R&D progress of the resistive DLC

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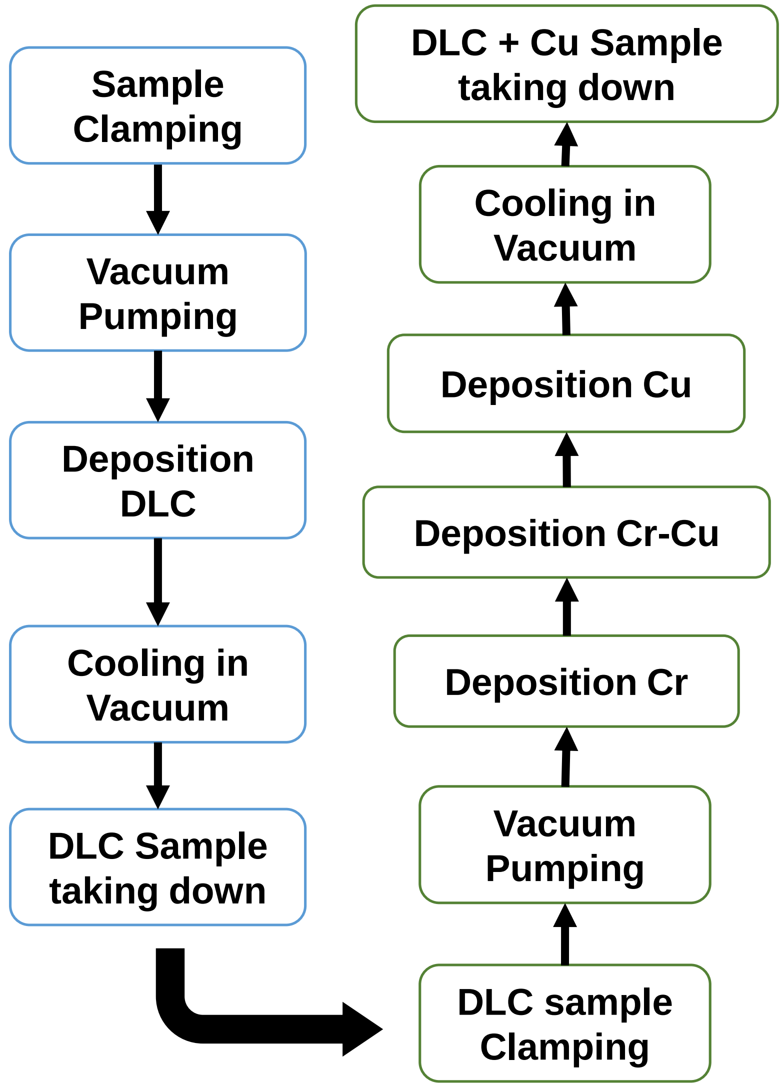
Outline

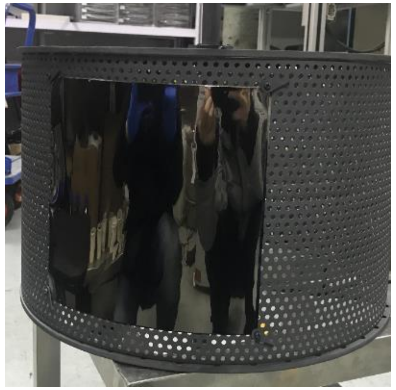
- Summary of the “DLC + Cu” on APICAL
- DLC coating for low resistivity applications
- First attempt of the large area DLC coating

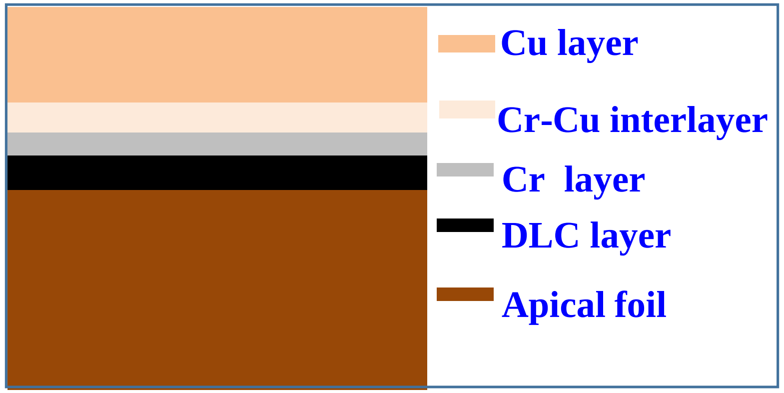
Preparation of the DLC + Cu on APICAL



Advantages of “DLC + Cu”

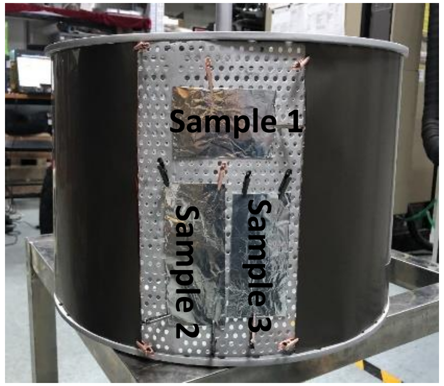
- Simplifying the manufacture process and improving the quality of resistive MPGDs
- Allowing precise printed circuit layouts on DLC resistive electrode thus realizing complex functions
- Expanding the capacity and applications of the MPGDs and opening the way for new MPGD architectures

