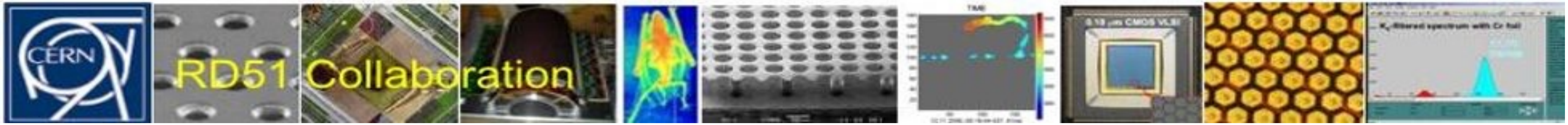
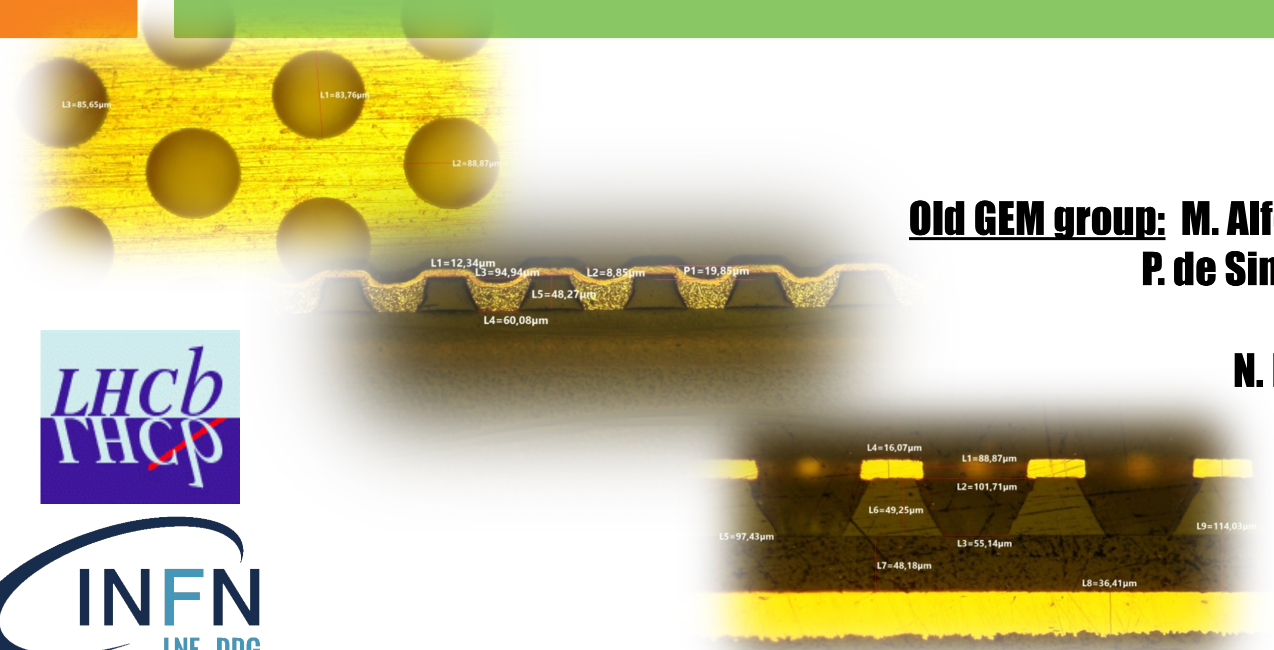


Irradiation effects on GEM detectors operated at RUN1 and RUN2 at the LHCb experiment



M. Poli Lener

<u>Old GEM group:</u> M. Alfonsi, G. Bencivenni, W. Bonivento, A. Cardini, P. de Simone, F. Murtas, D. Pinci, D. Raspino, B. Saitta,

⊕

N. Bondar, D. Brundu, M. Giovannetti, G. Morello

<u>EN-MME-MM CERN:</u> A. T. Perez, S. Sgobba

LHCb

INFN LNF - DDG

OUTLINE

- GEM detectors in the LHCb experiment
- Used materials for the detector production

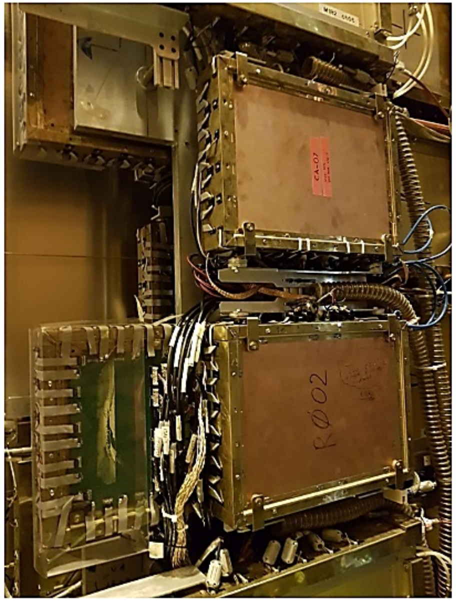
- Global irradiation test @ ENEA Casaccia with ^{60}Co source in 2004 (reminder):
  - Results
  - Tests on high irradiated chambers
  - Scanning Electron Microscope (SEM) and Elemental composition analysis (EDS-Energy dispersive Spectroscopy) performed by EN-MME-MM CERN Group (*)

