

Diamond Like Carbon for the Fast Timing MPGD

Piet Verwilligen INFN sez. Bari

DLC from a user's perspective

Seminario October 29th 2018, CEDAD

Micro Pattern Gaseous Detectors



Maxim Titov

MPGD Detection Principle



Figure 1.5.: Left: Layout of a double stage GEM detector and signal formation processes triggered by a trespassing muon (purple). The electrons (blue) freed along the track are guided into the GEM holes, where they are amplified, extracted on the other side and further amplified in the second GEM. The ion flow (red arrows) is indicated. The primary cluster number and charge densities are not to scale. Right: Electric field configuration in the GEM stack with the extended drift regions, confined amplification volumes and exemplary streamlines to indicate charge drift.

Who uses MPGDs?

Detector R&D

CERN Courier (5 pages) Volume, October 2015

Detector R&D

RD51 and the rise of micro-pattern gas detectors

Since its foundation, the RD51 collaboratii has provided important stimulus for the development of MPGDs.

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Coupling the micreelectronics infrastry and advanced PCB trachology has been important for the development of gas detection with increasingly smaller girch kitz. An degum transplic forthe one of a CMOS (piot ASE, suscealed directly below the GBM or Micromegas anglification structure. Modern "wolfer porprecessing technology" allows for the integration of Advancemegas pid alexely one opt of a Modiya or Timoga ching, thus forming and alexely one opt of a Modiya or Timoga ching, thus forming



Fig.1. The server-working groups of RD51, with illustrations of just a few examples of the different biads of work involved. Top left: the 20 year pre-history of RD51. shrange credits: RD51 Collaboration.)

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RD51 and its working groups

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Maxim Titov & Leszek Ropelewski

Who uses MPGDs?



 \sim 450 authors, 75 institutes, 25 countries

Who uses MPGDs?



MPGDs at the forefront of Gas Detector R&D

MPGDs: e.g. Gas Electron Multiplier (GEM)



Characteristics:

OK for HL-LHC, μ -tomography

• High rate capability (> 50 MHz/cm²), Good spatial resolution (50 μ m), High efficiency (\geq 95 %), Flexible detector structures, Time resolution of O(5-10 ns)

Missing:

Needed for FCC, TOF-PET, ...

• Spark protection (\rightarrow Resistive Materials) & Time resolution in range 25 ps - 1 ns

Fast Timing MPGD Principle



• slow drift velocity: $v_d = 70 \ \mu m/ns$ [Ar:CO₂ (70:30) @3 kV/cm] • number of primary ionizations: $\lambda = 2.5 \ mm^{-1} \Rightarrow \sigma_t = 5.7 \ ns$ • $P(x \ge a) = \int_{a}^{\infty} \exp(-\lambda x') \ dx' = 0.5 \Rightarrow a \ge 277 \ \mu m \triangleq 3.95 \ ns$

Fast Timing MPGD Principle



- \bullet resistive structure \Rightarrow signal from any layer induced in readout
- resistive structure \Rightarrow limits development of discharges
- time resolution improves with N = number of layers

Two Layer FTM Prototype :: Design

2-layer prototype



Single layer specifications:

- Drift layer: 250 µm drift layer
- Gain layer: 50 μ m kapton
- Resistive kapton: 25 μm
- Resistive coating: 10–100 nm

AMP

- 500V

Two Layer FTM Prototype :: Design



Single layer specifications:

- Drift layer: 250 μ m drift layer
- Gain layer: 50 μ m kapton
- Resistive kapton: 25 μm
- Resistive coating: 10–100 nm

(intrinsic) FTM Challenges



FTM requirements:

- detection of single ionization- e^- instead of all e^- in drift (charge \div 10-30)
- detection with single amplification structure that has very high gain $(10^4 10^5)$

Therefore:

- $\Rightarrow \ {\sf need high gain structure, with low spark/discharge rate} \qquad (\Rightarrow {\sf resistive electrodes})$
- ⇒ need fast electronics that can process pulse with low charge $(10^4 e^- = 1.6 fC)$ does not exist — typically high gain detectors are used for fast timing — $\sigma_t \propto t_{rise} \div S/N$

Signal Transparency vs Resistivity



 \Rightarrow Resistive materials with $R > 200 \text{ k}\Omega/\Box$ are nearly 100% transparent

