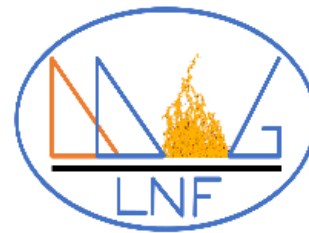


DLC Community Contributions from RD51 Common Project

Yi Zhou

On behalf of the Resistive DLC Collaboration



RD51 Common Project

DLC based electrodes for future resistive MPGDs

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

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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*

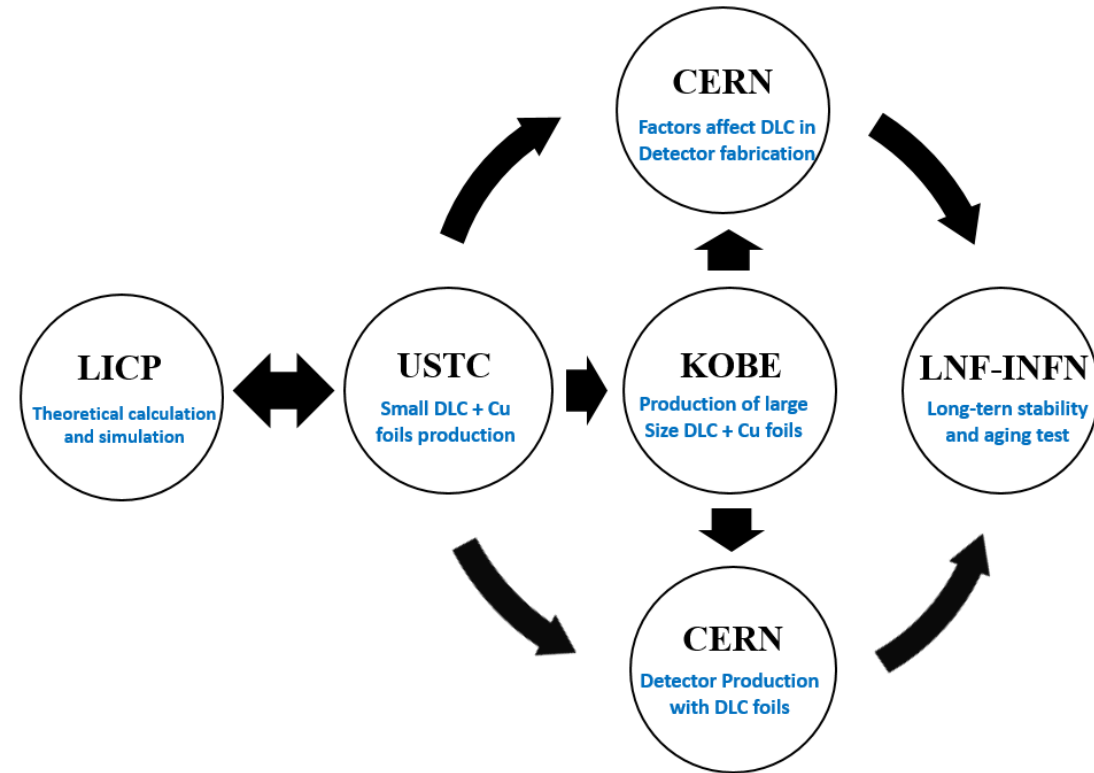
2. *Kobe University, contact person: Atsuhiko Ochi e-mail: ochi@kobe-u.ac.jp*

3. *CERN contact person: Rui de Oliveira e-mail: Rui.de.Oliveira@cern.ch*

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

Ext. Collaborators:

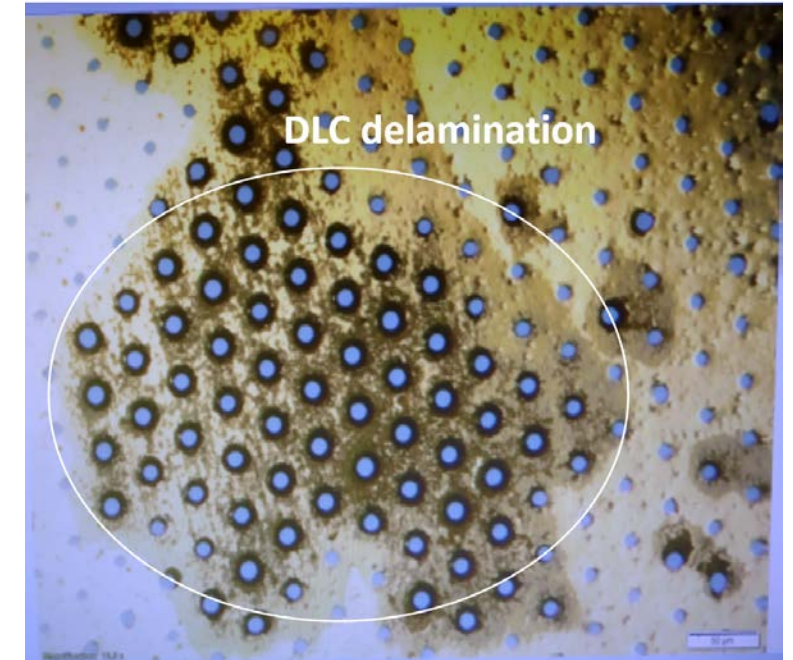
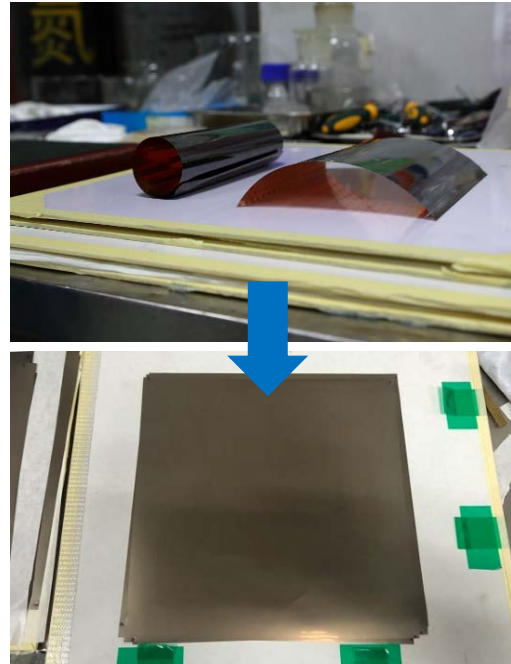
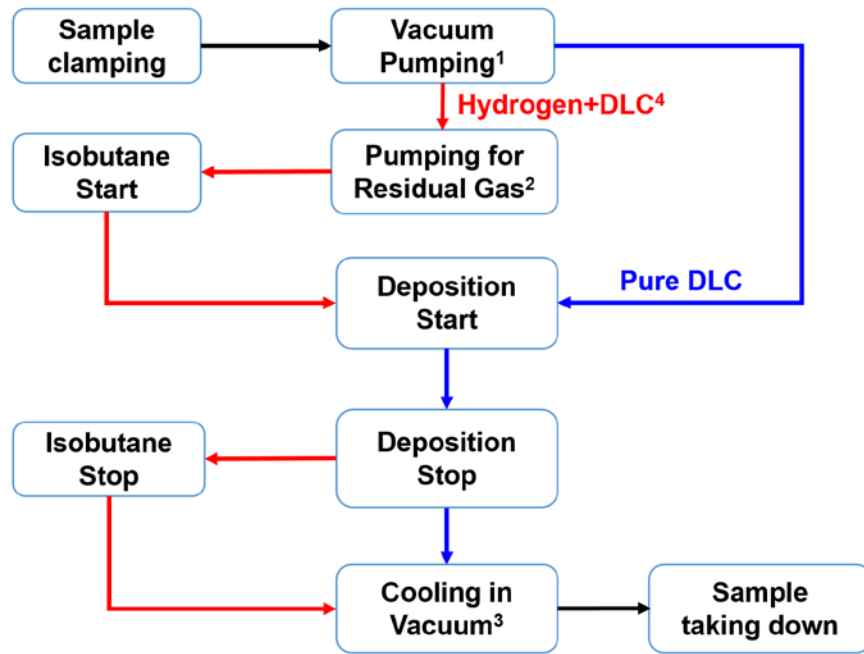
1. *State Key Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Chinese Academy of Science contact person: Lunlin Shang e-mail: shangll@licp.cas.cn*



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.