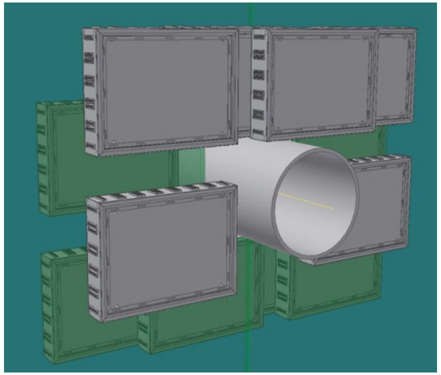
- Irradiation effect after the operation at LHCb (2010-2018):
  - SEM analysis
  - EDS spectroscopy

- Summary and outlook

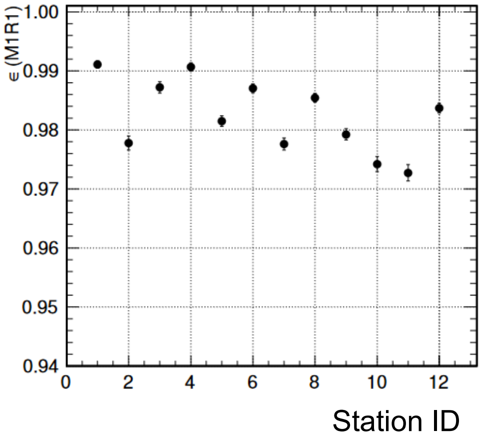
(*) https://en.web.cern.ch/group/mme

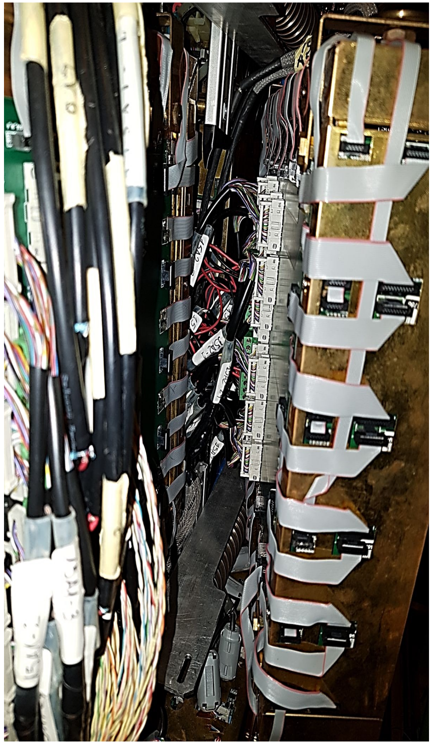
GEM detectors in LHCb





Station efficiency in 25 ns @ LHCb (**)





12 stations around the beam pipe composed of 2 Triple-GEM OR r/out (*)
GEM detectors operated in Ar/CO2/CF4=45/15/40 during RUN1 & RUN2 (2010 to 2018):

- **particle rate of 200-300 kHz/cm2**
- **efficiency ≥96% in the single BX (25 ns time window)**
- **gas gain of ~4000**

The detector gas is supply by polypropylene (~30 cm) & Cu tubes. The gas mixture is analysed with a gas chromatographer and a water and oxygen measuring system

(*) More details on the Davide's presentation 8th Nov 14:50 "The LHCb Triple-GEM Detectors: Operational Experience"

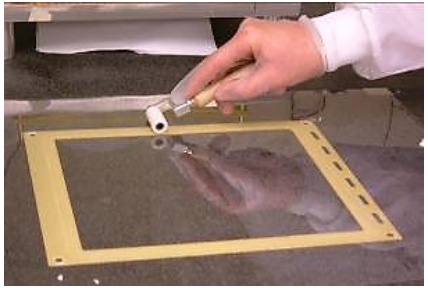
(**) https://cds.cern.ch/record/1495070/files/LHCb-PROC-2012-060.pdf

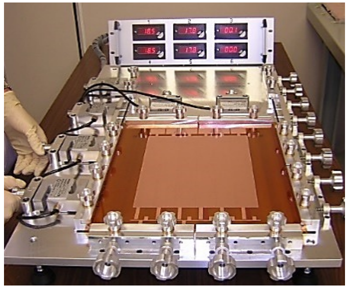
ASSEMBLY Procedure

The whole detector assembling is performed in a clean room class 1000

GEM FRAMING/ANODE preparation

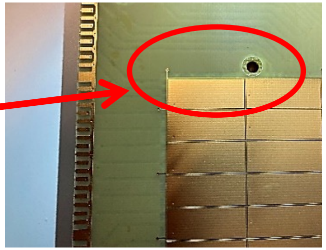
Before gluing the frame (**FR4**) is checked again for broken fibers, cleaned with isopropyl-alcohol and dried with nitrogen flow.