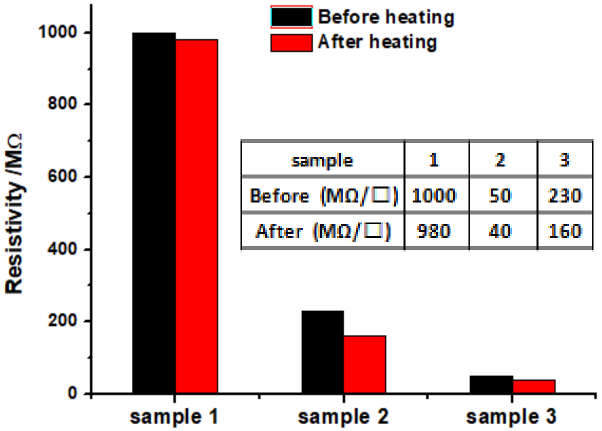




Problem 1: Resistivity decreasing during Cu coating

The bombardment of the Ar ion, sputtering of the Cu/Cr atom will increase the temperature up to 190℃;

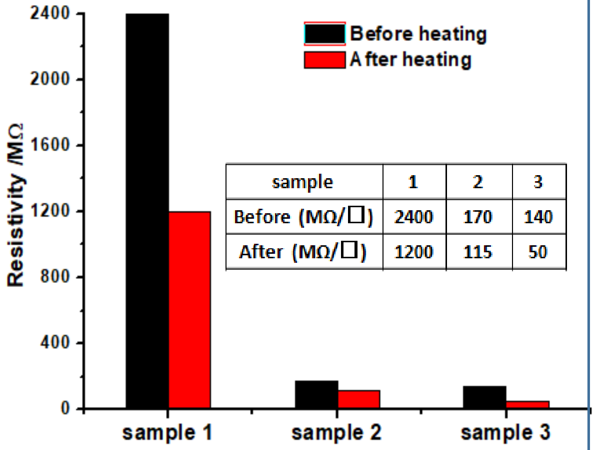




sample	1	2	3
Before (MΩ/□)	1000	50	230
After (MΩ/□)	980	40	160

Depositing Cu in Ar, ～190℃， 5h

Samples in the tube



sample	1	2	3
Before (MΩ/□)	2400	170	140
After (MΩ/□)	1200	115	50

Heating in Ar, ～200℃， 5h

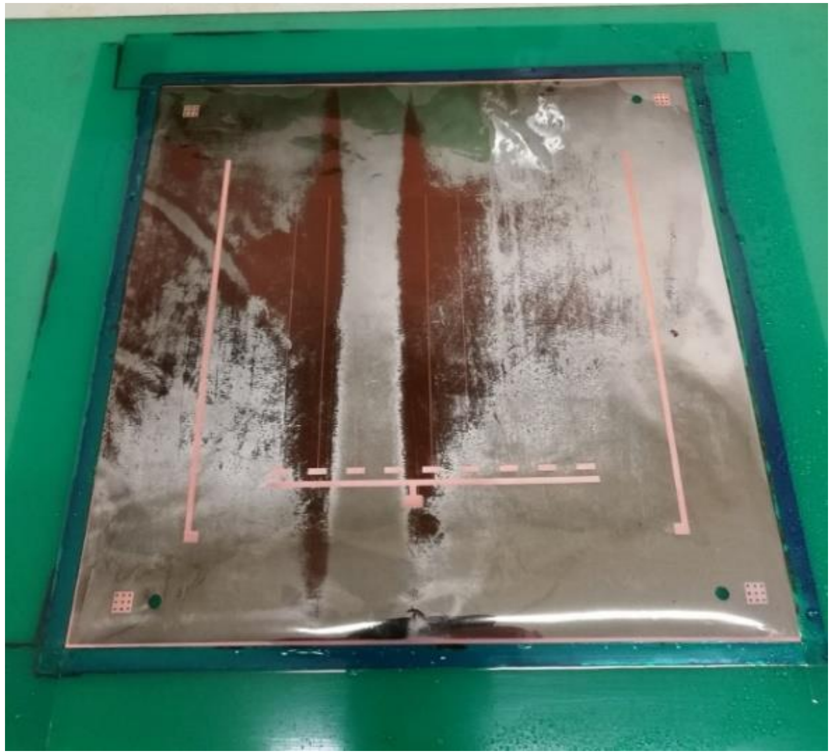
- The resistivity of DLC will be reduced a lot if it is stay under 200℃ in vacuum for 5 hours, the total reduction is not a constant and we don't know if it is sensitive to the roughness of APICAL;
- Currently we are still not able to control the resistivity of the "DLC + Cu" precisely, we can only use the "Calibration + Compensation" method to roughly control it;
- The Vacuum oven is arrived, we will use it to systematically investigate the heating effect(after DLC coating);

Problem 2: Cr & Cr-Cu thickness

THIS HAPPENS BEFORE THE DETECTOR MANUFACTURE

Cr layer will be delaminated from the DLC if the Cr and Cr-Cu co-deposition layers are too thin