Preparation of resistive DLC on APICAL



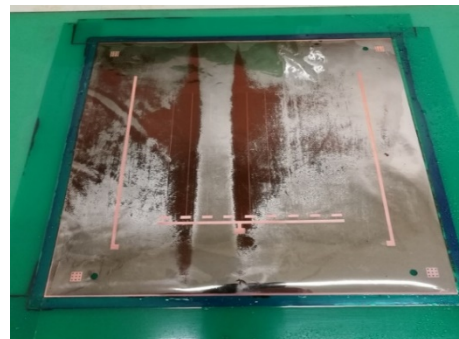
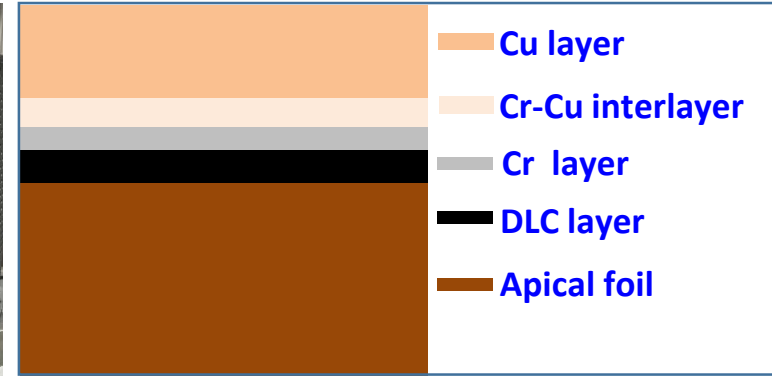
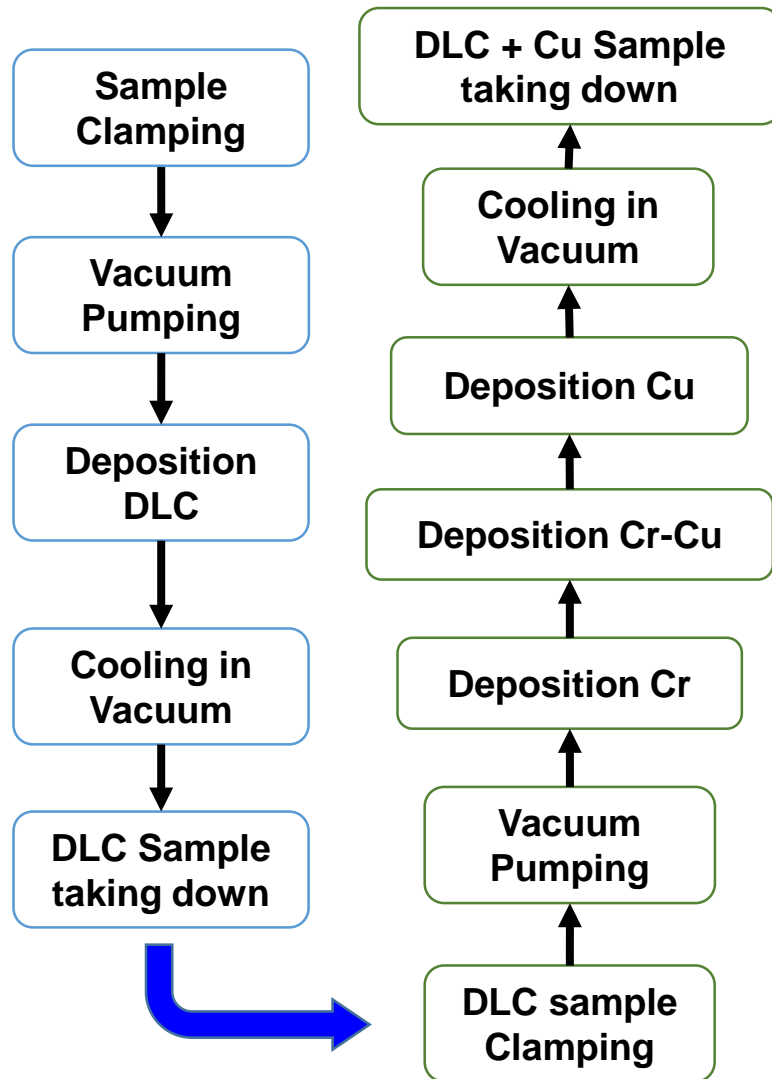
Challenges

1. Inner stress caused the bending of the APICAL
2. How to control the resistivity;
3. Adhesion between the DLC/APICAL;

Solutions

1. Low bias voltage, high roughness of APICAL;
2. Vacuum degree, thickness, doping;
3. High roughness of APICAL;

Preparation Cr/Cu on DLC



Challenges

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

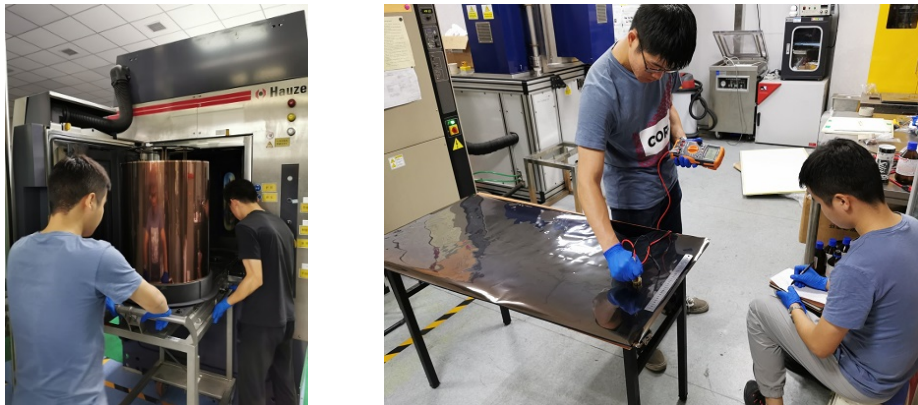
Solutions

1. Optimize the thickness of Cr layer, add Cr-Cu co-deposition layer, high bias voltage, high temperature, add DLC-Cr co-deposition layer, one-batch coating;
2. Optimize the thickness of each metal layer;
3. Extra Cu coating by galvanic method;

