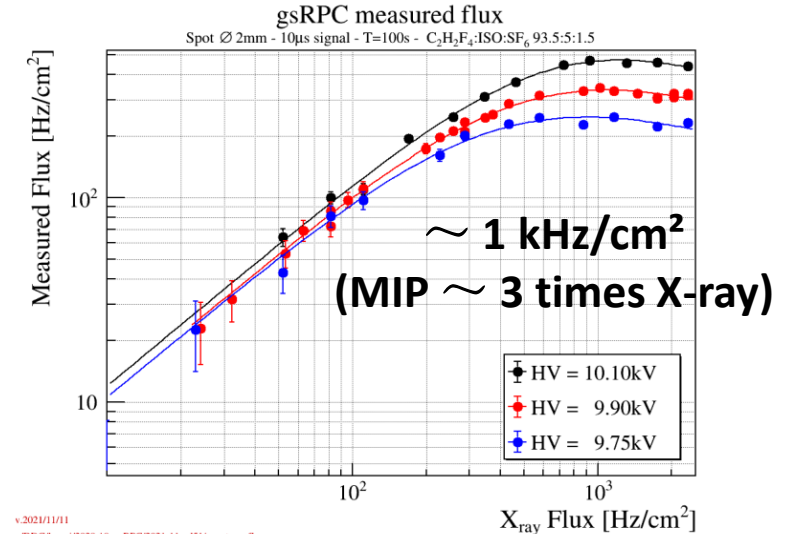
*More details can be found in presentation of K. Nikolopoulos on this conference*

# The Surface Resistive Plate Counter (sRPC)

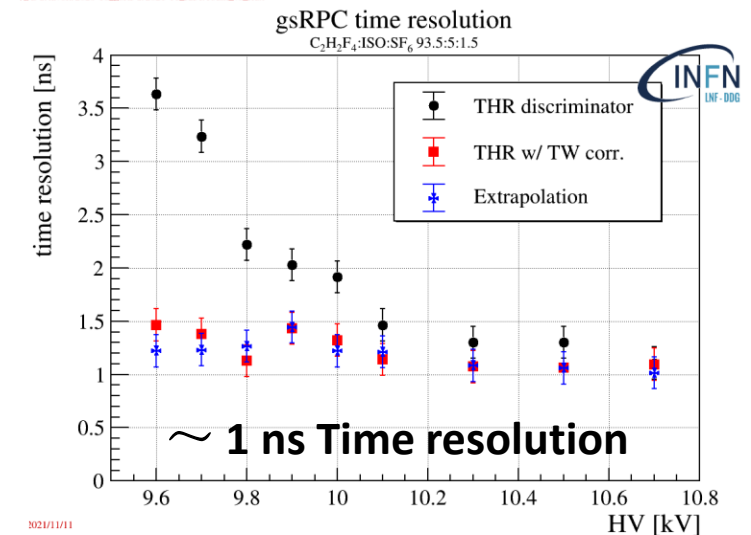
- **Surface resistivity electrodes** manufactured with industrial sputtering techniques of **DLC on flexible supports**
- The technology allows to realize electrodes with a surface resistivity in a **very wide range,  $0.01 \sim 10\text{G}\Omega/\square$**
- **High density current evacuation schemes**, similar to the ones used for **resistive MPGD ( $\mu\text{RWELL}$ )**, can be implemented to **increase the rate capability** of the detector



G. Bencivenni, RD51 Collaboration Meeting, 16-11-2021



v.2021/11/11  
~DDG/favori/2020-10\_gsRPC/2021-11\_rd51/count\_vs\_flux



2021/11/11  
DDG/favori/2020-10\_gsRPC/2021-11\_rd51/time\_2021119



1. Introduction of the DLC

2. Applications of resistive DLC in gaseous detectors

**3. Summary**



# Summary

- **DLC has been already introduced in the manufacturing of innovative MPGDs as resistive electrode, in spite of the possibility to sputter DLC on large area with good chemical/physical tolerances, there are still some open problems requiring for a dedicated detailed studies.**
- **DLC with Copper (DLC + Cu) is an advance material for MPGD manufacture, it is able to open new possibilities to produce advanced MPGD architectures.**

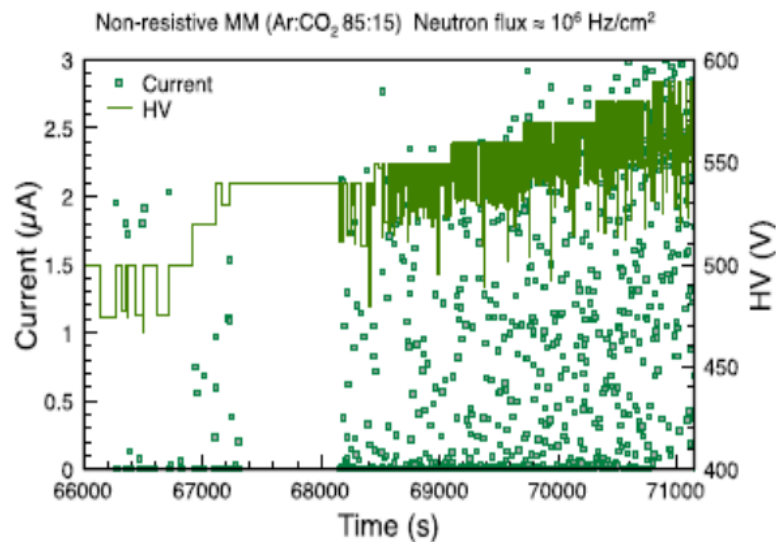
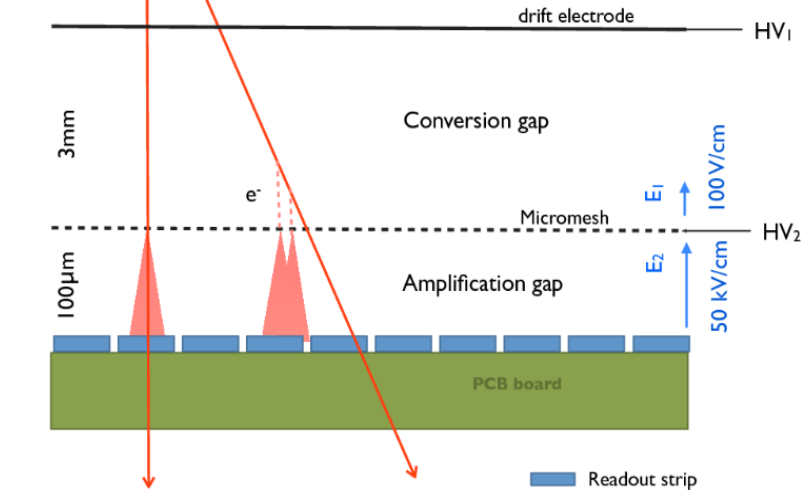
**Thanks**