Rate Capability vs Resistivity



P. Fonte et al. / Nuclear Instruments and Methods in Physics Research A 431 (1999) 154-159



- Effective Voltage due to current *I* and material with resistance $R_e = \rho I/A$:

$$V_{\rm eff} = V_{\rm app} - IR_e$$

- with particle rate R: $I = \langle q \rangle R$
- Voltage drop $\Delta V = \langle q \rangle R \rho l / A$
- *E*-field $\downarrow \Rightarrow$ detector $\epsilon \downarrow$
- $R_{
 m max}$ depends on Gain $\propto \langle q
 angle$ and ho
- Raether limit $(q_{max} = 10^8 e)$ $\Rightarrow G \times R = cte$
- **Typical MPGDs** at $G = \text{few} \times 10^3$ $\Rightarrow \text{MSGC } R_{\text{max}} \simeq 100 \text{ MHz cm}^{-2}$
- CMS RPC $\rho = (1 \div 6) \times 10^{10} \Omega$ cm \Rightarrow (CMS) RPC $R_{max} \simeq 300$ Hz cm⁻²
- low-resistive mat. ($\rho = 4 \times 10^7 \Omega$ cm $\Rightarrow R_{max} \simeq 1 \text{ MHz cm}^{-2} @G = 10^5$

Charge Spread due to Resistivity



Fig. 1. Schematic of a double GEM test cell designed for charge dispersion studies.

• charge density $\rho(x, y, t)$ defined from:

$$\frac{\partial \rho}{\partial t} = \frac{1}{RC} \left(\frac{\partial^2 \rho}{\partial x^2} + \frac{\partial^2 \rho}{\partial y^2} \right)$$

• pad 2 × 6 mm²; $R = R_S \times I/w = 570 \text{ k}\Omega/\Box \times 3$; $C = 0.21 \text{ F/mm}^2 \times 12 \text{ mm}^2$

Charge Spread due to Resistivity



- Vertical Cosmic Ray Muon passing through detector
- Avalanche charge spread $\sim 150\,\mu{\rm m}$; Observed hit pattern: 10 mm \times 30 mm;
- Charge weighting $\sigma_x \simeq 100 \, \mu {
 m m}$ (5% of stripwidth)

 $[R_S = 570 \,\mathrm{k}\Omega/\Box]$

Charge Spread due to Resistivity



Fig. 1. Schematic of a double GEM test cell designed for charge dispersion studies.

- Advantage: Improved position resolution through charge weighting with reduced number of readout-strips
- Disadvantage: If spread out over too many readout channels, signal might be below threshold
- Furthermore: larger part of the detector might become inactive due to voltage-drop after passing of particle

Typical Dimensions and Typical Resistivities



G.Bencivenni NIM A 886 (2018) 36-39

For FTM we would like to have the lowest R that allows for good signal transparency, obtaining as such the highest rate capability, w/o too much charge spread

- Choice of resistivity for an application is a trade-off between various effects:

- rate capability: high resistivity \Rightarrow Large Voltage drop \Rightarrow Low Rate Capability
- **charge spread:** low resistivity ⇒ Large charge spread (need good optimum)
- signal transparency: higher resistivity gives better signal transparency

Want to explore MPGDs w/ $R_S = 100 \text{ k}\Omega/\Box - 1 \text{ G}\Omega/\Box$, first try 100 M Ω/\Box

Which Materials can we use?

Resistive electrodes is one of best choice for reducing the sparks on MPGDs. However, it is not easy to find the "resistive material" for Micro Pattern. In case, surface resistivity of $1M\Omega/sq$. is needed; • In general, the electrodes for MPGDs has $0.1\mu m - 10\mu m$ thickness. • Those correspond to bulk resistivity of $0.1\Omega m - 10\Omega m$. [Qm] 0-8 10-7 10-6 10-5 10-4 10-3 10-2 10-1 100 101 102 103 104 105 106 107 108 109 1010 1011 1012 1013 1014 1015 1016 Bakelite AalPh Ge Si Paper-Δπ Mn ← Wood→ Stainle AL Water ← Glass Co ← Mica→ Ni Nichror Very few materials meets dur reduirement (1r itable zone or resistive MPGDs

Atsuhiko Ochi

Which Materials can we use?

Conductivity of materials

- Intrinsic materials are divided into insulator and conductors.
- Materials in the range of $10^{-9} \Omega^{-1} m^{-1}$ are limited:



Diamond Like Carbon (DLC)

- Diamond-like carbon (DLC) is hard, amorphous carbon film with a significant fraction of sp3-hybridized carbon atoms and lower concentrations of sp2-hybrid carbon, hydrogen and dopants (N,B,...)
- The hardest DLC is tetrahedral amorphous carbon (ta-C)
- Isolator Semiconductor Resistive
- Wide range of applications: reduce abrasive wear & friction, engines, Hard Disks, medical app ...