Cr or Cr-Cu layers can not be etched if they are too thick

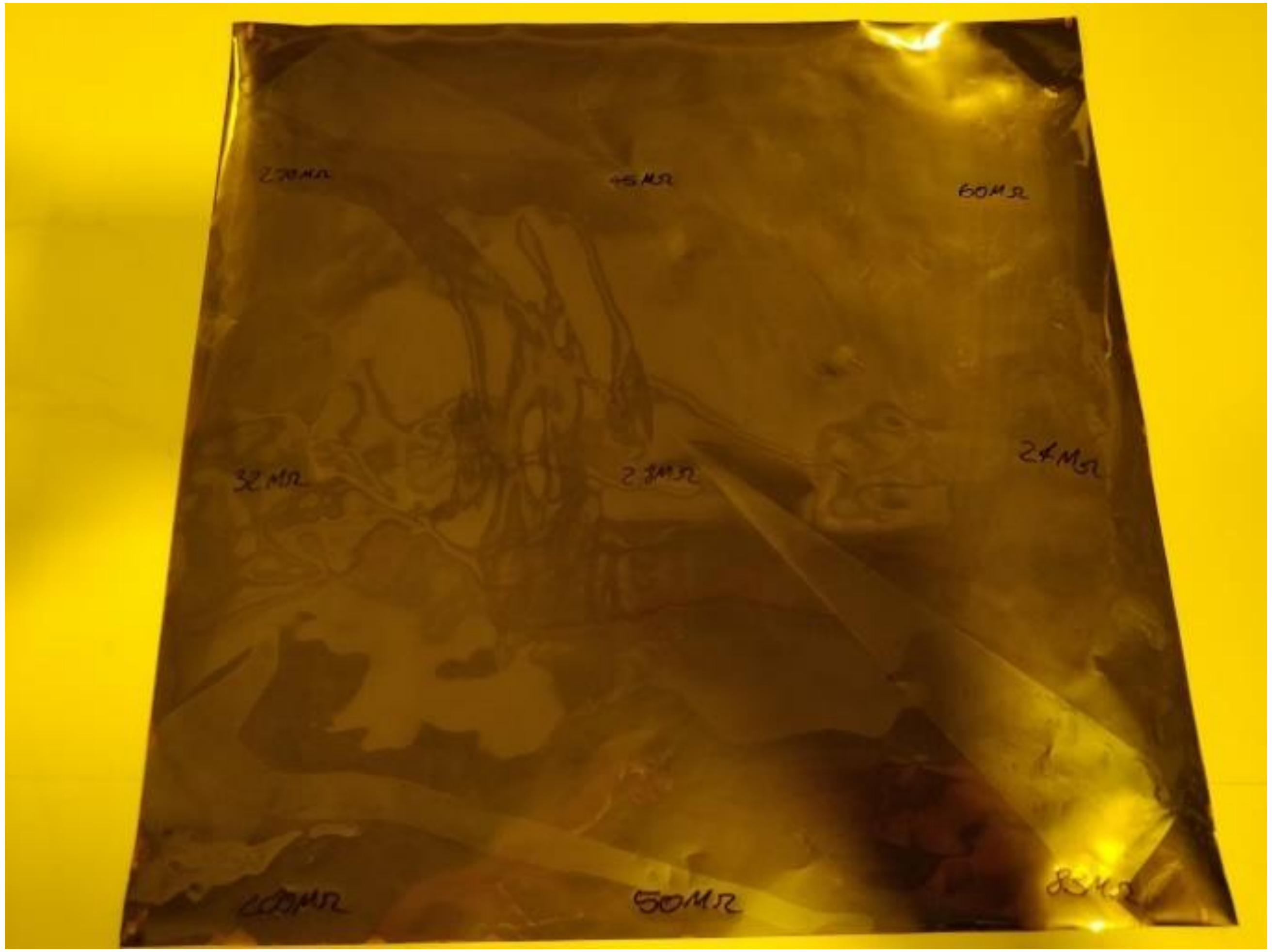


✓ Optimizing the Cr deposition(1A/75s) and Cr-Cu co-deposition(1A～0A/30s for Cr and 0A～1A/30s for Cu) can make a good balance;

× Not perfect, still have Cr delamination in some etching process

A failed production caused by Problem 2

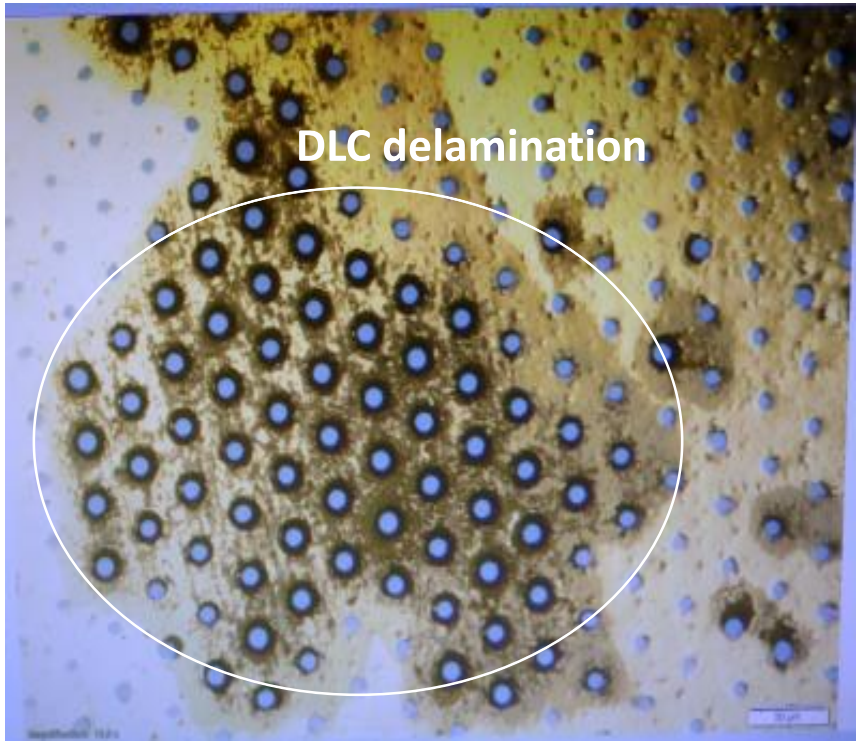
We tried a small production of "DLC + Cu" with active area 25cm×25cm, but failed due to the power of Cr target changed and caused the bad adhesion between Cr and DLC;