Current capacity of the sample production

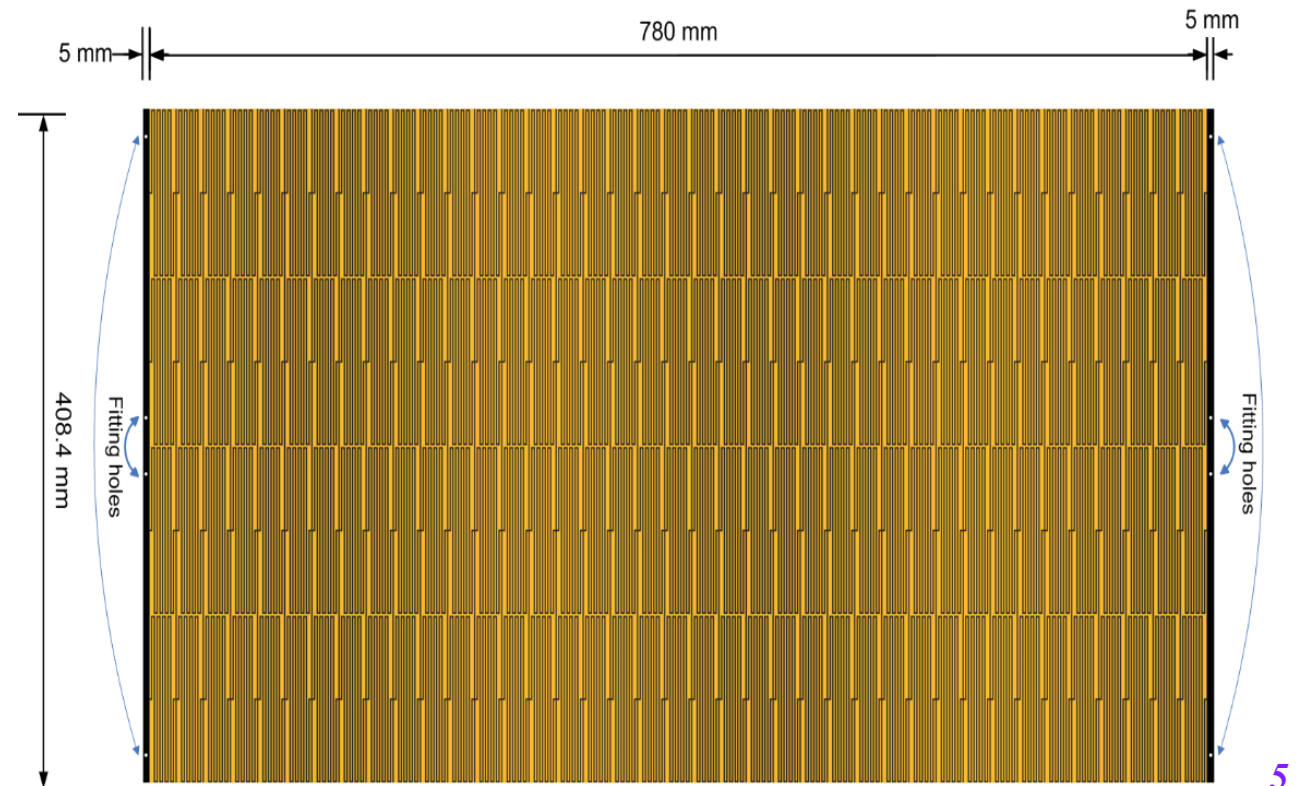
LICP & USTC

- 25cm × 25cm DLC/DLC+Cu, coat DLC 1 per batch(80min), then coat Cr/Cu 5 per batch(9h);
- 120cm × 60cm DLC/DLC+Cu, one by one (9h),
- High temperature(300°C) deposition (5h/11h);



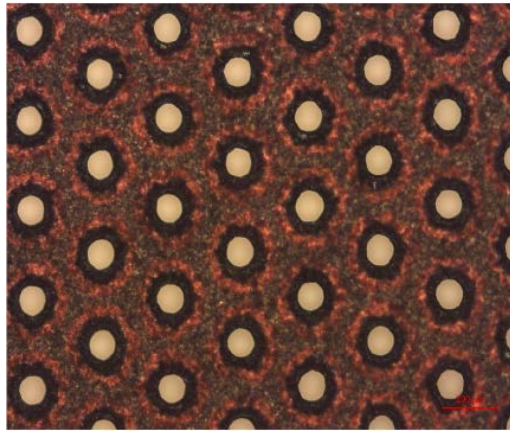
Kobe & Be-Sputter

- L × 60cm DLC samples (L > 100cm);
- Patterned(Lift-off) DLC foils with large size;
- Small size samples, many pieces in one batch;



Resistive GEM and μ RWELL-type FTM

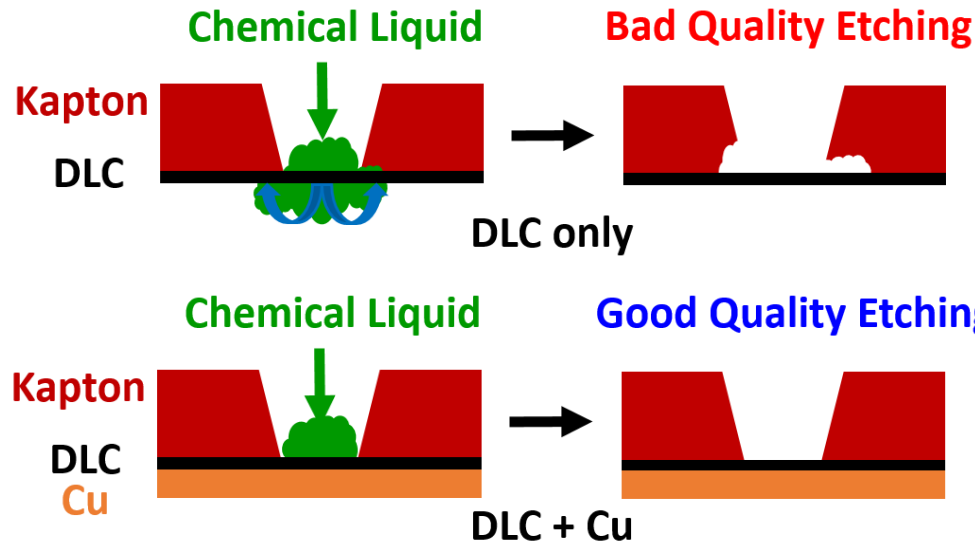
Resistive GEM



FTM



DLC+Cu improves the etching quality



Challenge:

There are cavities inside the sputtered copper which leads the copper can't perfectly protect the APICAL during etching process;

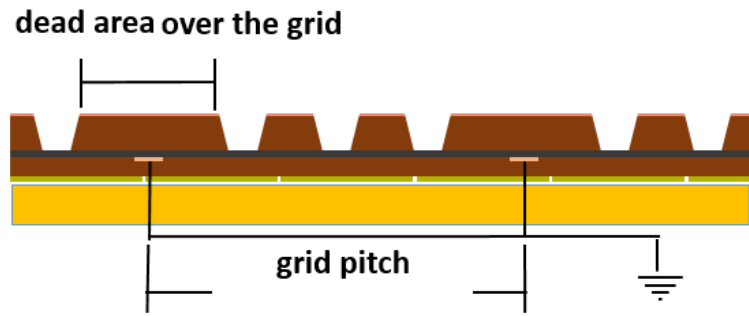
Solution



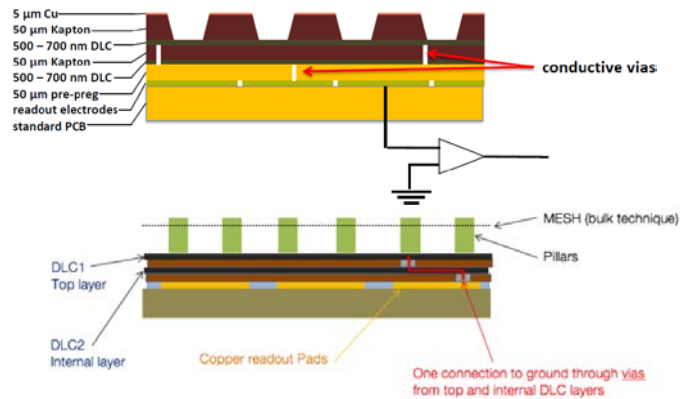
- Galvanized Cu
- Sputtering Cu
- Apical
- DLC
- Original Cu