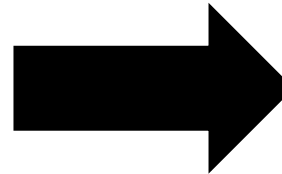
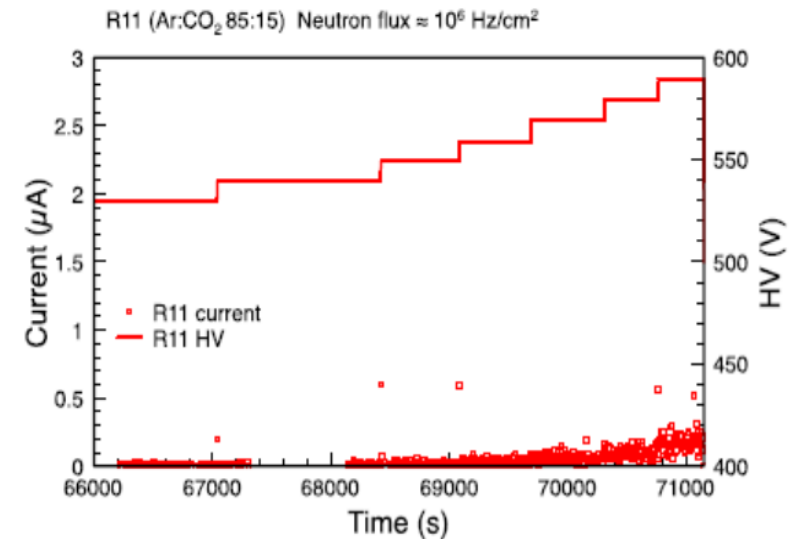
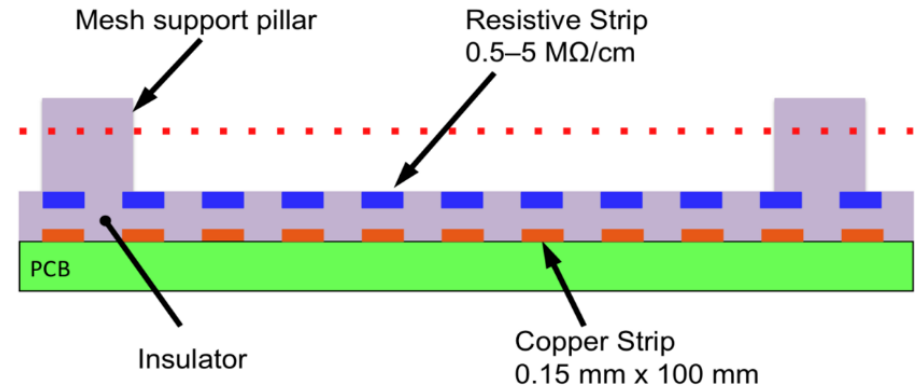
# Backup

# Resistive Electrode Can Effectively Suppress The Discharges

## Non-Resistive Micromegas

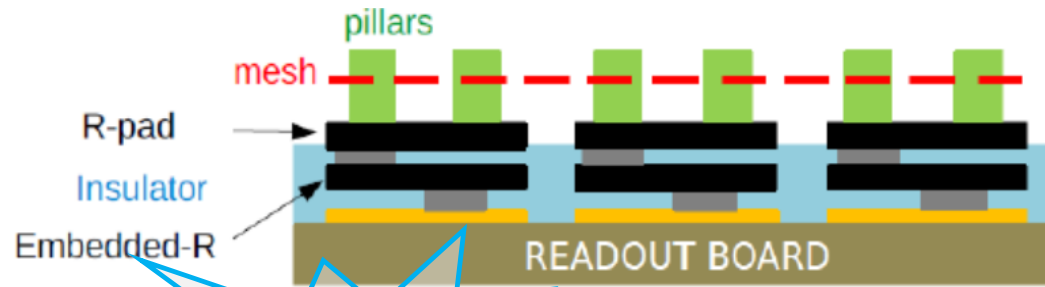
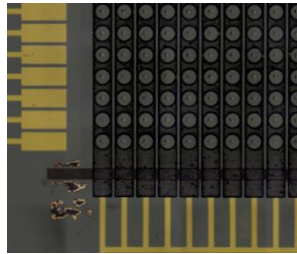
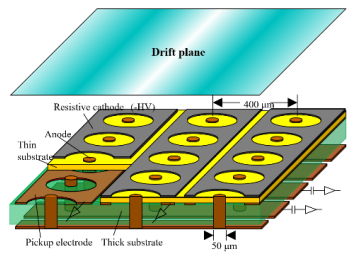


## Resistive Micromegas

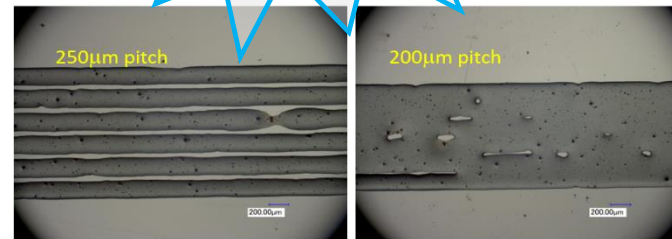
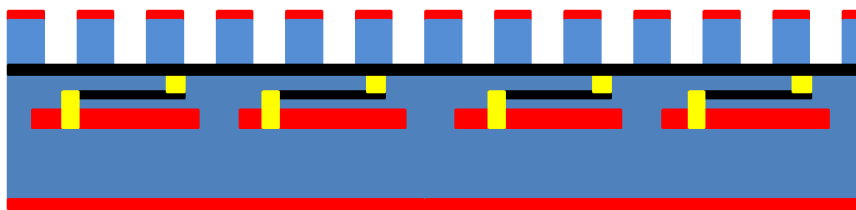


# Resistive Materials Based on Carbon Loaded Pastes

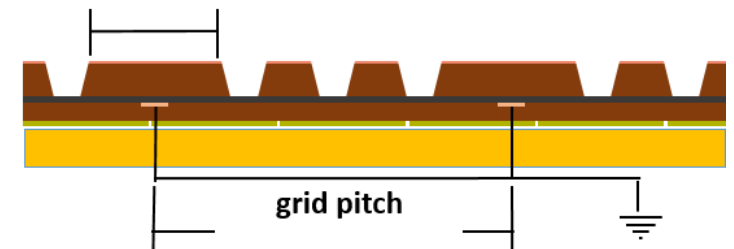
*Resistive materials based on Carbon loaded pastes have already widely used in MPGDs to suppress the discharges and reduce the damaging effects on MPGDs*



**However**



dead area over the grid



- × *Resistivity sometimes out of control in manufacture;*
- × *Unable to make fine structures;*
- × *Difficult to make conductive route on it precisely;*
- × *Can not open opportunities for new micro-structures;*

***New reliable resistive materials  
and preparation methods needed***

# RD51 Common Project & Resistive DLC Collaboration

## DLC based electrodes for future resistive MPGDs

**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, 230026*  
*telephone number: +86-551-63607940*  
*e-mail: [zhouyi@mail.ustc.edu.cn](mailto:zhouyi@mail.ustc.edu.cn)*

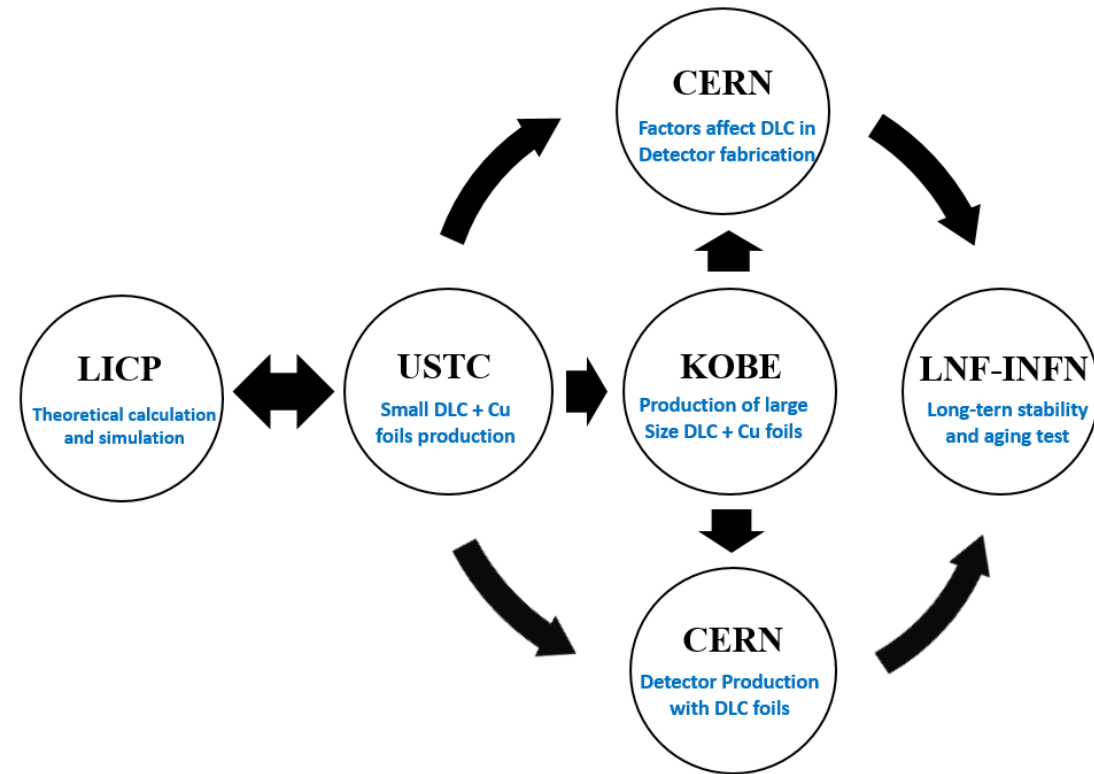
**RD51 Institutes:** 1. *State Key Laboratory of Particle Detection and Electronics, University of Science and Technology of China, contact person: Yi Zhou*  
*e-mail: [zhouyi@mail.ustc.edu.cn](mailto:zhouyi@mail.ustc.edu.cn)*