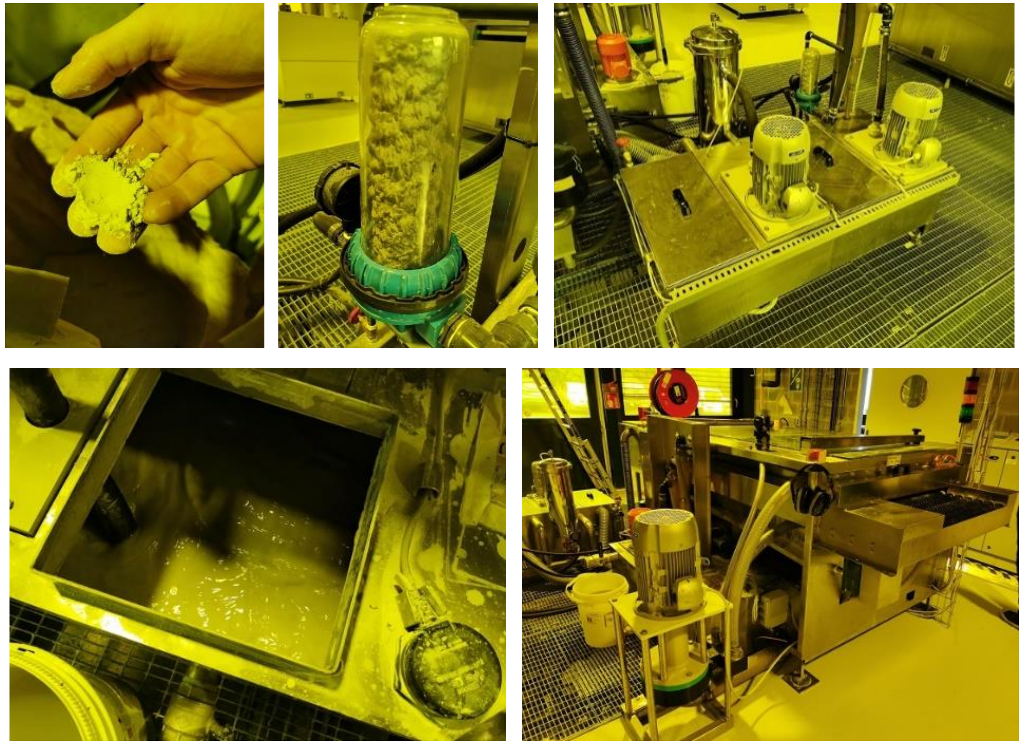
We propose to use these sample to do the resistivity measurement, to see if all the samples has the similar resistivity decreasing after Cu coating

Problem 3: DLC delamination

DLC delamination observed during the APICAL etching process



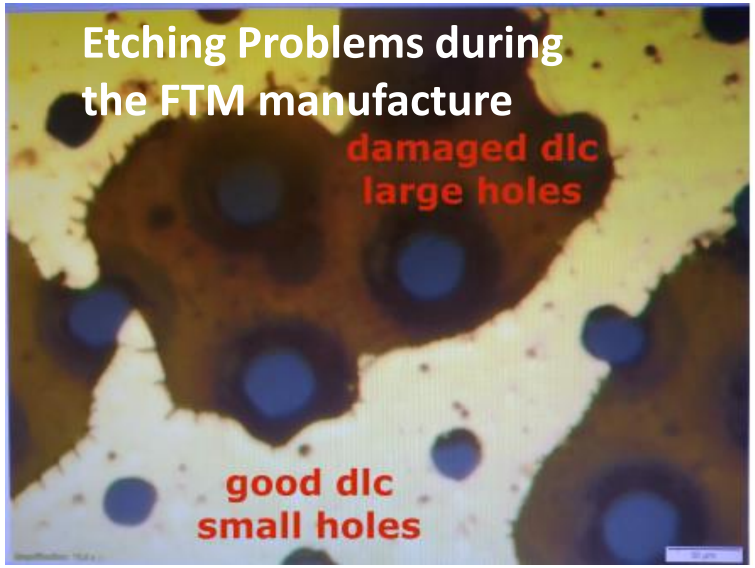
Sand blasting can increase the roughness of the APICAL and then increase the adhesion between DLC and APICAL



APICALs before & after the sand blasting

- ✓ Increasing of the roughness can GREATLY increase the adhesion between the DLC and APICAL
- × The adhesion is not perfect yet， there is still DLC delamination during some etching process

Problem 4: Penetration of the APICAL etching solution



- × The etching solution can penetrate the "DLC + Cu" layer and etch the APICAL below it;
- ✓ By calibrating the Cu etching speed, we found the sputtering Cu is not compact, there are holes inside. Using the galvanic method to coat an extra Cu layer on the sputtering Cu can PERFECTLY solve this problem.

Original "DLC + Cu"

Clean & etching the Sputtering Cu

Cu coating by the galvanic method

Problem 5: Cr delamination

Cr delamination happens sometime somewhere during the detector manufacture！ No certain solution yet!

Possible Solution：

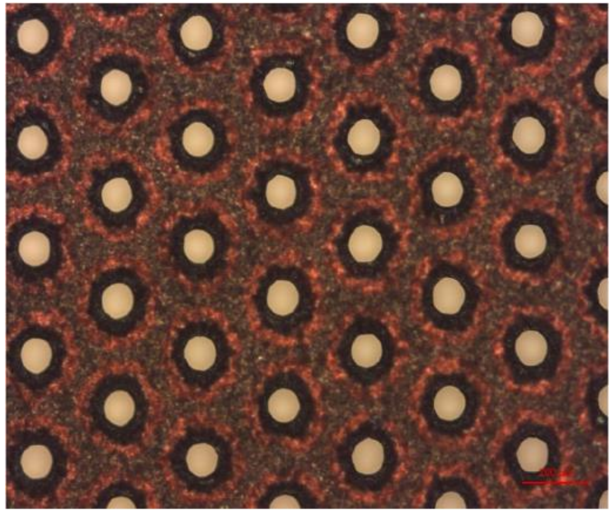
- After the DLC layer is prepared, Cr and Cu are deposited on the DLC directly instead of taking the sample out of the chamber, which can ensure the cleanness of the DLC surface and avoid passivation, this should be able to enhance the adhesion force between Cr and DLC.