High rate μ RWELL and MICROMEAS

Fast grounding μ RWELL



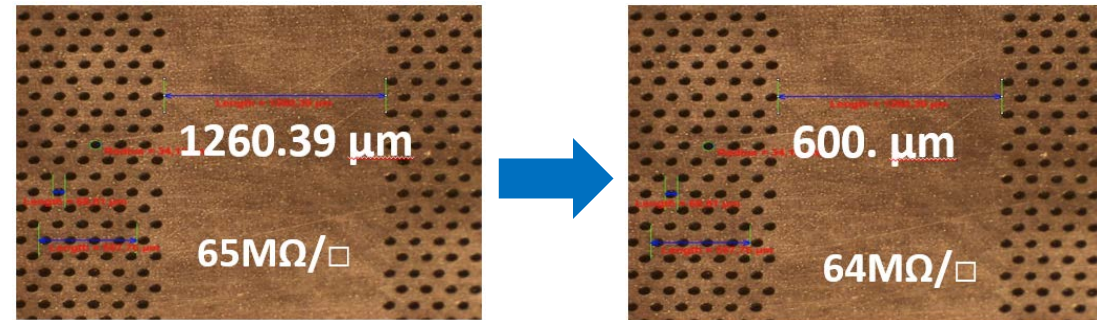
Double-layer μ RWELL/Micromegas



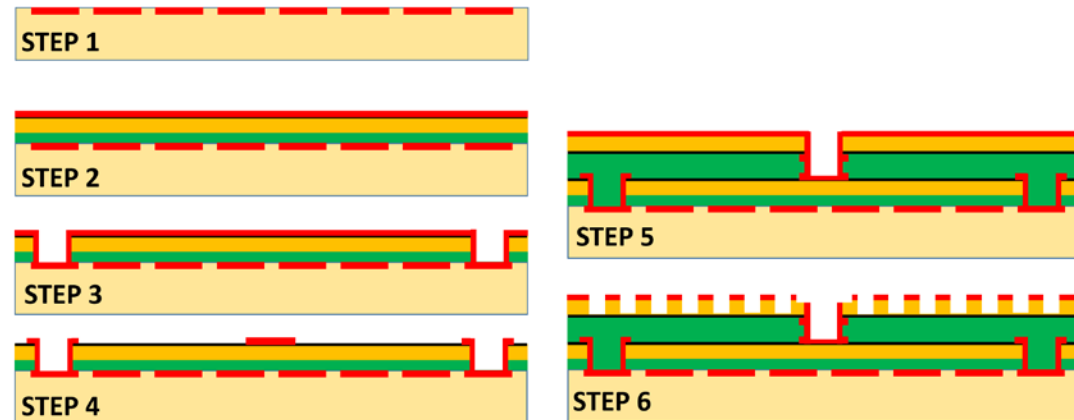
Challenge:

1. Resistivity decreasing after press gluing;
2. Cr/Cu delamination during drilling/etching;

DLC+Cu can decrease the dead area



DLC+Cu can simplify the manufacture process

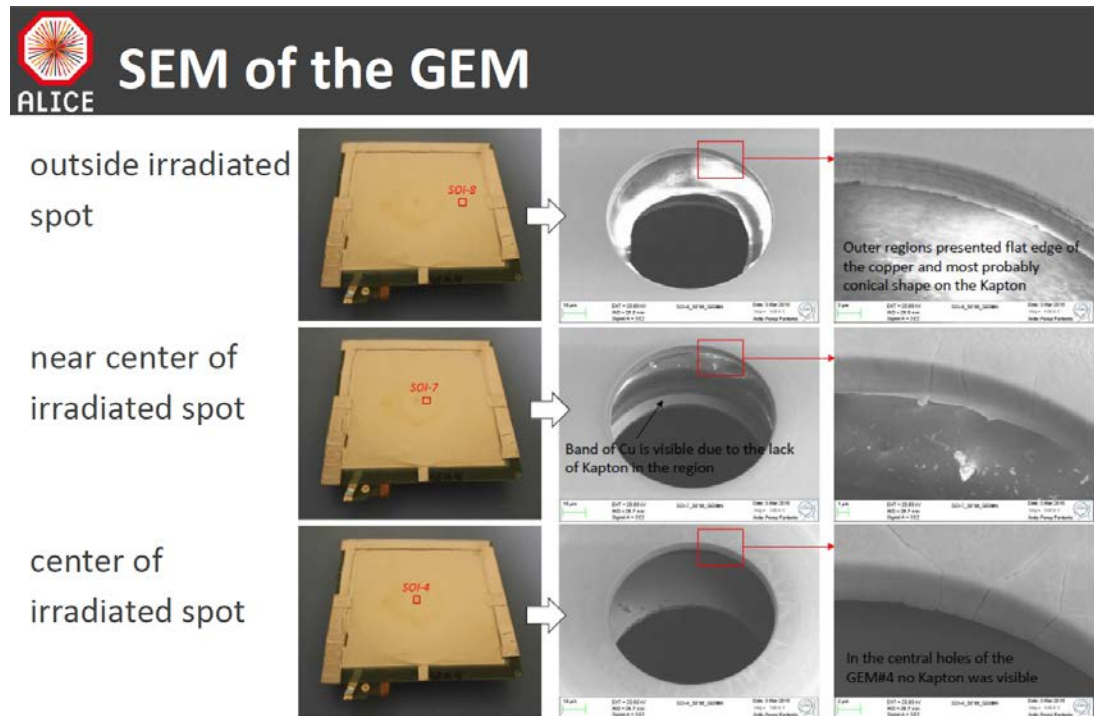


Solution:

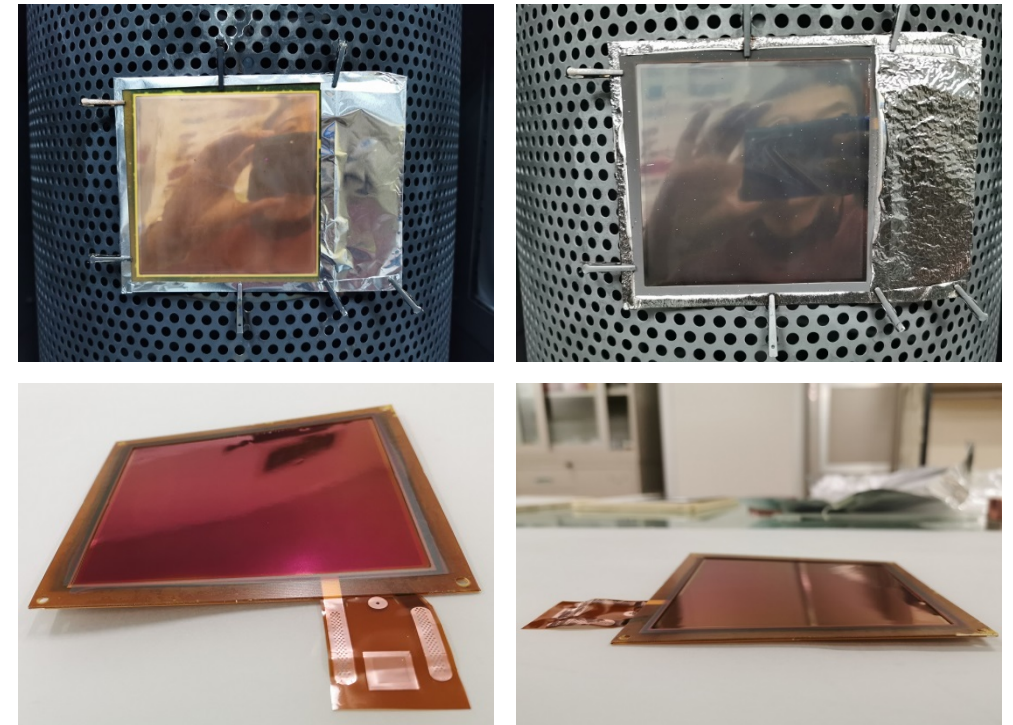
1. Make larger resistivity at the beginning as the compensation;
2. Improve the adhesion of the Cr/DLC;

GEM with thick-DLC (very high resistivity) in Holes

Kapton Etched during GEM operation



Coating DLC in holes to protect the Kapton



Challenge:

Large inner stress caused the foil bending

The foils are waiting for test and we are looking for a way to decrease the inner stress

Conductive DLC for low mass GEM

➤ By applying high vacuum, large thickness and element doping, we can greatly decrease the resistivity of the DLC