Raman Spectrum Magnetron Sputtered C-film



Table: Electr. param. (296 K, 100 pA)

Sheet Resistivity (Ω/\Box)	$6\cdot 10^9\Omega/\square$	
Bulk Resistivity (Ωcm)	590 Ωcm	
Mobility (V/cm s)	564 V/cm s	
Density carriers (p-type)	$1.87\cdot 10^{13}$	



Diamond Like Carbon produced in Kyoto, Japan

Traditional Resistive Material: C-black

- Carbon black loaded paste/sheet have been used for resistive material
 - Carbon black: small particles, made from mainly graphite.
 - Those are used by mixing in plastic, epoxy, solvant etc.
 - Mechanism of resistivity development



- Carbon black particles contact each other on point, and it makes electrical path.
- We need very small carbon black particles for fine structure of MPGD electrodes.

Atsuhiko Ochi

New Approach (in MPGD): Sputtering



DLC Resistivity & Stability

- Resistivity dependence on carbon thickness
 - 300Å → 2GΩ/sq.
 - 3600Å → 500kΩ/sq.
 - Conductivity is not proportional to the thickness (t < 1000Å)
 - At t > 1000Å, good reproducibility found
- No time variation founds after several days from sputtering

- However, deposition rate is very slow.
 - 500-600Å / hours are maximum rate in industrial chamber.
 - For ATLAS MM, 3600Å = 6hours are needed!!
 - The MSW foils were made by this longtime sputtering.
 - But we need faster way for mass production.



Atsuhiko Ochi

Reduce Resistivity with N-doping

- The structure of the sputtered carbon is amorphous diamond like carbon (a-DLC).
- It is thought that the charge carrier is very few in the DLC
- So, I got an idea of nitrogen doping as a supplier of carrier electrons.
 - This is same story as the n-type semiconductor production.
- The nitrogen is easy to introduce into the sputtering chamber with Argon gas.



Resistivity vs Thickness



Atsuhiko Ochi



Polyimide Etching in CERN, Switzerland

Polyimide Etching Process



- PI-foil is one-side DLC; one-side Cu
- Single-Mask etching procedure starts on Cu clad side
- DLC side is protected from etching liquid: glued on PCB
- A well understood process (for Cu) became a very delicate process (for DLC)

FTM-v4 :: production problems



test production



Left: DLC top

- Right: Cu bottom
- DLC layer (electrode) good, but still holes way too big diameter. Holes in Cu OK.

failed production



Left: DLC top

Right: PI bottom

 DLC layer (electrode) too small between two holes and breaks. Holes in PI OK.

Electric Field & Gain Simulation (I)



Etching problem hypothesis

- 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



 Rui started new production (Oct 2017), entry hole (Cu) will be reduced from 85 μm to 65 μm diameter and etching will be carefully monitored and timed to stop etching upon arrival at bottom PI and avoid over-etching. Result: bi-conical hole, not exactly what we want from point of view of high gain: instead of a gain of few 10⁴ we will have only 7–8 10³ at 550 V in Ar:CO₂ 70:30.

Electric Field & Gain Simulation (II)

produced DLC 65/45/80

produced DLC 80/45/65

simulated E_{amp} & Gain













Pioneering new FCCLs



NEW FCCL: 2xDLC covered by Cu: Cu - Cr - DLC - PI - DLC - Cr - Cu 5 - 0.1 - 0.1 - 125 - 0.1 - 0.1 - 5 (um)

Double mask etching procedure:

Cu (&Cr) photolithography + DLC sand blasting Chemical etching of PolyImide (PI) Removal of Cu (&Cr)

Advantages:

Can etch thicker Polyimide (up to 125um) Allowing higher gain structures

Better protection of DLC during etching procedure

FCCL construction procedure: PLD of DLC, followed by PLD of Cr and Cu, Polyimide film does not exit vacuum chamber

Idea developed during MPGD-Next by R. De Oliveira (CERN) & M. Maggi (INFN);

already adopted by USTC Colleagues (China - Magnetron Sputtering)



Diamond Like Carbon produced at USTC, China

Time Dependent N:DLC Resistivity



Zhou Yi

Double-sided DLC

Problems and Possible Solution



Problems in double-side coating:

- Caused extra non-uniformity of the surface resistivity;
- The maximum sample size is greatly decreased;

The edge area is closer to the target than the center area, caused the resistivity in edge area is much smaller than it in the center area

The size of the sample is limited by the space of the chamber and the structure of the rotator, the maximum size is about 15cm × 15cm

Possible Solution:

- Try to add Boron into the DLC to greatly decrease the inner stress of the thick DLC foils;
- After we are able to get the thick DLC foils with low inner stress, we can use the 1-axis rotator for DLC deposition, which allow us to produce large size thick DLC samples;

Highly tetrahedral amorphous carbon films with low stress

M. Chhowalla,¹⁰ Y. Yin,¹⁰ G. A. J. Amaratunga, D. R. McKenzie,¹⁰ and Th. Frauenheim⁶¹ Department of Electrical Engineering, University of Liverpool, Liverpool L69, 3 BX, United Kingdom

(Received 15 April 1996; accepted for publication 1 August 1996)

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Zhou Yi

Depositing Cu on DLC



Depositing Cu on DLC





First Test

• Directly deposit copper on DLC for 60min

Second Test

- Coat Chrome (~100nm)on DLC
- 5min Chrome + Cu coating
- 60min Cu coating



The bending stress test (Crazy kneaded by me)





The DLC foils don't have enough toughness for attaching the Copper



The crosscut test (Glued many times)

Adding etchable Chrome transition layer between DLC and copper can provide good adhesion force to each interface





Diamond Like Carbon production in Lecce, Italy

FTM-Next

FTM-Next CSN-V experiment 2019-2021

Proposal to continue R&D of the Fast Timing MPGD (FTM) based on experience and lessons learnt 2015-2018

• Team:

- INFN-BA (1.3FTE) detector simulation, design & test
- INFN-LE (1.2FTE) DLC deposition & characterization
- INFN-PV (0.7FTE) detector test
- Finances: 130 kEUR in 3 years

Goals:

- Reduce production time of Polyimide foils with thin DLC coating
- Develop procedure for DLC deposition with **reproducible resistivity** through **Pulsed Laser Deposition** process & increase DLC strength
- demonstrate FTM principle with small-size prototype
- Continue Fast Electronics development for smal signals (1.6 fC)

High Quality Thin DLC films through PLD



will allow for fast iteration to converge to reproducible results

• Characterization of DLC films:

- AFM & SEM to study morphology & topography of DLC;
- DLC-PI bond & DLC internal stress investigated through Raman spectroscopy; 4-point Hall probe to characterise electrical properties

• Excellent expertise in field of Thin Films in Italy (Lecce)

Detector Construction & Tests





Gaseous Detectors for Future Colliders



- Future Colliders (FC) will operate at higher \sqrt{s} and $\mathcal L$
- For muon detection systems of FC experiments:
 - $\begin{array}{l} \frac{\Delta\rho}{\rho} \propto \frac{1}{BL^2} \Rightarrow \text{high } B \text{ field, large instrumented area} \\ \mathcal{L} \Rightarrow \text{higher rate capability, Pile-Up vertices} \end{array}$
- To instrument large areas, gas detector technology will remain unchallenged
- Detectors need to have high rate capability
- Fast Timing will enable to identify the correct interaction (as many as 1000 collisions will overlap with the interesting collision = Pile-Up)

ttbar event overlayed with 140 additional pp-collisions (Pile-Up)

Time-Of-Flight Positron Emission Tomography



PET principle, w/ & w/o TOF. [source: sublima-pet-mr.eu/]



PET scans of a patient with colon cancer. The use of TOF improves the lesion detectability (arrow). [source: J. Karp, U. Pennsylvania.]

PET is a technique to visualize organs with **high metabolic activity** and is used to spot tumors

- β^+ sugar (FDG) is administered to a patient and concentrates at regions of high metabolic activity (tracer)
- e^+ is released and looses energy during travel (~1 mm) before annihilation
- 2 γ (511 keV) are emitted back to back, their coincident detection determines the Line of Response (LOR)
- w/o TOF equal probability assigned to each point along the LOR
 - w/ TOF few 100 ps measurement will lead to $\sim 5\,{\rm cm}$ precision along the LOR

the use of fast timing in PET results in high contrast images

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- Giuseppe De Robertis & Francesco Licciulli for desiging and testing the FATIC electronics;
- Antonio Ranieri & Anna Paola Caricato for their trust and endless stimulation;
- Archana Sharma, Marcello Maggi and Rui De Oliveira, mother and fathers of the FTM for their continuous support.

Questions?