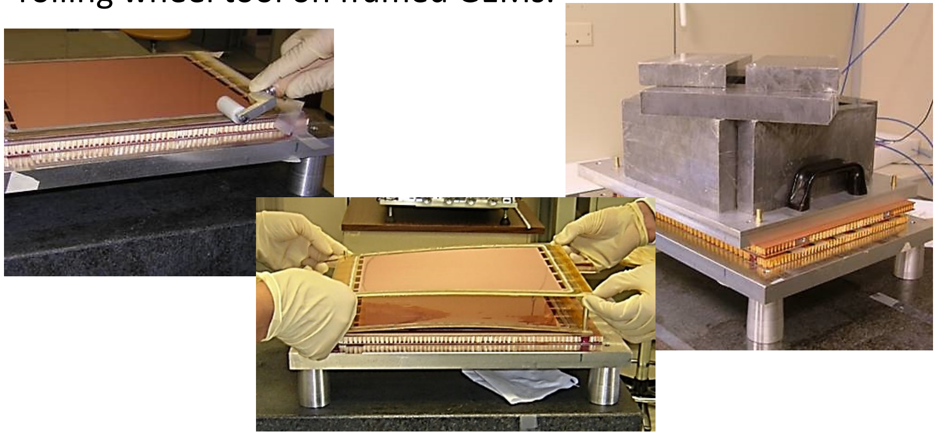
Araldite 2012 epoxy is applied with a rolling wheel tool on the frame. A **glue rim** is also applied on the ANODE gas insert.

Araldite 2012 work life: 4 minutes; curing time: 2 hours.



CHAMBER ASSEMBLY

The chamber assembly procedure is divided in 2 steps. Epoxy araldite **AW103 + HY991** is applied with the usual rolling wheel tool on framed GEMs.



One after the other the 3mm, 1mm, 2mm framed GEMs, plus an additional bare 1mm-frame (induction gap), are positioned on the cathode PCB panel.

The assembly operation is performed on a machined ALCOA reference plane. Over the whole structure a load of 40 kg is uniformly applied for 24h, as required for epoxy polymerization.

Summary of the used materials (in contact with gas mixture)

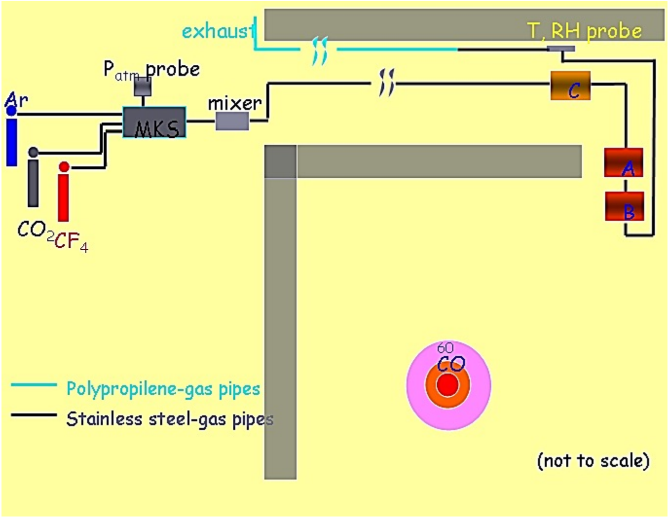
Material	Type	Note
ARALDITE 2012	Epoxy Compounds	GEM framing & gas inserts passivation (only ANODE)
AW103 + HY991		Det. Assembly & sealing
FR4	Rigid Material	Frame
Gold plated PCB		Anode & cathode surfaces
FR4 & Brass		Gas Insert
Polypropylene & Cu tubes	Piping	
Cu + Kapton	Flexible Material	GEM base material

More information on Outgassing/Effect on detector are reported in:
https://detector-gas-systems.web.cern.ch/Equipment/outgassing.htm#plastic

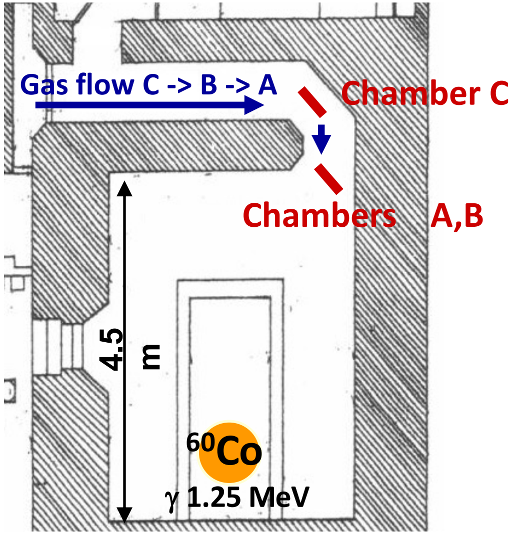
Casaccia Global Irradiation Test in 2004 (reminder)