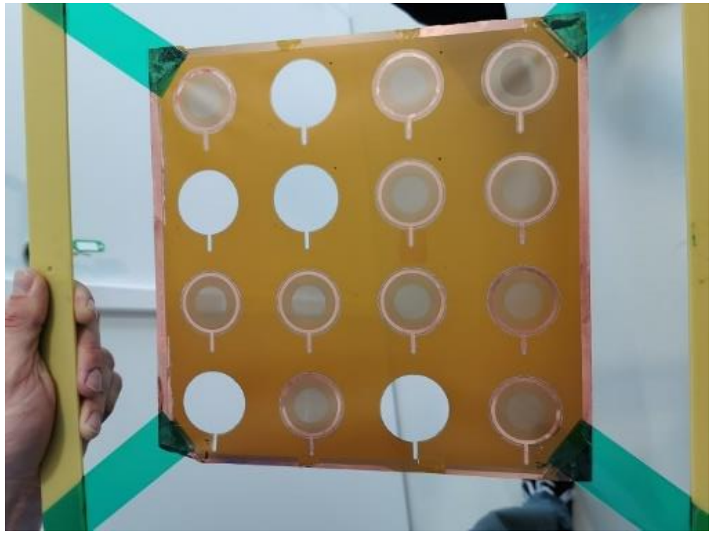
  The problem is that the surface resistivity of DLC resistive layer can not be detected. If the repeatability of DLC preparation process is not good enough, the qualified rate may be reduced and the cost will be greatly increased.

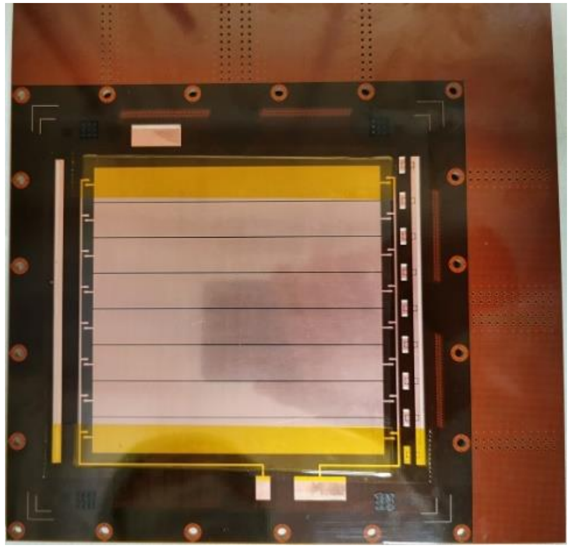
- Before the deposition of Cr and Cu, the DLC and APICAL will be heated, the heating should active the DLC surface and effectively improve the adhesion force between Cr and DLC.

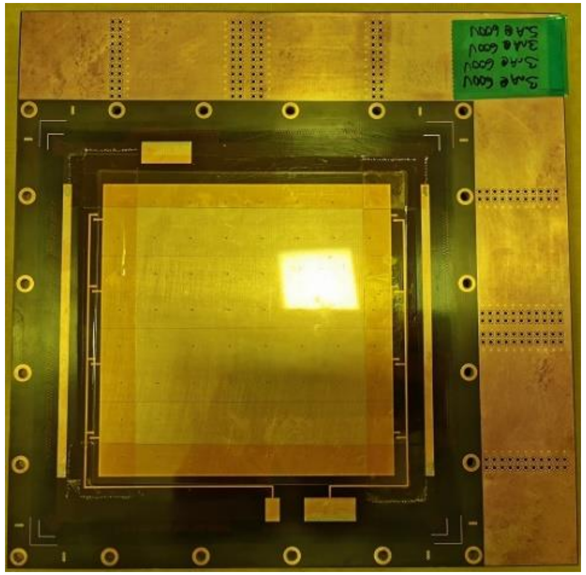
  The problem is that heating will change the surface resistivity of DLC and increase the uncontrollable factors

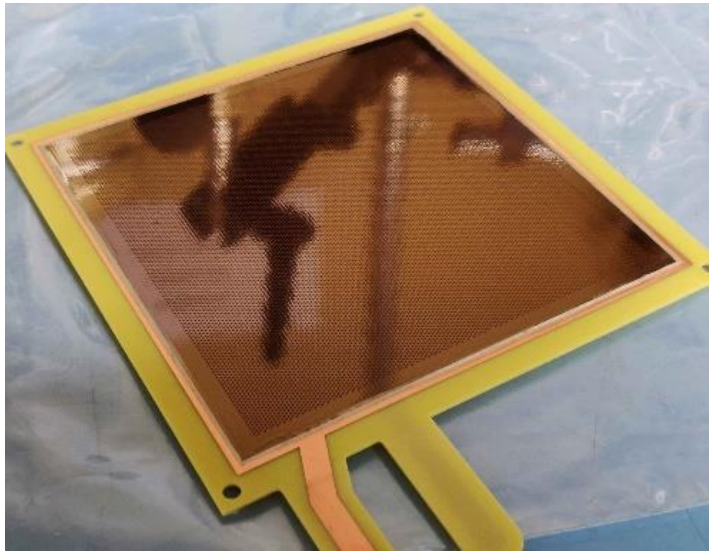
Applications of the “DLC + Cu” on APICAL