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

3. *CERN contact person: Rui de Oliveira*  
*e-mail: [Rui.de.Oliveira@cern.ch](mailto:Rui.de.Oliveira@cern.ch)*

4. *Laboratori Nazionali di Frascati dell'INFN contact person: Giovanni Bencivenni*  
*e-mail: [Giovanni.Bencivenni@lnf.infn.it](mailto:Giovanni.Bencivenni@lnf.infn.it)*

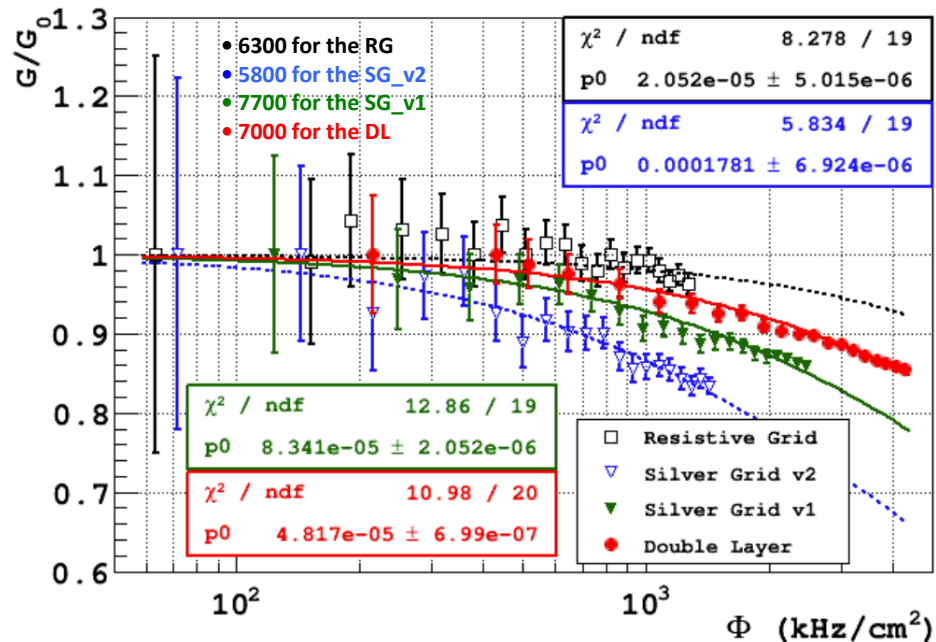
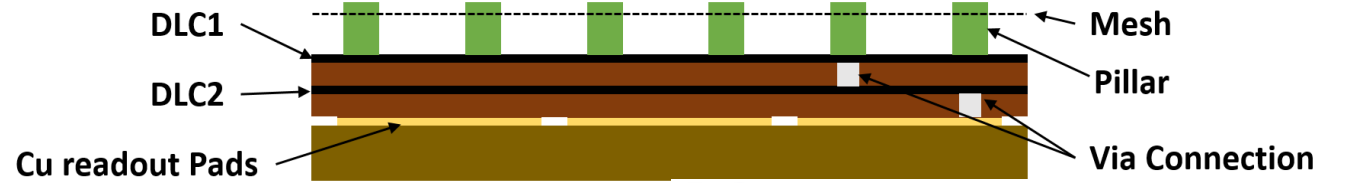
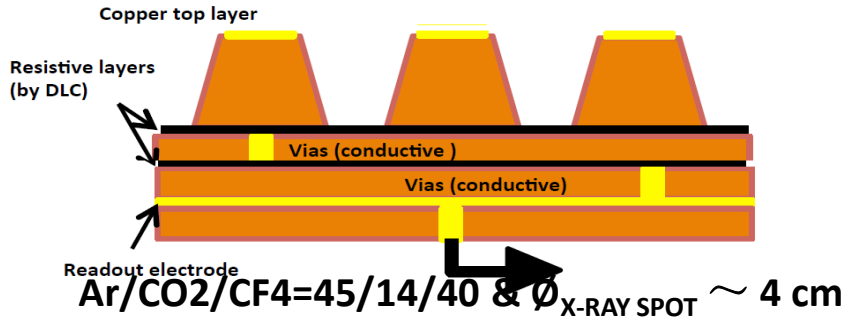
**Ext. Collaborators:** 1. *State Key Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Chinese Academy of Science contact person: Lunlin Shang*  
*e-mail: [shangll@licp.cas.cn](mailto:shangll@licp.cas.cn)*



### Goal of this project:

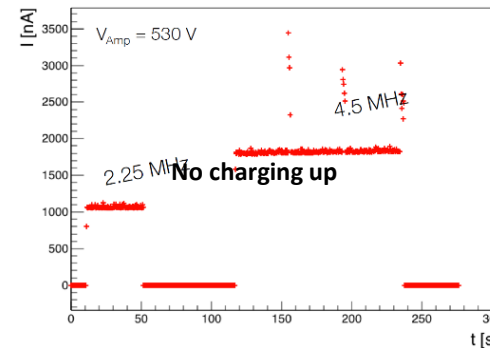
1. Define a stable and well controlled DLC and DLC+Cu processing method for the production of MPGD electrodes
2. Studying the long-term stability under irradiation of DLC and DLC-based detectors.

# μRWELL & Micromegas with Resistive DLC

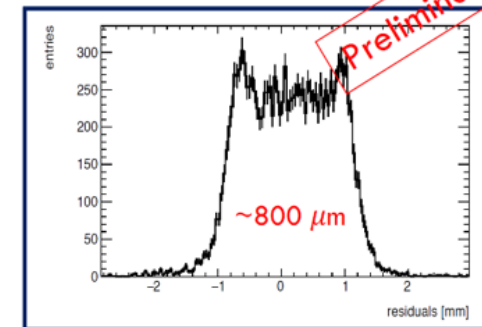
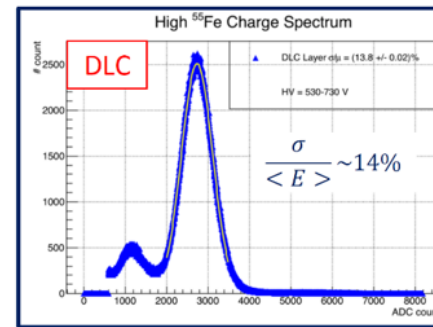
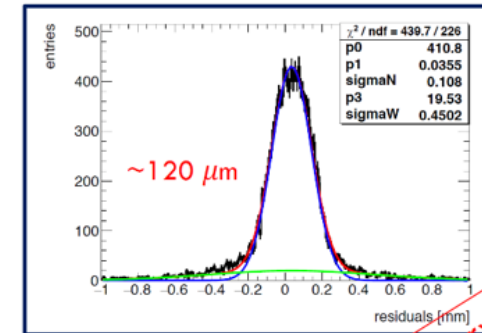


✓ High rate capability (>1MHz/cm<sup>2</sup>)