Batch No.	Substrate	Vacuum (10^{-5} Torr)	Current(A)	Cr & Cu Current(A)	Time(min)	Resistivity (Ω/\square)
8-27-06	FR4	1.4	3.5	0	80	180k
9-01-01	FR4	1.4	3.5	0.3	80	350
8-24-01	APICAL	0.06	2.8	0	30	36k

Challenge:

1. Etching though the thick DLC is not perfect yet;
2. In some applications(X-ray fluorescence analysis), metal doping is not a good way;

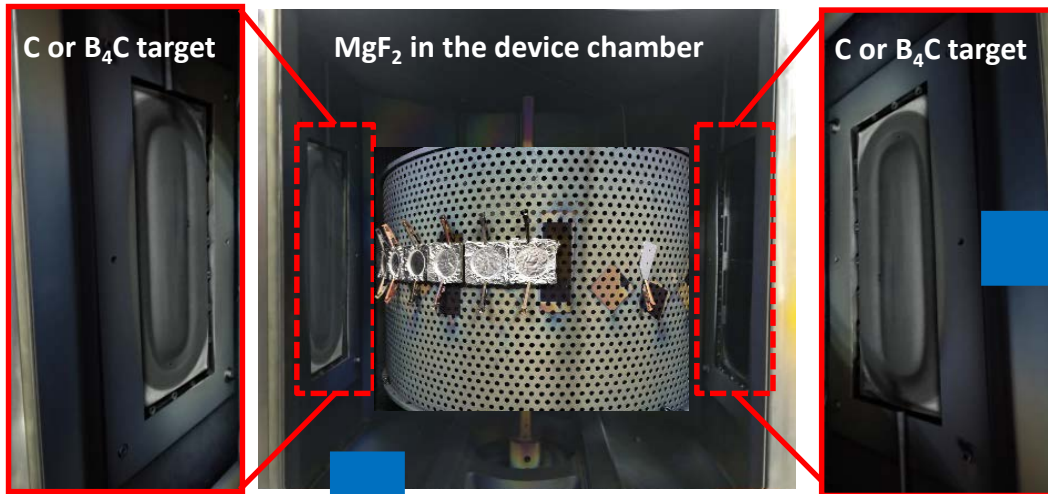
Possible solution:

1. Improving the etching process;
2. Try to doping nonmetal element: Nitrogen, Boron;
3. High temperature coating;

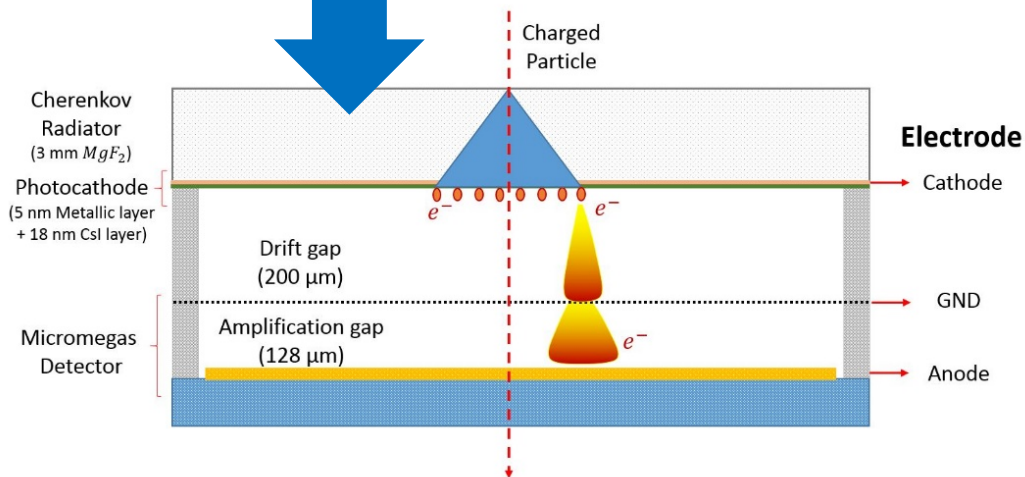
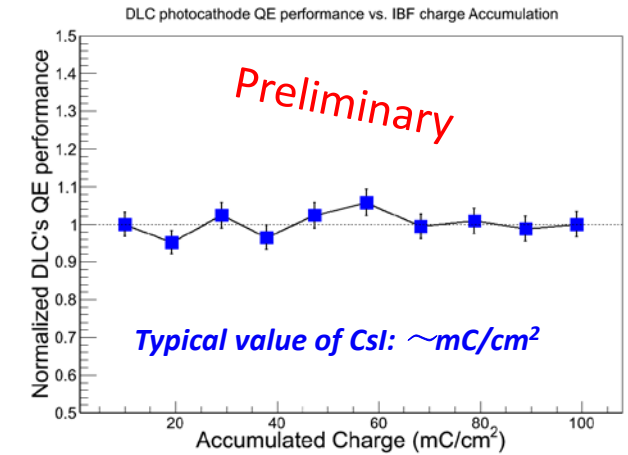


DLC on MgF₂—Robust photocathode

DLC 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



Challenges:

- Optimization of thickness;
- Optimization of the *sp*³/*sp*² ratio;
- Optimization of the energy band;
- Surface treatment (No hydrogenation);

Solutions:

- Thickness scan;
- PLD, Cathodic arc, and other method;
- Boron doping;
- Metallic oxide deposited on surface by ALD (1st attempt was failed...); 11

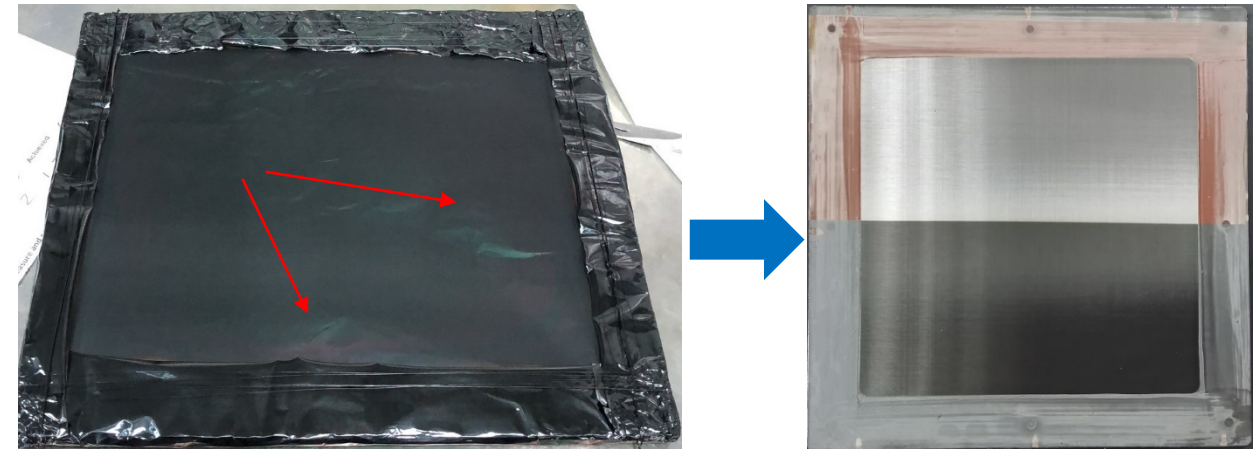


DLC on stainless steel mesh—Black mesh

Black mesh for MICROMEGAS with optical readout (IRFU/DPhN, CERN, USTC, LICP)



Color of different Hydrogen doping



Advantages of the black mesh: Less reflection photons

- In detector manufacture: less reflected UV light and provide a shaper edge between exposed & non-exposed coverlay, then the etching chemical remnants in amplification zone will be lessened, and this will yield a more stable MM operation.
- In application: less reflected visible light can decrease the defusion of the image captured by the camera, thus minimize the degration of image definition.