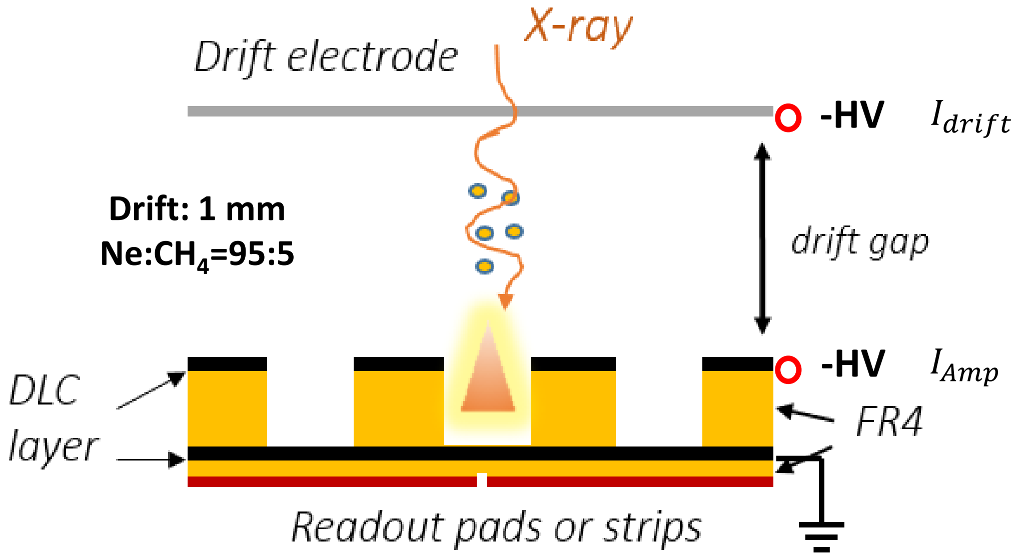


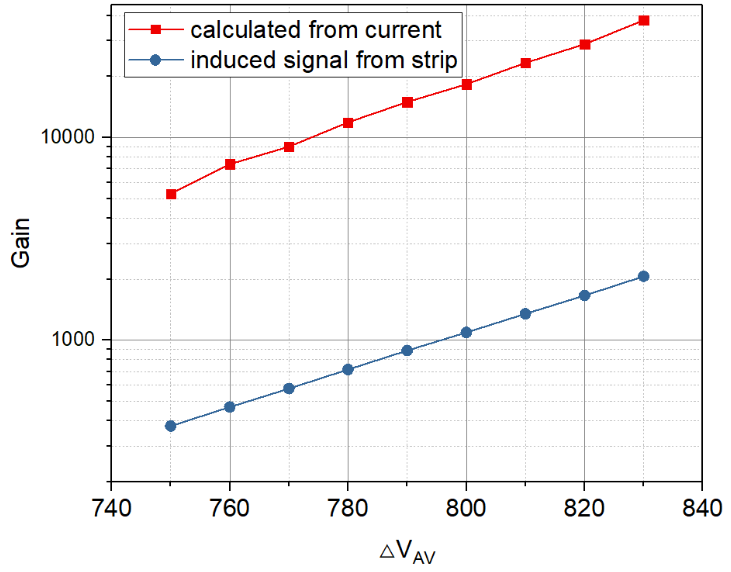


- Resistive GEM
- µRWELL
- FTM
- Resistive THGEM
- Small pad MICROMEGAS
- …………

Applications of the Low resistivity DLC

We already observed that the full resistive detectors have much smaller induced signals





- **In low mass applications, low resistivity DLC can increase the amplitude of the induce signals**

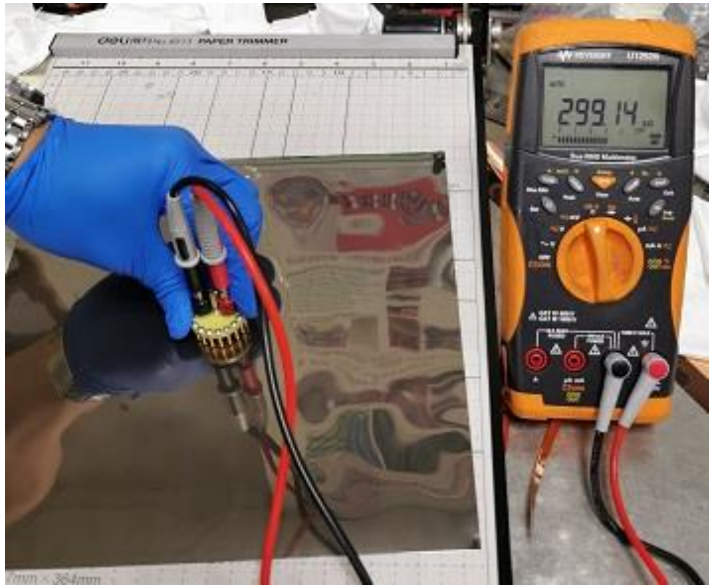
- **In X-ray fluorescence application, copper less detectors are interest, most of these studies are at high rate so that the low resistivity DLC is a potential candidate to be used as the electrode;**

Preparation of the low resistivity DLC

Batch No.	Vacuum Degree(10^{-5} Torr)	Current(A)	Time(min)	Resistivity1 (Ω/□)	Resistivity2 (Ω/□)
8-24-01	0.059	2.8	30	36k	43k
8-25-01	0.056	1	40	300k	320k
8-27-01	0.046	1	40	300k	340k
8-27-01	0.046	1	40	300k	341k
8-28-01	0.049	1	40	400k	342k

To decrease the resistivity of the DLC with thickness around 60nm:

- **High vacuum degree (generally one night pumping)**
- **Heating after the DLC preparation (eg: 200℃ for 5 hours)**
- **Element doping(Nitrogen, Cu, Cr);**
- **Heating the APICAL before DLC preparation (eg: 200 ℃ or higher)**