G. Morello, RD51 Mini Week, 12-Dec-2017



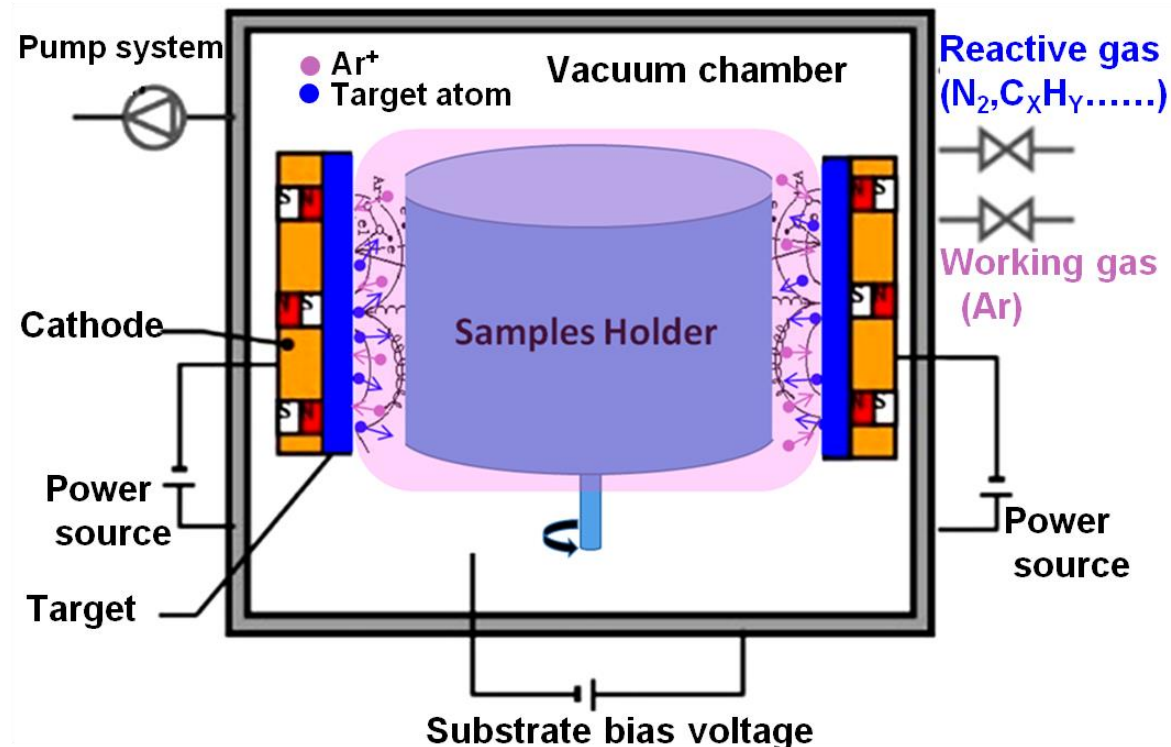
DLC



- ✓ Very good position resolution and energy resolution
- ✓ No charging up effects anymore

M.Iodice, ICHEP 2018, Seoul, 7-July-2018

# Hybrid Physical-Chemical Vapor Deposition (HPCVD)



- *Can be Deposited by chemical reaction or magnetron sputtering (or together)*
- *Low deposition temperature, high bonding strength, high deposition rate*
- *Pure DLC, Cr, Cu are deposited by magnetron sputtering*
- *Hydrogen doped DLC (a-C:H) is deposited by graphite targets sputtering and hydrocarbon gas dissociating at the same time*

***A common and flexible method for DLC deposition***

# The DOCA measurement

Data on this page comes from Resistive DLC Collaboration

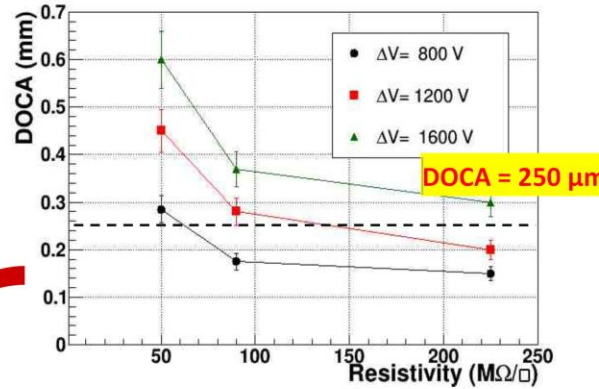
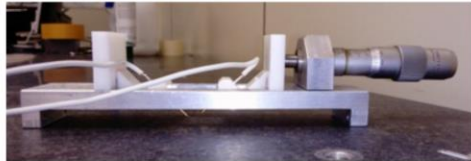
## ➤ For better understand of the safety distance



### Conductive Grid: optimization



In order to reduce the dead area, we studied the Distance Of Closest Approach (DOCA) without discharges between two tips connected to an HV power supply. We recorded the minimum distance before a discharge on the DLC occurred vs the  $\Delta V$  supplied for foils with different surface resistivity



DOCA = 250  $\mu m$

$\rho > 60 M\Omega/\square \rightarrow DOCA < 250 \mu m$

## Conclusions:

M. Poli Lener, MPDG 2019 - La Rochelle, 10/05/2019

28

- 250 $\mu m$  is safe enough for resistivity larger than 60 $M\Omega/\square$ ;
- More systematically measurements should be done in future;

-DOCA distance varies on the samples (Distance Of Closest Approach)  
 -between 1.0-0.1 mm  
 -DLC 60M  
 -11 samples per row

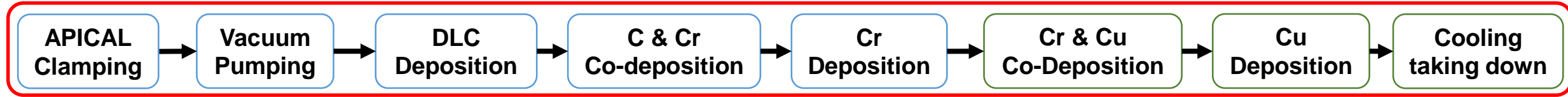
### Discussion on DOCA

- First surprise : the voltage to reach instabilities (up to 800V in air)
  - We were expecting 650V/670V for a 50 $\mu m$  gap
- After 30 sec with a limitation to 30nA we can already observe a voltage drop
  - It stabilize at voltages between 550V to 650V
  - An average current of 30nA per hole means 15mA for a 10cm x 10cm detector
  - This current is too high and not realistic.
  - We need to repeat the test with lower currents.
- We aren't able to define how many "low energy sparks" are created.
  - We would like to study the current peaks with a fast oscilloscope
- No real difference from the different DOCA with 60M DLC



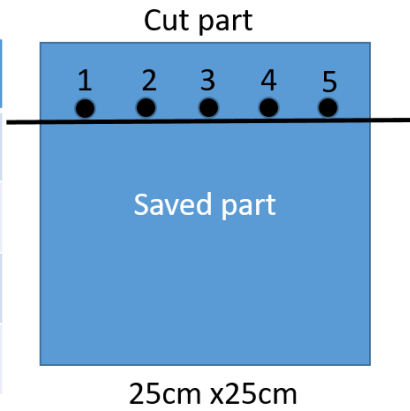
# Increasing the Adhesion by “One-Batch” Method

## Flow chart of the DLC + Cu by “One-batch” method

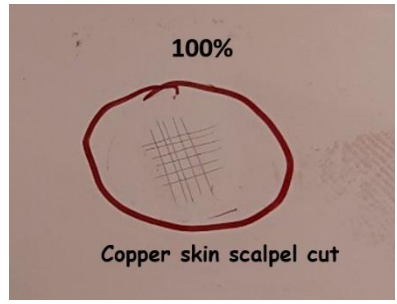
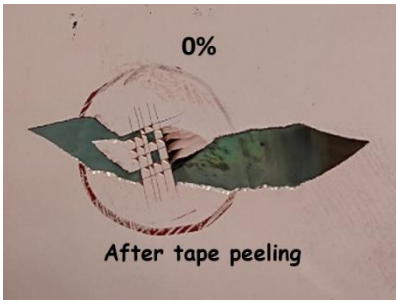
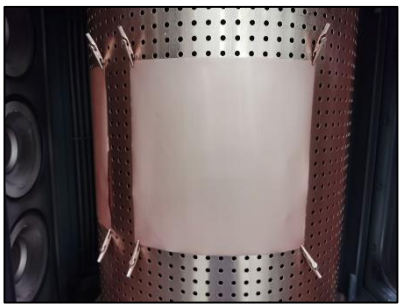