Global stability test: setup

A full size (20x24 cm^2) prototype (C) in low irradiation position ~ 1 MHz/cm^2,
2 full size prototypes in high irradiation position: ~ 15 (chamber A) and ~ 20 MHz/cm^2 (chamber B)
Ar/CO2/CF4 (45/15/40) at gain ~ 6x10^3 & 35 irradiation days



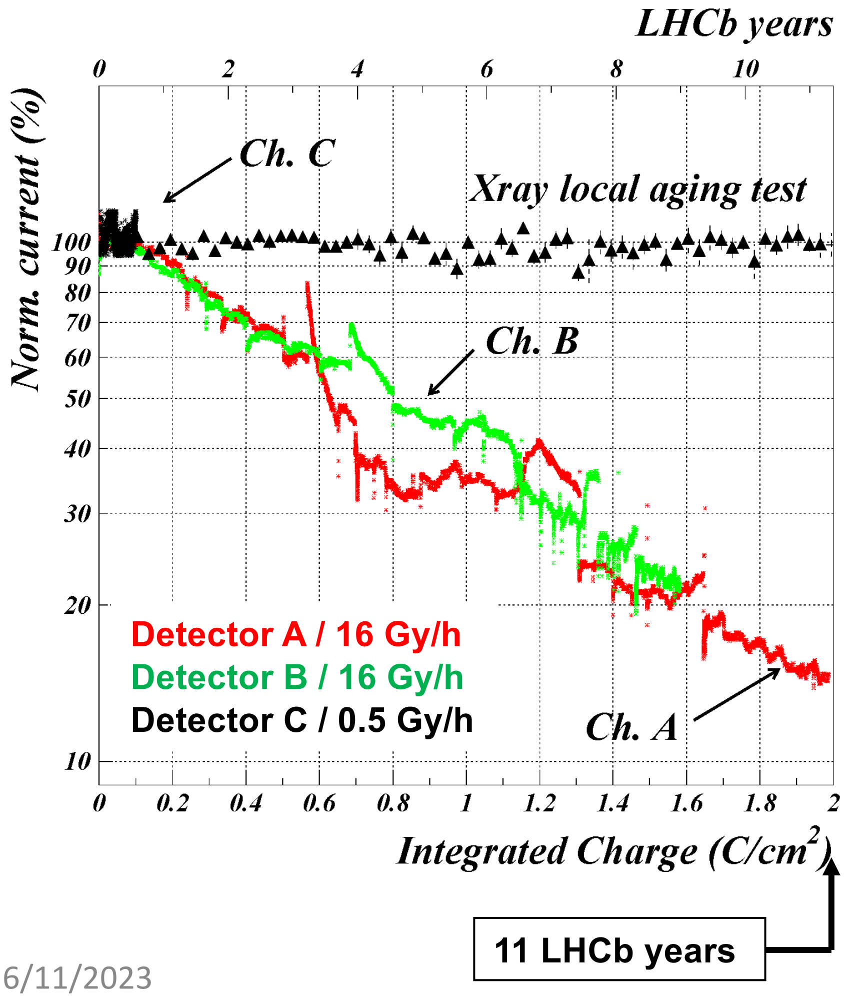
ENEA - Casaccia



Ambiental parameters: H2O (± 1ppm), T (± 0.1°K), atmospheric P (± 0.1mbar)
Gas flows: C → B → A → T/H2O Probe -> Out initially Φgas= 200 cc/min, then Φgas= 350 cc/min
Gas inlet line → stainless-steel tubes
Exhaust gas line → polypropylene tubes (not hygroscopic)

IEEE TRANSACTIONS ON NUCLEAR SCIENCE, VOL. 52, NO. 6, DECEMBER 2005

Global aging test: results



Integrated charge:

detector C ~ 0.16 C/cm^2 ⇔ 1 LHCb y

detector B ~ 1.6 C/cm^2 ⇔ 8.5 LHCb y

detector A ~ 2.2 C/cm^2 ⇔ 11.5 LHCb y

High-irradiated chambers exhibit a drastic current drop during the test

A → -89%

B → -80%

Chamber C as well as **X-ray** results show no current drops after an integrated charge of **0.16 C/cm^2** , while Chamber A & B show **$\Delta G/G$ ~ -10% after the same integrated charge !**