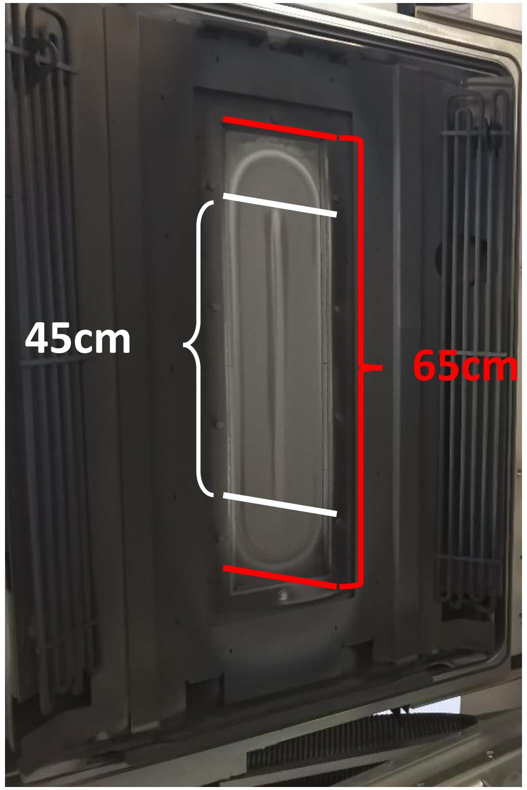
low resistivity DLC on PCB

Thickness of DLC is around 800nm

Batch No.	Vacuum Degree(10^{-5} Torr)	Current(A)	Cr & Cu Current(A)	Time(min)	Resistivity1 (Ω/□)
8-27-06	1.4	3.5	0	80	180k
9-01-01	1.4	3.5	0.3	80	350

- Coating DLC in the same condition, the DLC resistivity on APICAL will be much smaller than that on PCB, it is possible for achieve low values with much smaller thickness of the DLC on APICAL;
- For the applications require only low resistivity, the metal doping is a very good method;
- For low mass or X-ray fluorescence application, metal doping maybe not a good way, we have to try nitrogen doping in future;
- Heating the APICAL before DLC preparation (eg: 200℃ or higher)

Large area coating device: Hauzer 850

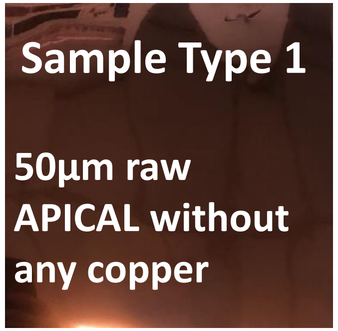


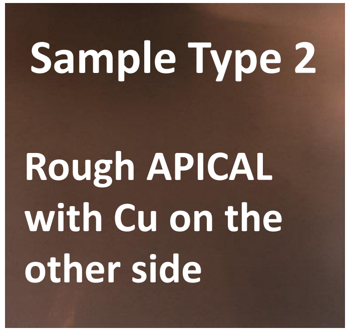
- Chamber size: Φ800mm×900mm
- Target size: 65cm, the DLC produced in the middle 45cm area have good uniformity;
- With heating function, allow to pre-heating the substrate up to 500℃ before coating;
- Equipped automatic shutter, allow the DLC coating and Cr/Cu coating in one batch;

First test with 25cm×25cm APICALs

Goal value: 100MΩ/sq

Sample Type	Thickness (nm)	Vacuum (mbar)	C_2H_2 (sccm)	Heating (℃)	Resistivity (Ω/sq)
1	85	5×10^{-5}	10 sccm	30	～30G
1	85	5×10^{-5}	2 sccm	30	70M
1	85	5×10^{-5}	3 sccm	30	1G
1	85	5×10^{-5}	2 sccm	30	80M
1	85	5×10^{-5}	2 sccm	200	300k
2	85	5×10^{-5}	2 sccm	30	800M
2	85	5×10^{-5}	1 sccm	30	22M
2	85	5×10^{-5}	1.5 sccm	30	200M
2	85	3.4×10^{-6}	2 sccm	30	5M



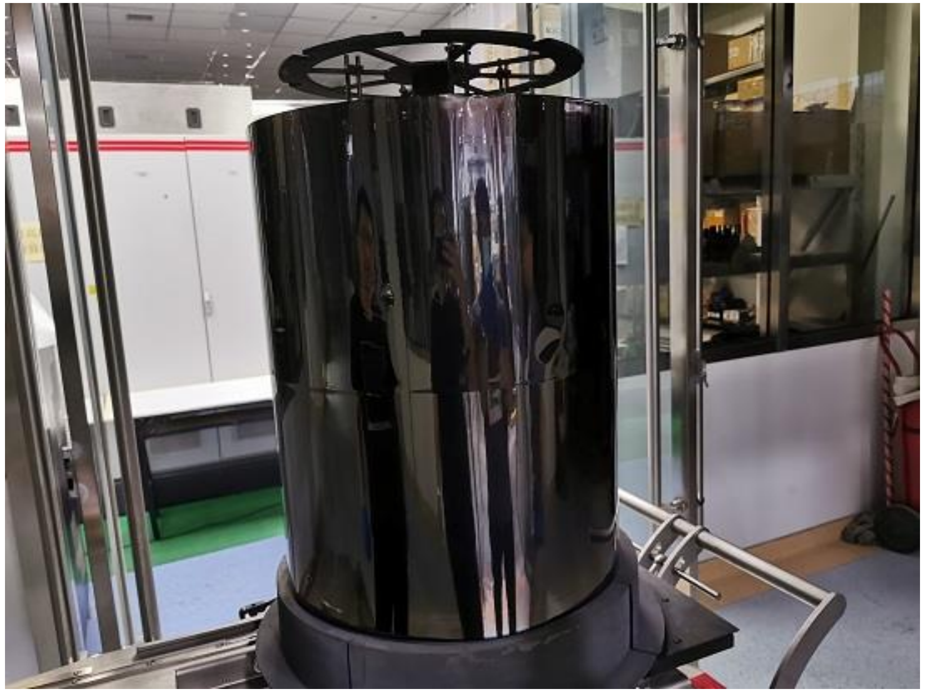


Put back to the chamber and heating up to 200℃ for 5 hours, the resistivity decreased to 23MΩ/sq