Challenges:

- Bad hydrogen doping will cause colorful colors;
- Much worse adhesion compared with the DLC on stainless steel disc ;

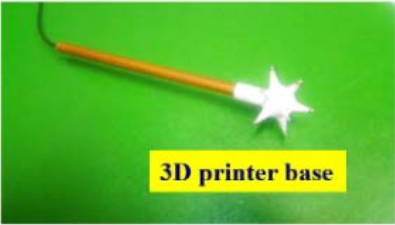
Solutions:

- Optimize the amount of hydrogen doping;
- Add DLC-Cr co-deposition layer;
- High temperature coating, PSE pre-process for mesh clean

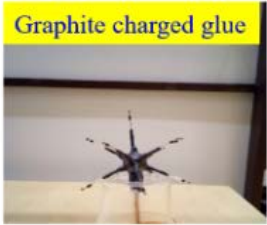
DLC on spherical resin—Resistive ACHINO

Resistive ACHINOs for the spherical detector


Multi-ball 'ACHINOS' structure
Developed in Saclay in collaboration with University of Thessaloniki



3D printer base




Graphite charged glue



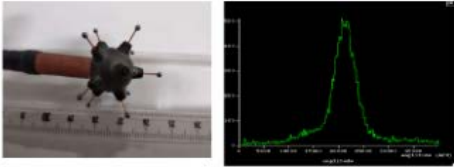
copper charged glue

Problem of robustness of charged glue:
At high voltages because of discharges in the bulk it becomes conductor!!!

↪ **DLC layer (collaboration with USTC)**
It is a fantastic technical solution:
Robust, stable, precise



resin nylon glass



<https://indico.cern.ch/event/843711/contributions/3607162/attachments/1930459/3197217/RD51-19.pdf>

Challenge: Uniformity is bad (but not important)

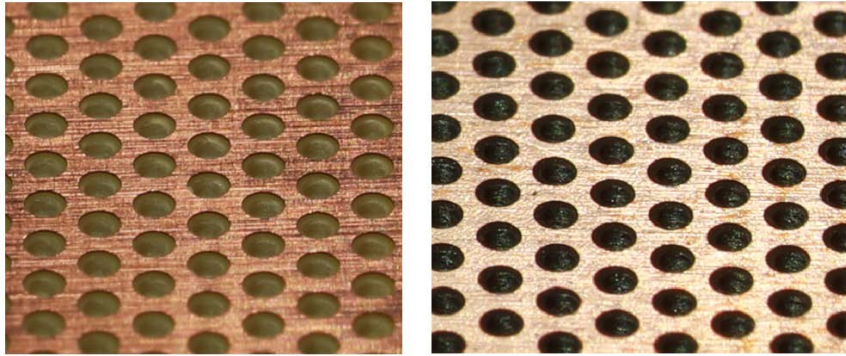
DLC coating



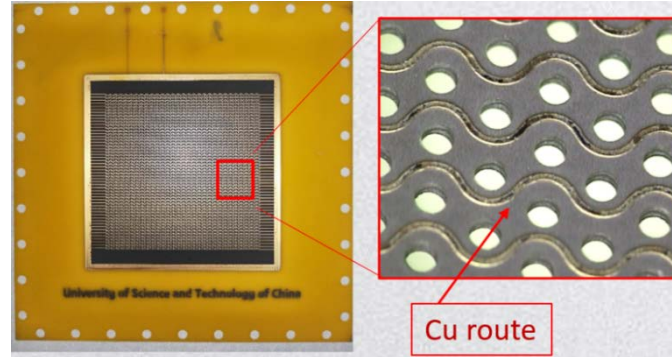
Solution: Multi-coating if necessary

DLC on PCB—RTGEM based detectors and μ -PIC

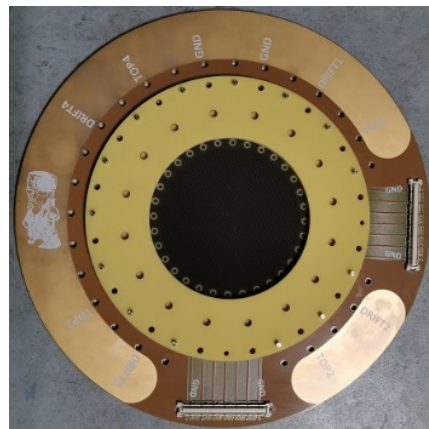
Charging-up free THGEM



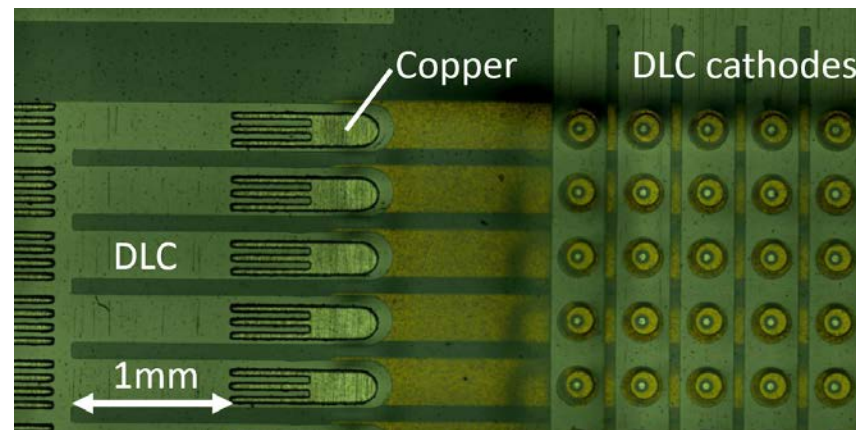
Resistive THGEM



RWELL-type FTM



μ -PIC



Challenges:

1. Difficult to coat DLC on copper;
2. Operating temperature should be below 200°C;
3. Multi-time lift-off takes very long time;

Solutions:

1. Use lift-off to coat Cr before DLC coating;
2. Try to avoid high temperature operating;

Resistivity decreasing caused by heating

- The DLC will be heated during the copper coating and detector manufacture

Resistivity decreasing of normal DLC+Cu

Batch No.	R1(MΩ/□)	R2(MΩ/□)	Ratio
8-26-3	150	3.8	39.5
8-25-4	150	27	5.6
6-27-5	115	7.2	16.0
6-27-3	80	7.5	10.7
8-25-7	150	20	7.5
8-25-2	100	20	5.0
8-26-1	160	28	5.7
8-26-6	240	43	5.6
8-26-7	140	18	7.8
8-25-5	140	25	5.6
8-24-6	200	34	5.9
8-26-4	120	33	3.6
8-25-4	150	54	2.8
8-24-5	180	32	5.6

I doubt the Cr is not fully etched on this sample!

Resistivity decreasing of low resistivity samples

Batch No.	R1(kΩ/□)	R2(kΩ/□)	Ratio
8-28-4	342	32.7	10.5
8-27-1	340	44	7.7

Resistivity decreasing of double-side samples

Batch No.	R1(MΩ/□)	R2(MΩ/□)	Ratio
8-29-9	1400	340	4.1
8-27-4	75	17	4.4
8-29-8	750	550	1.36
8-27-3	450	89	5.1

Unknown reason

To be checked systematically:

- If the resistivity decreasing varies with the roughness of the APICAL;
- If the resistivity decreasing varies with different resistivity value;

Calibration plan for resistivity decreasing

- We will use the vacuum oven to calibrate the resistivity decreasing under 200°C for 5 hours
- We can measure the same position with the same pressure before/after heating



Adhesion of Cr/DLC test and next work plan

➤ Delamination occurred during detector manufacture due to bad adhesion

Roughly Rank of the adhesion:

Base material :	→ reference 100 %	→ really good
DLC+Cu coating under 300°C:	→ 80% of the reference	→ good
Center of normal DLC+Cu:	→ 50%	→ medium, but good enough for fast grounding
Outer part of normal DLC+Cu:	→ 20%	→ not sufficient for processing

Current problem:

1. For the normal samples, the adhesion of the center area is better than it of the outer area;
2. For high temperature deposition (300°C), the resistivity is hard to control, and we don't know if it can be removed by alcohol (on glass, yes)

Possible reason:

1. The sample is fixed by 4 clamps on the 4 corners, these clamps changed the electric field and caused the different adhesion;
2. We have metal at the edge area, the edge effect caused the different adhesion;
3. High temperature deposition produced a lot of sp^2 structures inside the DLC;

Next work:

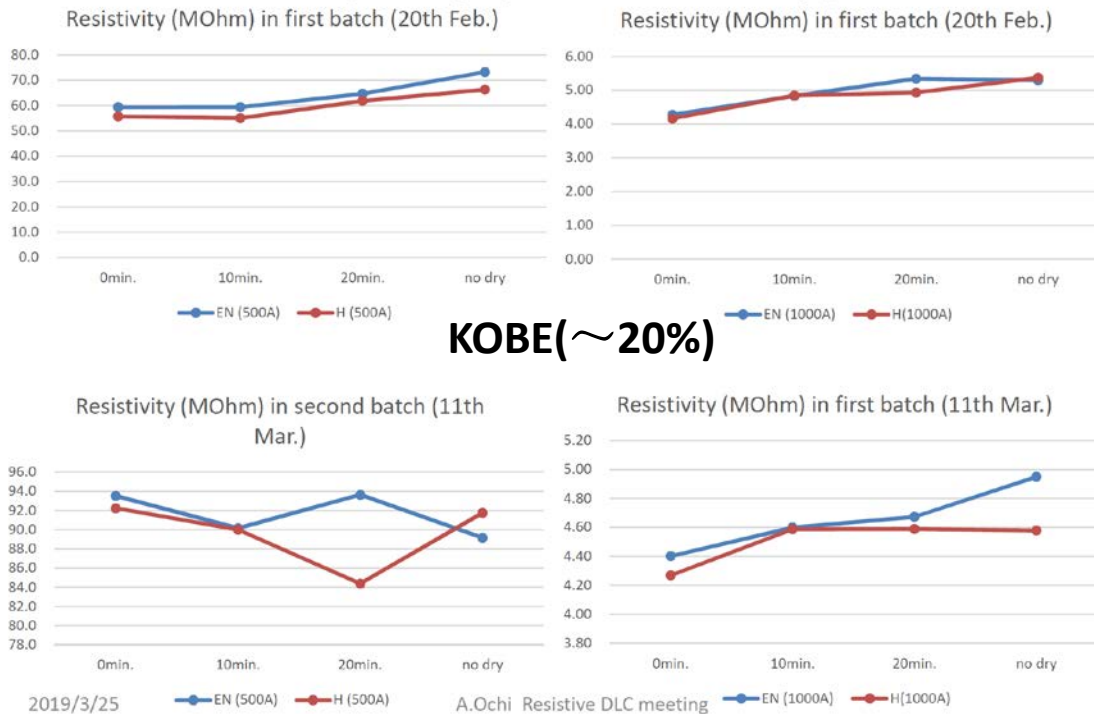
1. Try to use the Teflon screw to fix the sample then check the adhesion;
2. Use larger size samples to see if the worse adhesion area moves far away from the center;
3. We have to check if the alcohol can remove the high temperature DLC on APICAL;



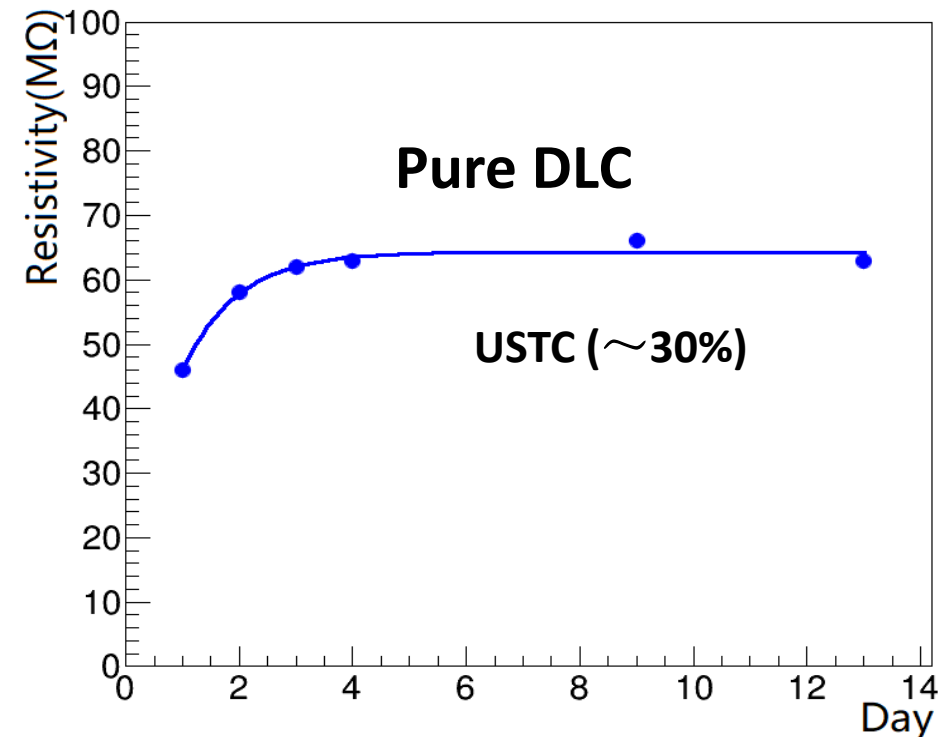
Passivation in air after sputtering

- 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.

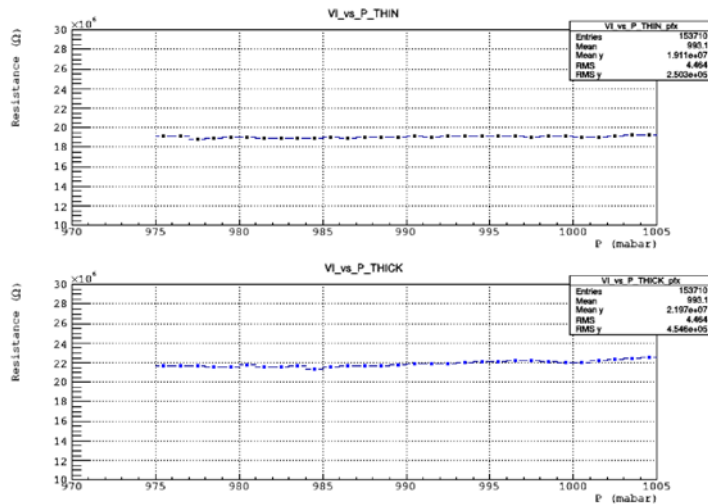


- DLC resistivity will increase in air after it was take out from the sputtering chamber;
- The test results are more or less compatible with USTC results;