Preliminary conclusions

REMINDER

The obtained result was due the **low gas flow rate** (**350 cc/min**, the maximum flow reachable with our mass-flowmeters) ⇒ **LOW with respect to the very high particle rate** (~ 15-20 MHz/cm^2 equivalent m.i.p. on the whole detector area ⇔ 400-500 μA)

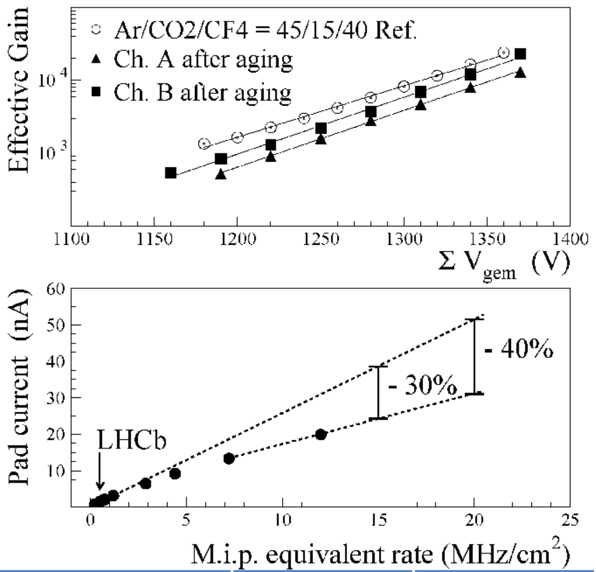
high-irradiated chambers suffered of **gas mixture pollution** ⇔ submitted **to a strong plasma etching due to F** (CF_4 fragmentation) **not quickly removed by the gas flow**

Several **tests and checks on aged chambers** to understand the aging process have been performed:

- gain and rate capability measurements with X-rays
- performance at beam test
- reproducing the low gas flow effect observed at Casaccia
- SEM & EDS analysis

Test on aged chambers

Aged chambers exhibit NO rate capability loss up to **~3MHz/cm²** (expected LHCb rate ~0.5MHz/cm²)



	Chamber A	Chamber B
Gain reduction	~ 55%	~32%
Rate reduction	~ 30%	~40%
Total reduction	**~ 85%**	**~ 70%**

Test on aged chambers

REMINDER

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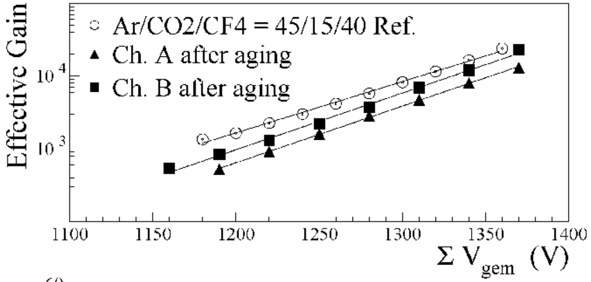

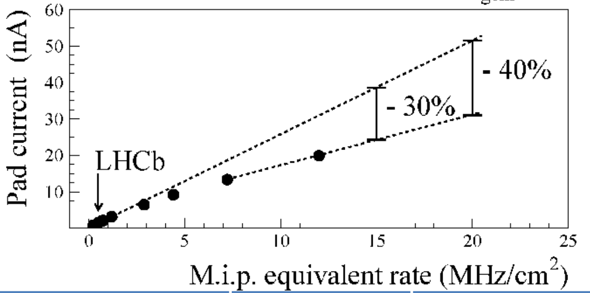

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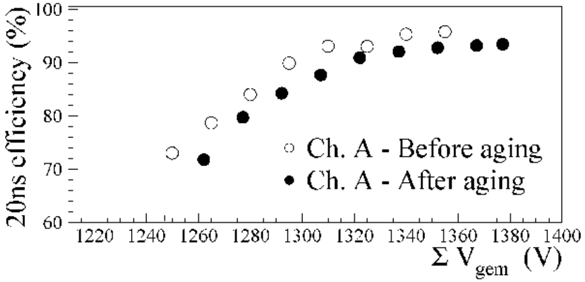
Chamber A (with the larger gain loss) shows ONLY a shift of the working point of **~15V**



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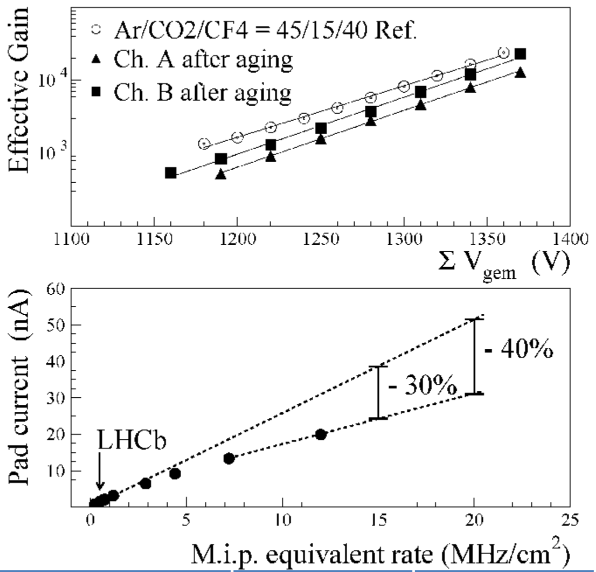