## Adhesion Test

Batch No.	Adhesion1	Adhesion 2	Adhesion 3	Adhesion 4	Adhesion 5
H2020-06-28-3	100	100	100	100	100
H2020-06-28-3	100	100	100	100	100
H2020-06-28-4	100	100	100	100	100
H2020-06-28-4	100	30	30	60	60

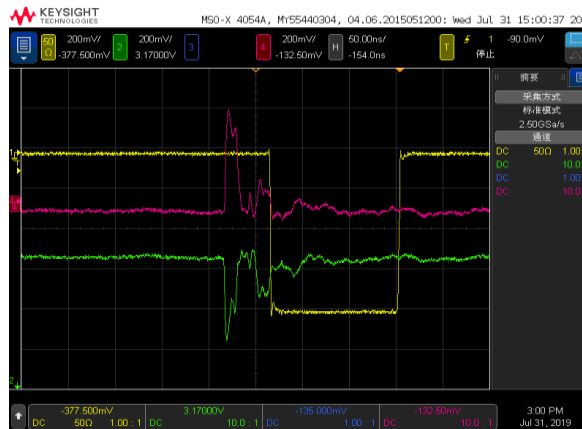
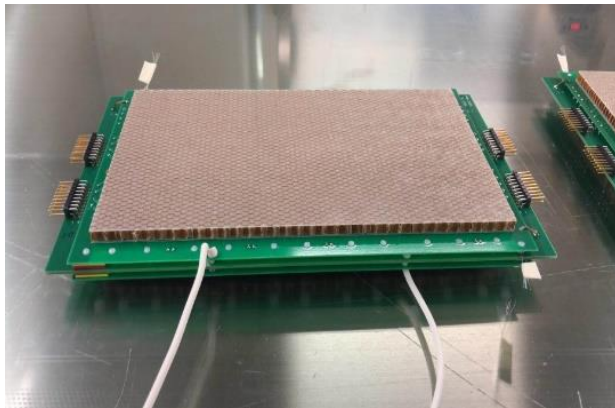
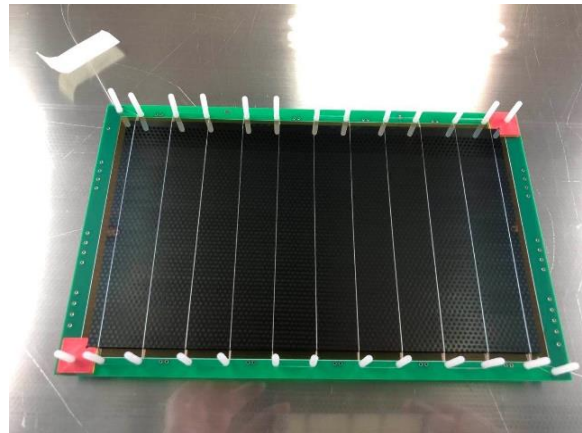
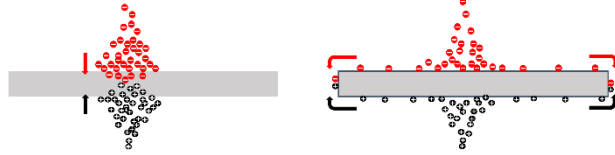


- “One-Batch” method shows very good adhesion, almost the same as the Cr/APICAL of the raw material;
- The 4<sup>th</sup> sample is not perfect, the possible reason is the surface is not clean and the PSE process is missing;
- This method is “blind” to the DLC resistivity, maybe it is (only?) suitable to the very low resistivity application;

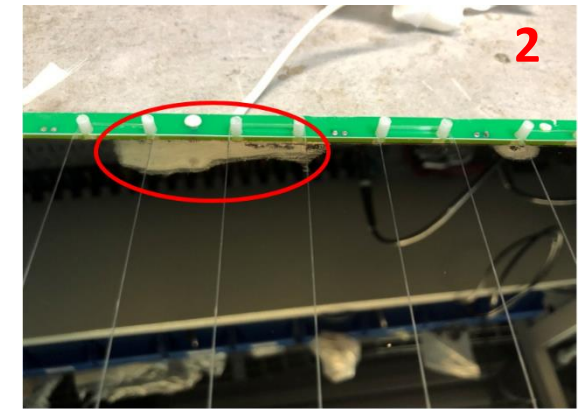


# DLC on glass—High rate MRPC

## Using DLC to speed up the charge neutralization in MPRC



## Problems found in current DLC-MRPC



### Challenges:

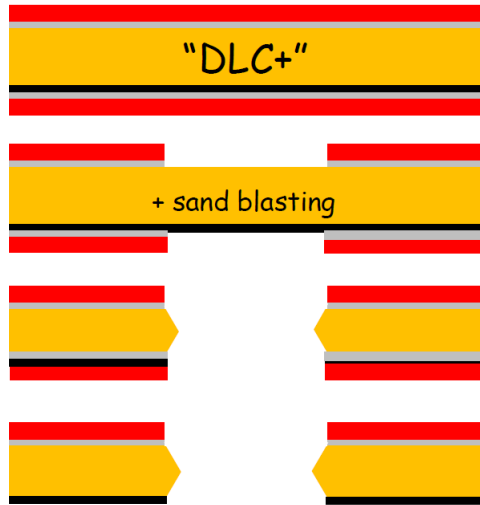
1. Large DLC inner stress compared to the other substrate;
2. Worse adhesion;
3. Difficult to coat on the side edge due to super large roughness
4. Can be removed by alcohol when use high temperature deposition

### Possible solutions:

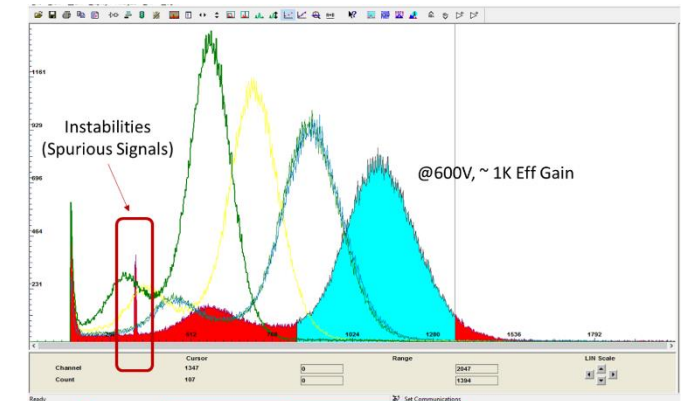
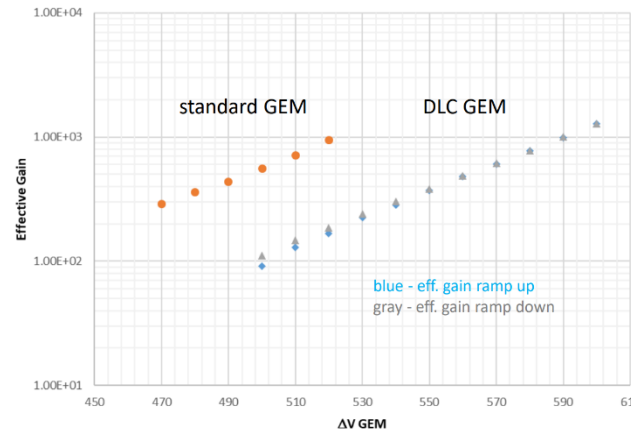
- Try to make the edge more smooth;
- Try to add some interlayer based on Silicon/Silicon compound;

More details can be found in:  
<https://agenda.infn.it/event/19942/contributions/108461/>