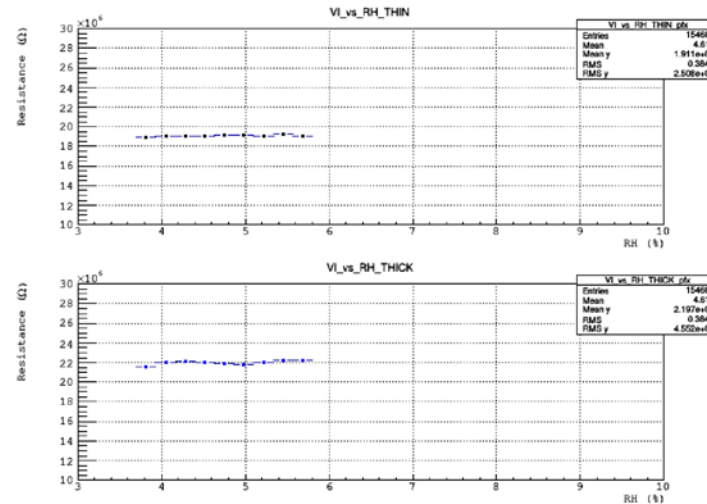
Stability in air

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

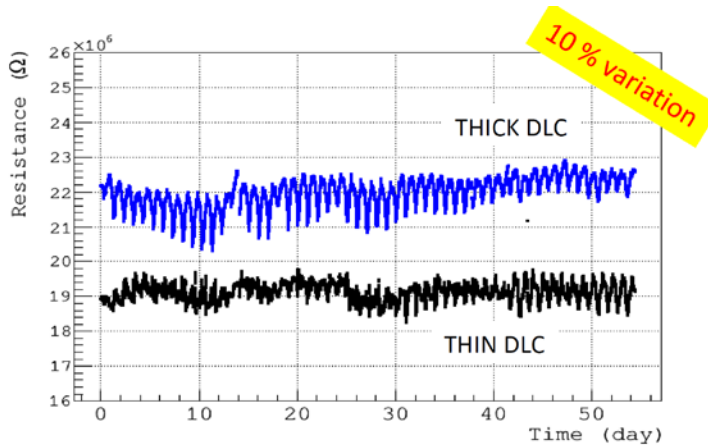
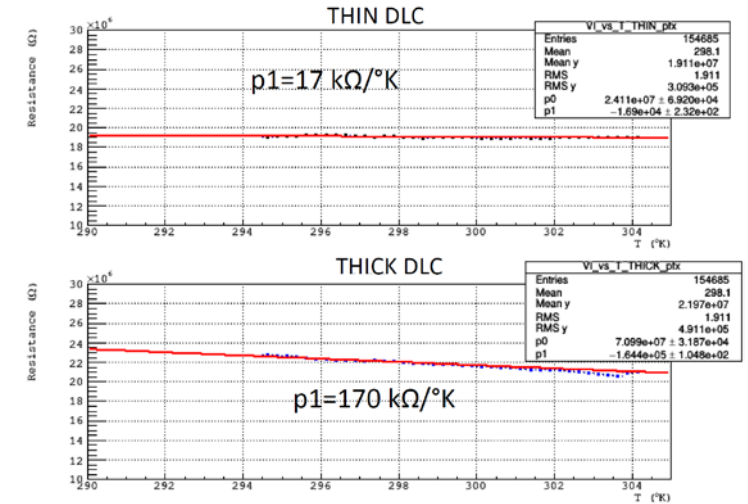
Resistive .VS. Pressure



Resistive .VS. Humidity



Resistive .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 DOCA measurement

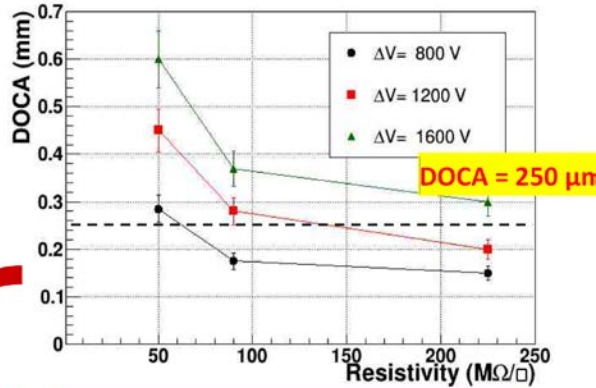
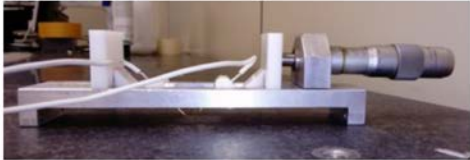
➤ For better understand of the safety distance



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 ΔV supplied for foils with different surface resistivity



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

Conclusions:

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

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- 250μm is safe enough for resistivity larger than 60MΩ/□;
- 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

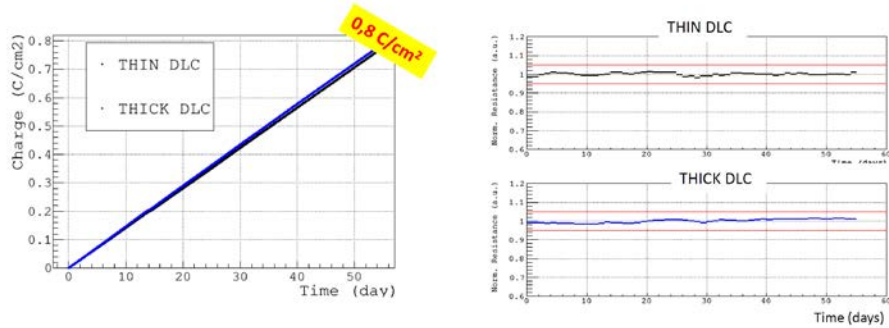
Discussion on DOCA

- First surprise : the voltage to reach instabilities (up to 800V in air)
 - We were expecting 650V/670V for a 50um 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

The aging measurement

➤ To make sure the DLC is radiation hard

DLC stability under current drawing

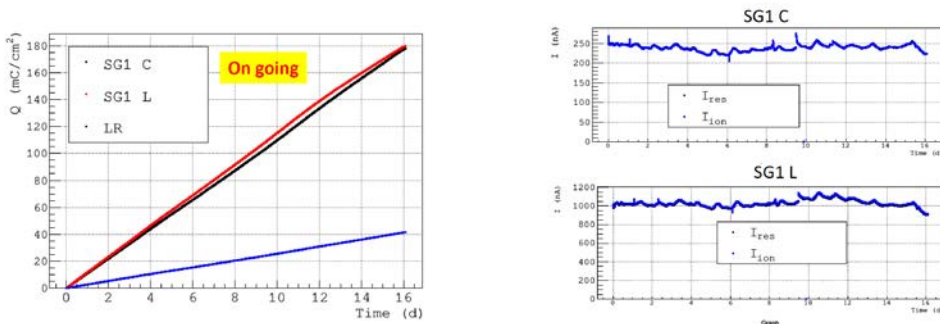


No effects observed after an integrate charge of $\sim 1 \text{ C/cm}^2$

Detector aging: X-Ray LNF



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

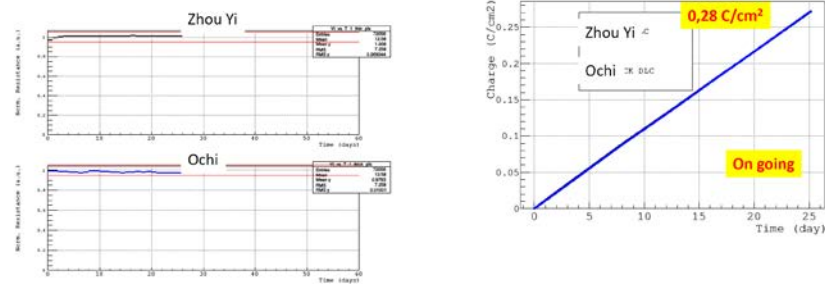


DLC stability under current & X-ray



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

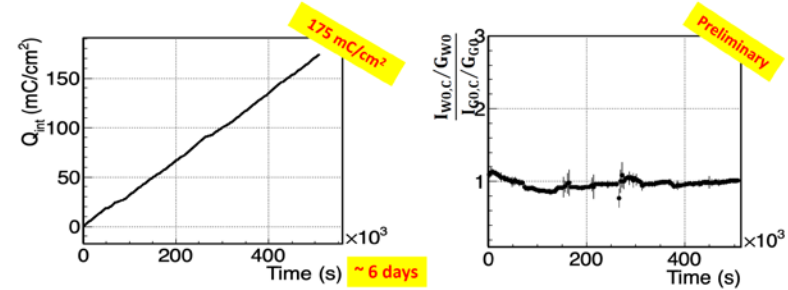
DLC foil (in black) produced by Zhou Yi
DLC foil (in blue) produced by Be- Sputter - Ochi



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.

Thanks

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