Backup:

- Amplification Structures
- DLC for ATLAS NSW MM
- FTM-v1 Results
- TOF-PET FTM Simulations

Amplification Structure: Resistive μ -WELLs



- By constr: FTM <u>inverted</u> hole (50-70 μm), μ-RWELL <u>normal</u> hole (70-50 μm)
- Electric Field calculation in gas very similar (independent of base details)
- FTM (multip. layers) \Rightarrow micro-gap (250 μ m); while 3–4 mm drift for μ -RWELL

ATLAS New Small Wheel Upgrade



Atsuhiko Ochi

DLC Sputtering vs Carbon Screen Printing

Two options for resistive electrodes of ATLAS MM

- Sputtering+liftoff
 - Pros.
 - Large area (>2m)
 - Fine pattern (<100µm)
 - Uniform resistivity
 - Strong attachment on substrate
 - Cons.
 - Production speed (Now, it will be OK, next slide)
 - High cost



- Screen printing
 - Pros.
 - Large area (>2m)
 - Fast production speed
 - Low cost for mass production
 - Cons.
 - Stability of resistivity
 - Thick pattern (~20 µm)
 - Lower tolerance for breakdown for high voltage



Atsuhiko Ochi

Trials of Different Resistive Materials 1

Our experiences of resistive materials

- Resistive Polyimide sheet
 - Dupont Kapton XC series
 - · Commercially available (not in Japan, Rui's help was needed)
 - $\circ\,$ Typically, 2M $\Omega/sq.$, 25 μm thickness
- Resistive polyimide paste
 - Produced in Toray
 - It is not commercially available
 - $\circ\,$ A few MQ/sq. , 10–20 μm thickness
- Resistive epoxy paste
 - ESL RS12500 series
 - Commercially available
 - $\circ\,$ Typically, $1M\Omega/sq.$, $10\mu m$ thickness
- Carbon sputtering
 - Carbon deposition by dry sputtering
 - $\circ~100k\Omega/sq.$ $100M\Omega/sq.$, $0.1\mu m$ thickness









Trials of Different Resistive Materials 2

Summary

- \triangleright Various resistive materials were tested for $\mu\text{-PIC},$ MM and GEM prototypes.
- > For fine structure, photo lithographic method is suitable.
 - Most fine structure will be formed by carbon sputtering
 - Also huge size (>1m) production is available
- Screen print of resistive paste is very low cost
 - Less fine, but it is enough to ATLAS NSW MM resistive strip

Materials	Resistive sheet	Paste (polyimide)	Paste (epoxy)	Sputter
Resistivity	2M	1M~2M	0.5M~2M	50k~100M
Fineness	~50µm	~100µm	~100µm	~10µm
Process	Etching Laser drilling	Liftoff Screen print	Screen print	Liftoff Laser drilling
Detector	µ-PIC GEM (RIKEN)	µ-PIC MicroMEGAS	MicroMEGAS	µ-PIC MicroMEGAS GEM

Atsuhiko Ochi

First FTM Prototype (FTM-v1) :: Results



[source: arXiv:1503.05330]

[source: NIM A 845 (2017) 313]

- Simulations show time resolution decreasing for increasing number of layers
- Pion Test Beam for 2 layer prototype shows time resolution of $1.7\pm0.1\,\mathrm{ns}$
- Muon Test Beam for 2 layer prototype shows time resolution of 2.4±0.1 ns

•
$$\sigma_t = 1/(\lambda v_{\text{drift}} N)$$
 with $\lambda_{\text{Ar/CO}_2 \ 70/30}^{\text{MIP}} = 25 \text{ cm}^{-1}$, $v_{\text{drift}} = 8 \text{ cm}/\mu \text{s}$, $N = 2 \Rightarrow \sigma_t = 2.5 \text{ ns}$

TOF-PET FTM Simulations



- Simulation split in two steps:
 - Search for optimal resistive convertor material and extract electron energy distribution at exit material
 Simulate Primary Ionization of sub-relativistic electron
 - Simulate Primary Ionization of sub-relativistic electron and extract expected time-resolution
- Simulate two-amplification layer (Scheme A) to understand whether electron can perforate thin amplification structure to decide between scheme-A and scheme-B

Photon Conversion

- Study to guide design of FTM for PET scheme, materials, geometry, ...
- GEANT4.10.03 version used
- FTFP_BERT_HP physics list
- Simulation of 511 keV γ interaction in different materials and thicknesses:
 - PCB (FR4), kapton, glass (G4_GLASS_LEAD), lead glass (G4_GLASS_PLATE)





511 keV γ conversion in different materials is studied. Electron production probability and energy spectra for electrons entering the drift region.

Sub-relativistic Electron Ionization



- Search for right approach to simulate the ionization of sub-relativistic electrons
- HEED gives good agreement with NIST ESTAR and Bethe-Bloch
- Garfield Microscopic Tracking (Magboltz cross sections) ok for low-energy
- Time resolution estimate made for single-layer FTM with convertor:
- Electron Momentum: 50 keV 200 keV 300 keV :: 100 ps 1 ns 2 ns