The time of whole process is less than 5 hours

- Heating before the DLC coating will get much lower resistivity than heating after the DLC coating;
- Higher vacuum get lower resistivity;

First test with 120cm × 60cm APICALs



Sample Type	Thickness (nm)	Size (cm × cm)	Vacuum (mbar)	C_2H_2 (sccm)	Heating (℃)	No.
2	125	60 × 120	5×10^{-5}	2	30	1
2	125	60 × 120	2×10^{-5}	2	200	2

Resistivity of the DLC on 120cm×60cm APICALs

Vacuum (mbar)	C_2H_2 (sccm)	Heating (℃)	No.
5×10^{-5}	2	30	1

Vacuum (mbar)	C_2H_2 (sccm)	Heating (℃)	No.
2×10^{-5}	2	200	2

Resistivity of Sample No.1 (GΩ/sq)

	2.6		6	
	1.6		2	
	2.6		6.6	

Resistivity of Sample No.2 (MΩ/sq)

60 cm

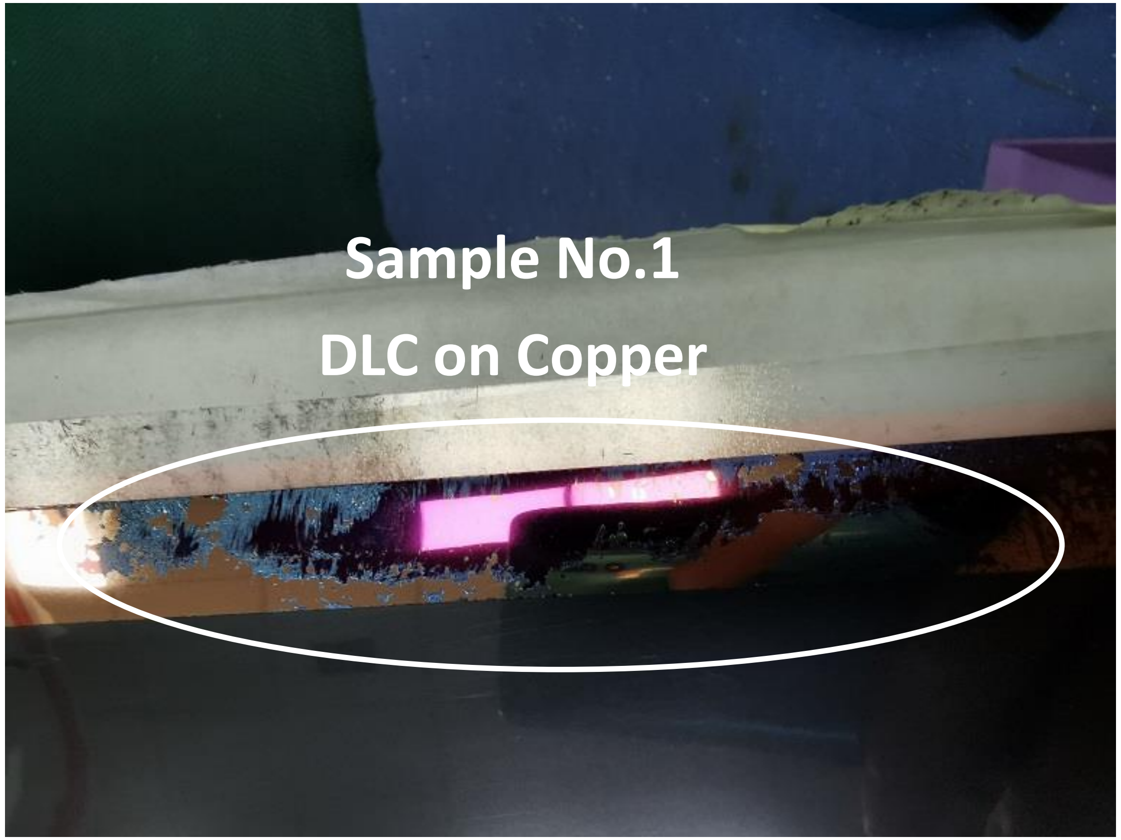
800	940	850	850	1000	1050	1000
270	260	260	300	320	300	320
180	180	165	165	170	165	180
210	185	150	160	150	155	160
270	260	210	210	200	200	220
580	550	480	400	430	450	430

40 cm

Sigma/mean=25%

- **Large area samples (No.1, 125nm, 2G～6G,) will get much higher resistivity than small area samples(85nm,～0.8G);**
- **Heating up to 200℃ can greatly decrease the resistivity(No.1, 2G～6G → No.2, 150M～320M);**
- **The area in the middle 40cm area has good uniformity, but the edge area is very bad;**

Heating effect of the adhesion



- Without heating, the DLC on copper can be easily removed by a gently wipe;
- After 200℃ heating, we need emphatic scratch if we want to remove the DLC on copper;
  - ✓ Heating should be a effective method to increase the adhesion for DLC/APICAL, Cr/DLC;

Summary & Outlook

Summary:

We still have a lots of problems, but we think we are able to solve them step by step!

Near future work plan:

- ➢ Use the vacuum oven to do the heating test for the DLC samples, try to find some rules of the resistivity decreasing;
- ➢ Try to produce "DLC + Cu" in one batch using the Hauzer 850 device;
- ➢ Try to make some large area DLC samples which can really used to make large detectors;
- ➢ More systematically research on the heat effect before DLC coating;
- ➢ Try to use "pre-heating + N-doping" to make conductive DLC