# Resistive GEM



## GEM with Resistive DLC Electrode on Bottom side

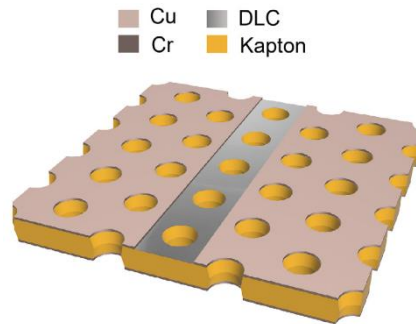


## Problem:

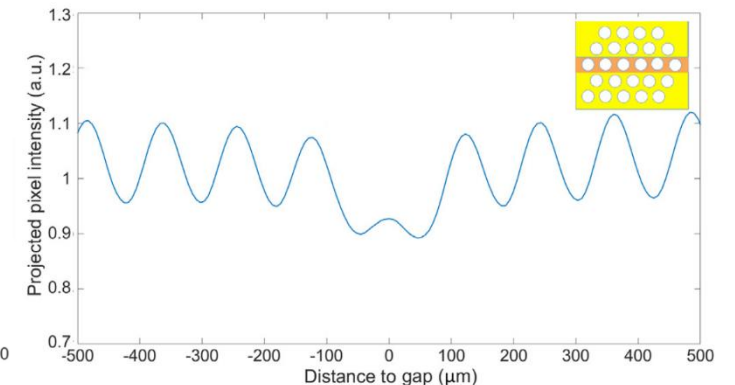
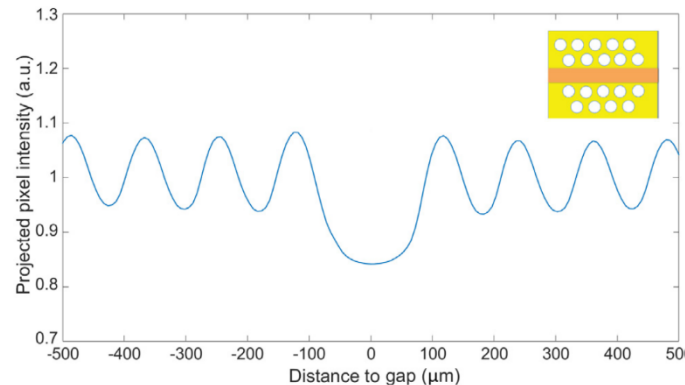
Sandblasting may cause the delamination

## Possible Solution:

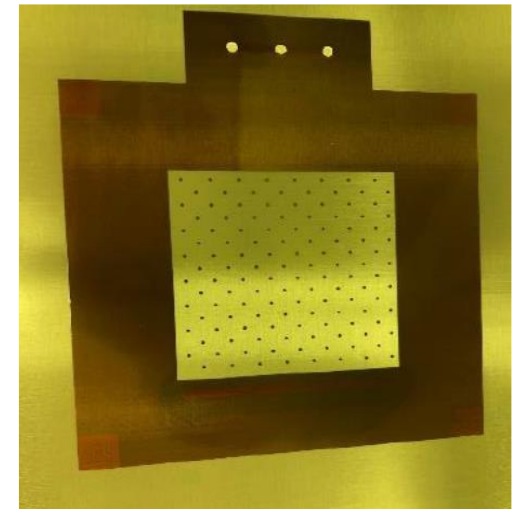
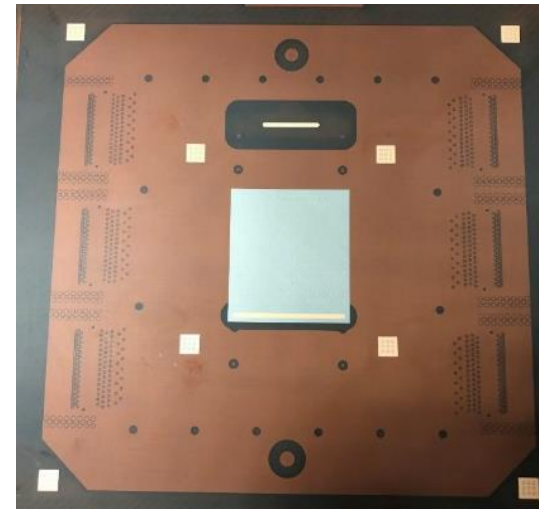
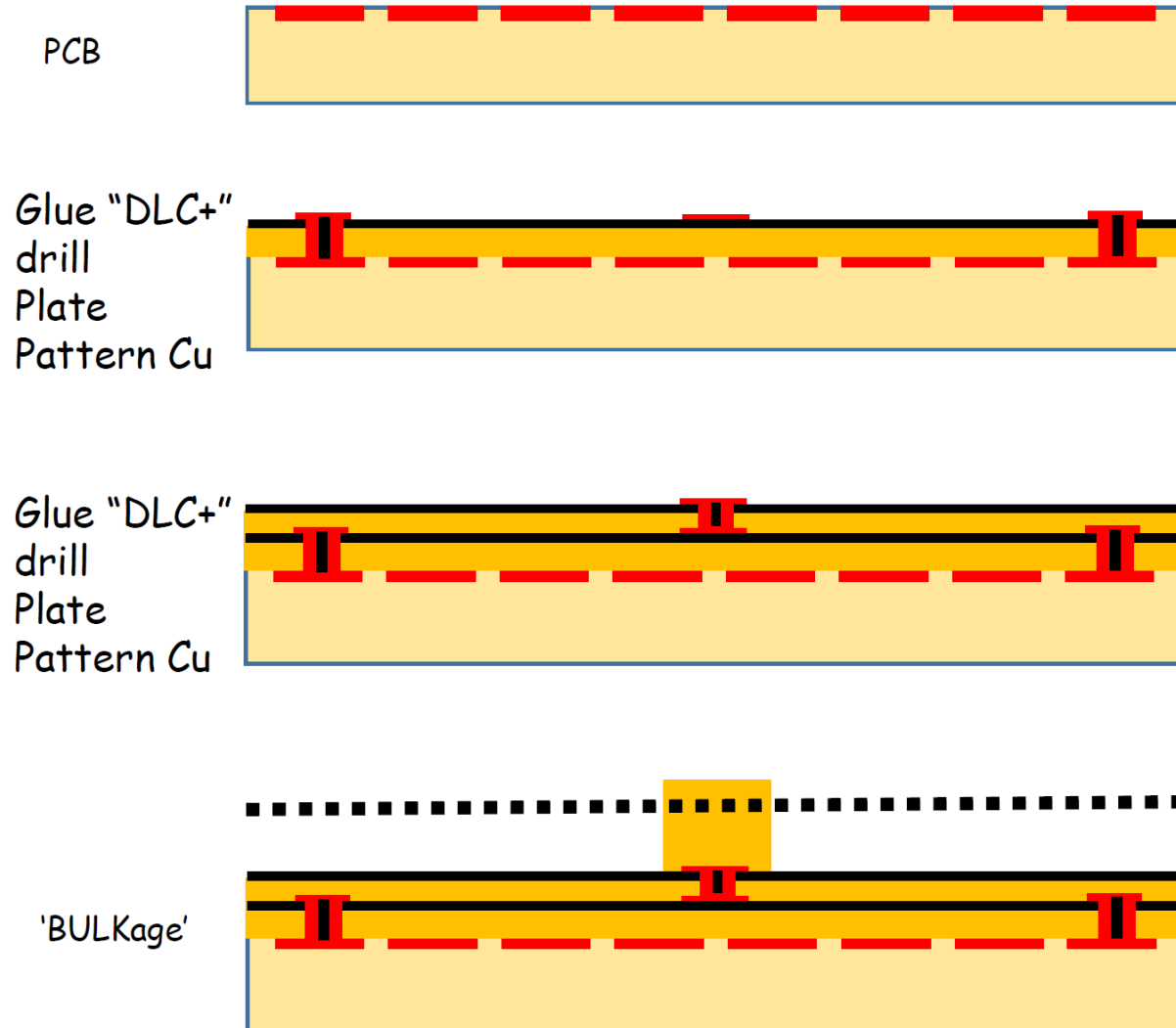
Keep the photoresist on Cu during the Sandblasting process