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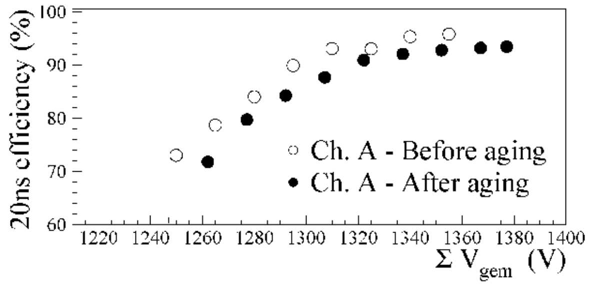
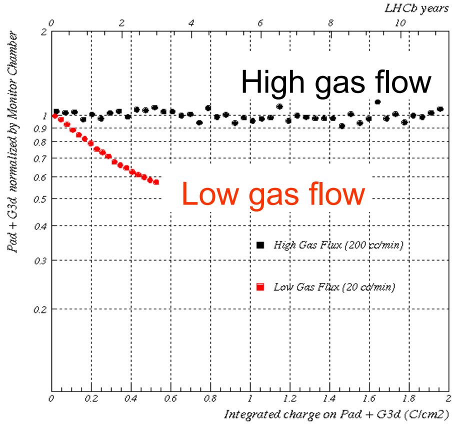




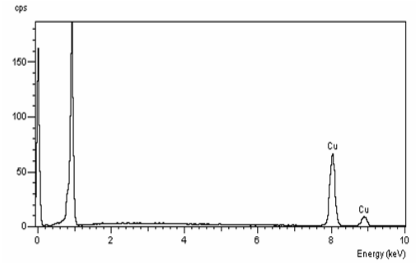
To reproduce the Casaccia test results, a small chamber has been irradiated with X-rays (total current ≅ **2 μA on ≅ 1 cm²** irradiated spot) flushed with a low gas flow rate (**20 cc/cm**)

A current drop of **~ 40%** for a **0.55 C/cm²** integrated charge (~3 LHCb years) is found on the low gas flow measurement

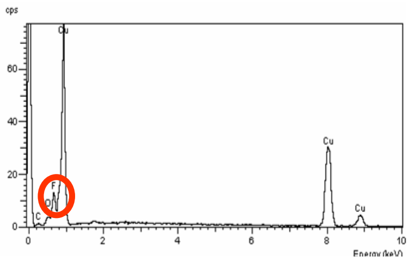
SEM analysis & X-ray spectroscopy on aged chambers

REMINDER

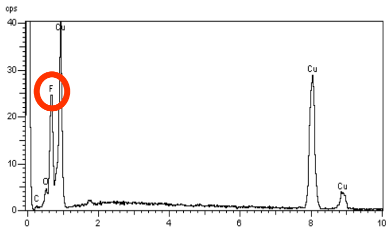
Fluorine etching not only widens the copper hole, but also removes the Kapton inside the hole