## GEM with Holes between the Sectors



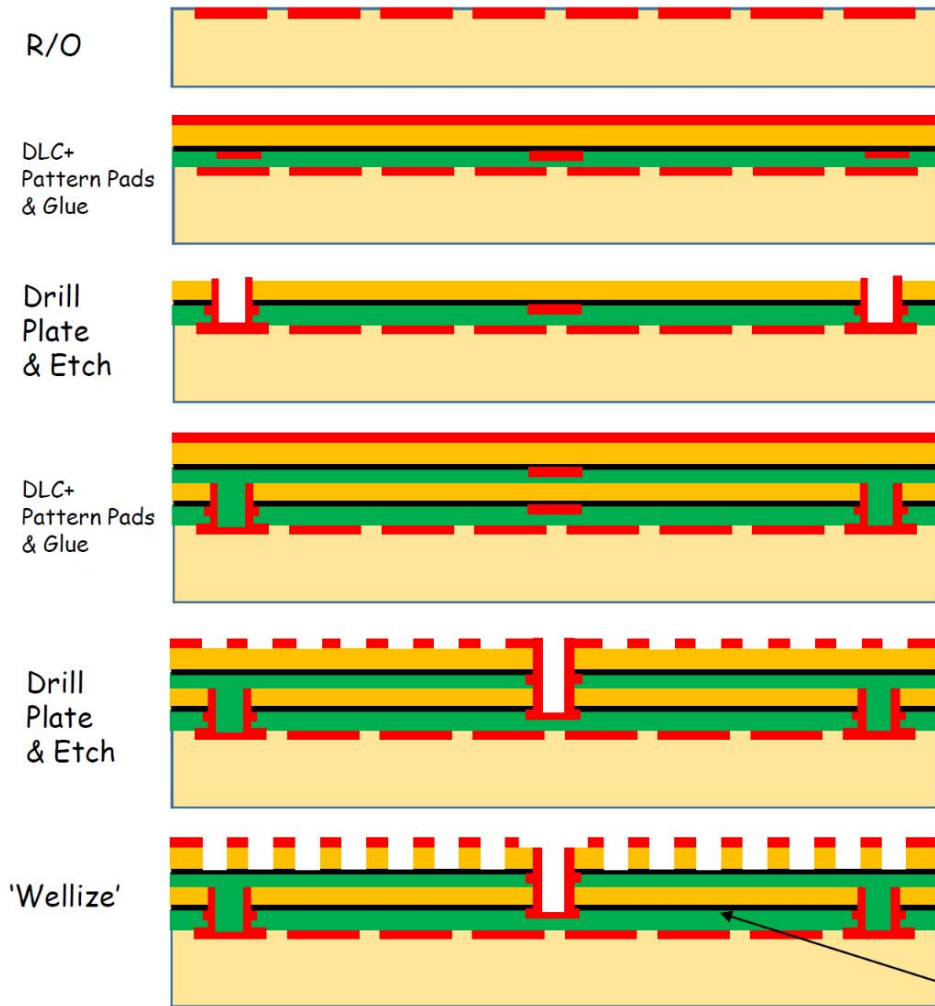
# Micromegas Made by Sequential Build-Up (SBU) Technique



- Extra Large DOCA(3mm)
- Adjustable evacuation density VS rate
- No problem with layers registration
- Good energy resolution
- 100% compatible with STD PCB processes
- Needs "DLC + Cu"

# $\mu$ RWELL Made by Sequential Build-Up (SBU) Technique

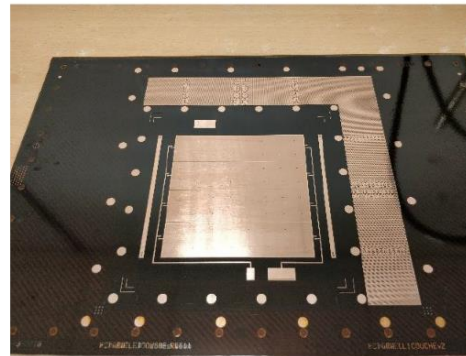
## SBU type Sequential Build Up



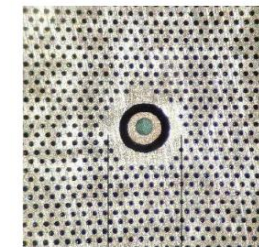
- Extra Large DOCA
  - Adjustable evacuation point density VS rate
  - 100% compatible with STD PCB processes
- Needs DLC+ base material

### Problem:

When the detector goes to large area, the deformation of the APICAL foil will be significant and cause alignment problems when drilling holes