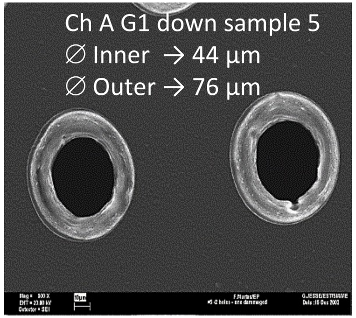
G1: No fluorine No etching

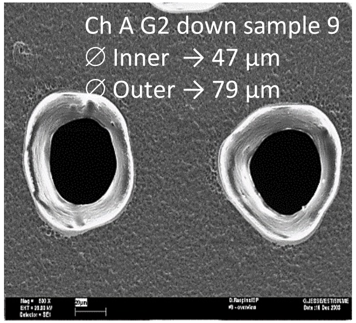


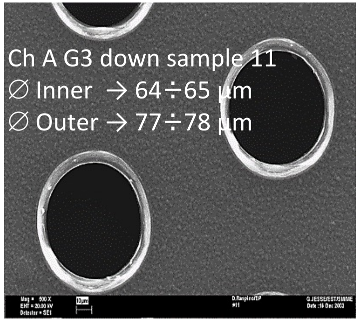
G2:Small fluorine, etching started



G3:Large fluorine etching enhanced

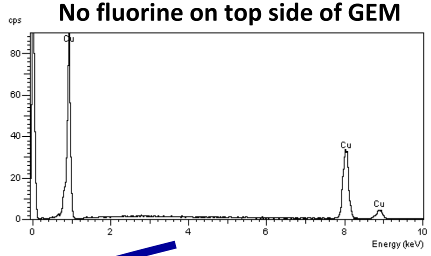


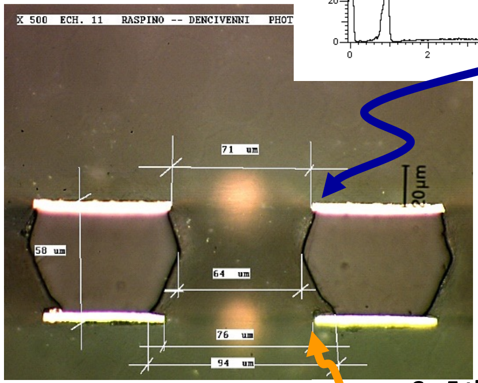




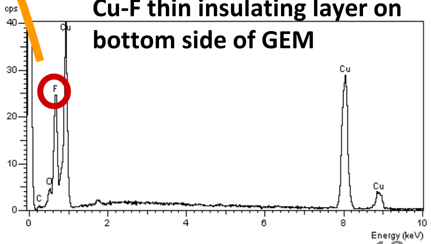
Fluorine found on the **bottom side of G2 and G3** ⇒ Cu-F compound forming a thin insulating layer

No fluorine on top side of GEM





Cu-F thin insulating layer on bottom side of GEM



Fluorine etching explains observed effects

REMINDER

The effects of fluorine etching is twofold:

1) widening of amplification holes

→ **gain reduction** (*)