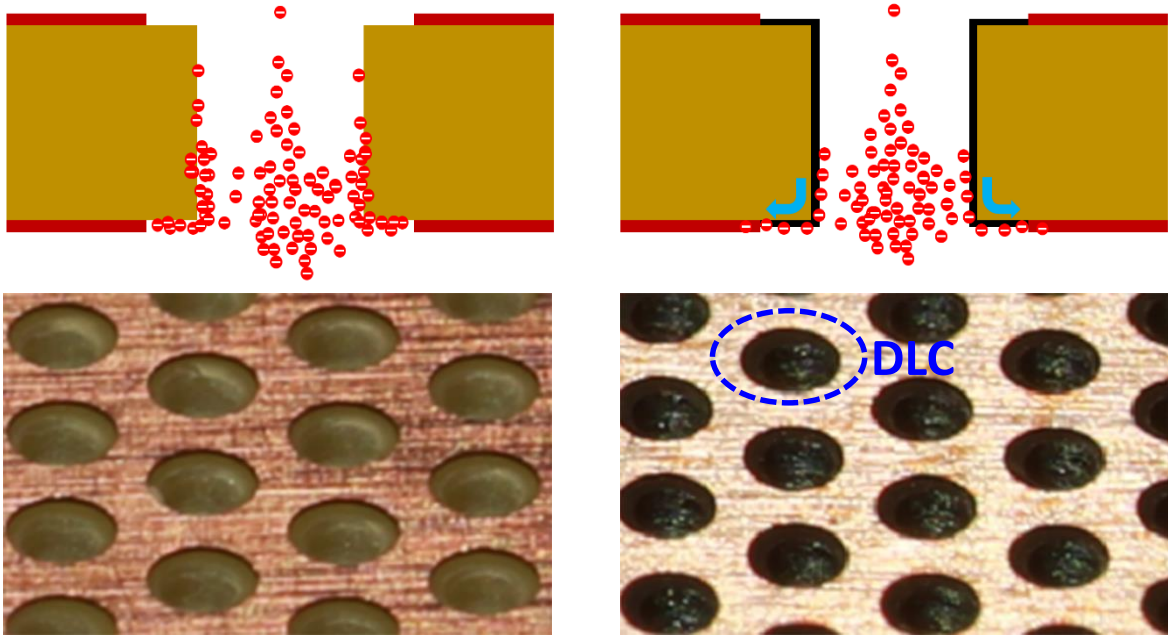


DOCA : 6mm



0.7mm

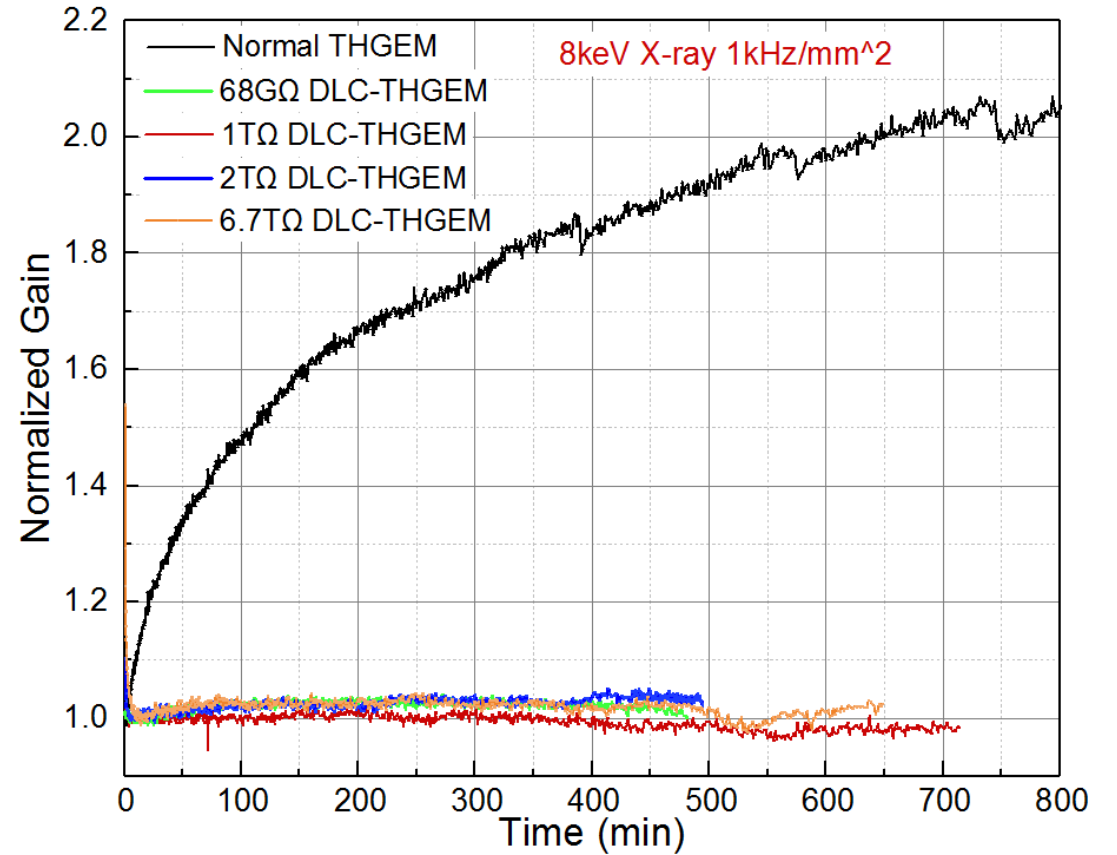
# Remove Charging-up of THGEM by High Resistivity DLC



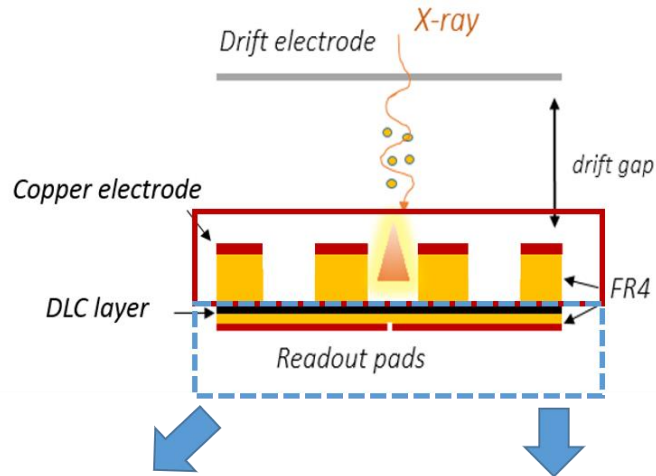
**Coating  $\alpha$ -C:H DLC on the rim and hole area**

**Doping hydrogen by adding isobutane from 7sccm to 9sccm**

- ✓ Easily applied on the current THGEMs;
- ✓ Charging up effect almost removed;



# RWELL Detector Using Separating Readout PCB

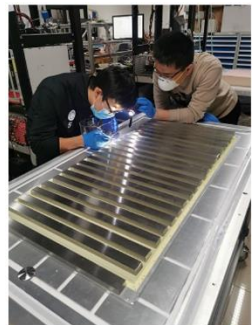
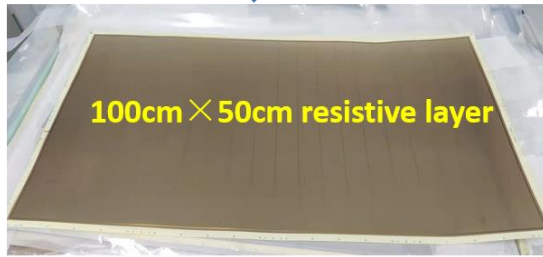


## Design methods:

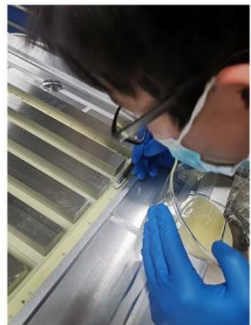
- Divide RWELL foil into 20 sectors
- Separate resistive layer from readout PCB
- Grounding circuit was designed on the resistive layer
- Large readout PCB was composed of 8 pieces



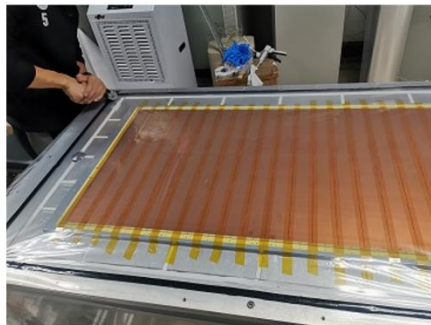
Gluing



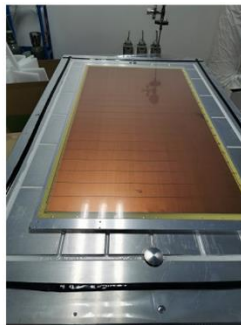
Painting the glue



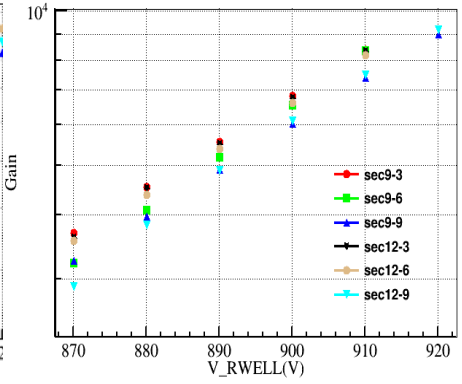
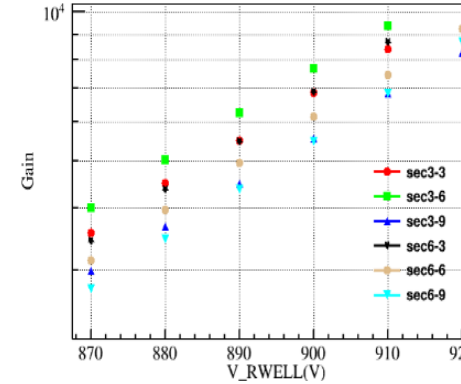
Seal the platform



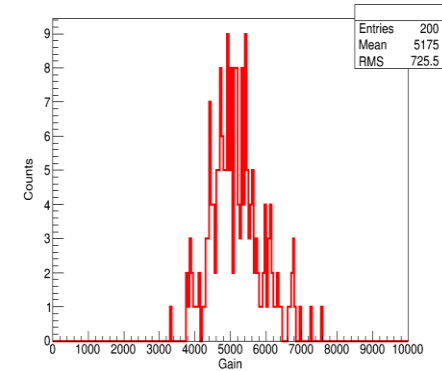
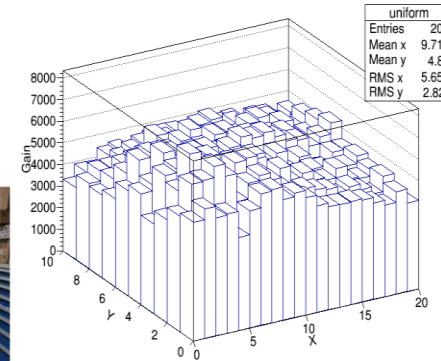
Pumping and drying



Assembling



Maximum Gain : ~8000



Gain uniformity : ~14.0% @  $G_0 = 5175$