2) Cu-F compound forming an insulating layer near the hole

→ **enhanced charging-up effects**

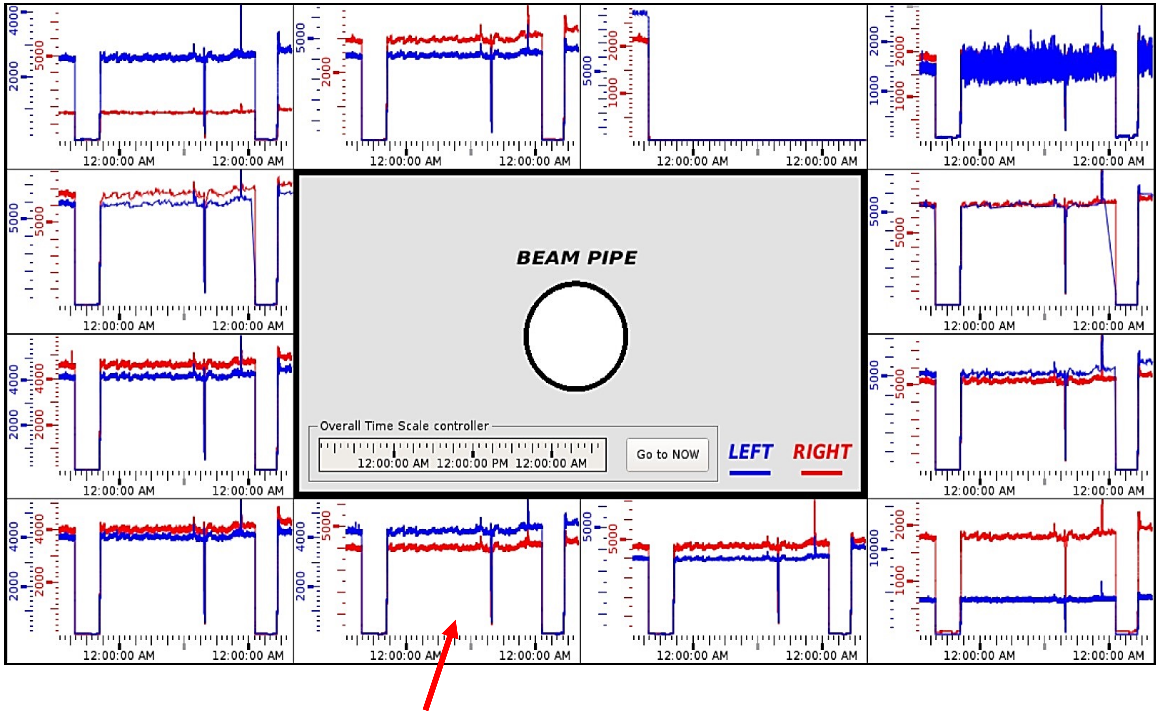
→ **rate capability reduction**

(*) S.Bachmann et al., NIM A 438(1999), 376-408

Irradiation effect @ LHCb

GEM detectors operation @ LHCb

Current up to 5 µA with beam collision

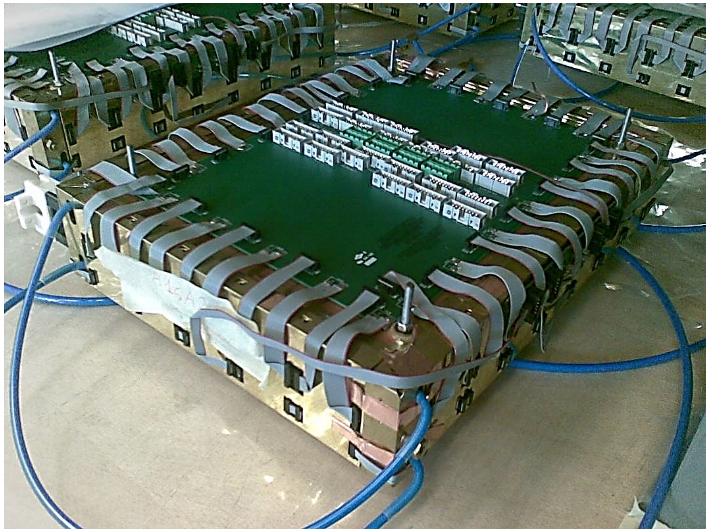


Station A15A1 (two GEM detectors called RIGHT & LEFT) has recently analysed after RUN1&RUN2

439 days colliding beams @ <Φ>~ 250 kHz/cm² @ G ~ 4*10³

→ Q_{int}~300 mC/cm²

Station A15A1



Gas enters on the anode of the RIGHT chamber and exit on its cathode.

Gas enters on the cathode of the LEFT chamber and exit on its anode.

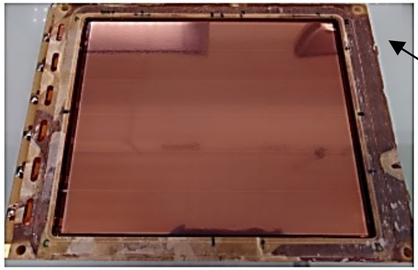
GEM Analysis @ CERN

RIGHT Ch.

Gas flow during operation →

LEFT Ch.

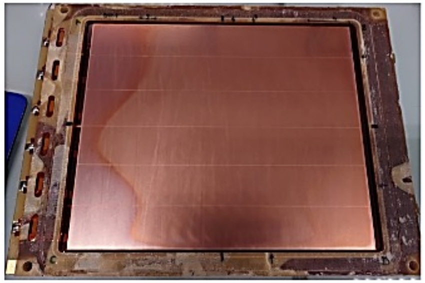
GEM #1



Cathode Gas Insert side

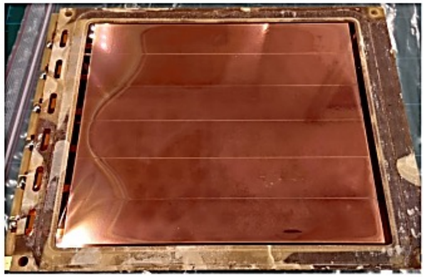
GEM #1

GEM #2



GEM #2

GEM #3



GEM #3

Anode Gas Insert side

RIGHT Anode

Gas flow during operation, entering from the anode

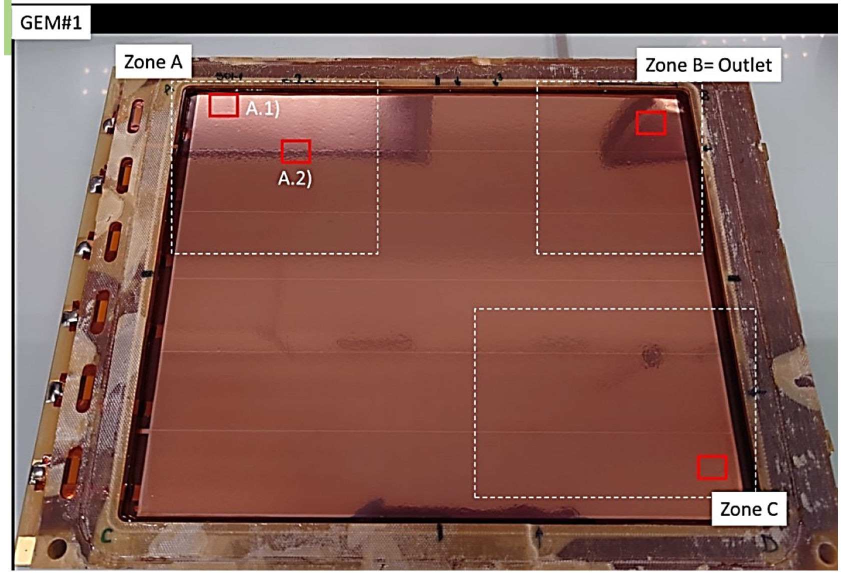
Anode Gas Insert side

Gas flow during operation, exiting from the anode

[Microscopic examination of triple-GEM detectors after installation and operation on LHCb with CF4-based gas mixture https://edms.cern.ch/document/2802473/1](https://edms.cern.ch/document/2802473/1)

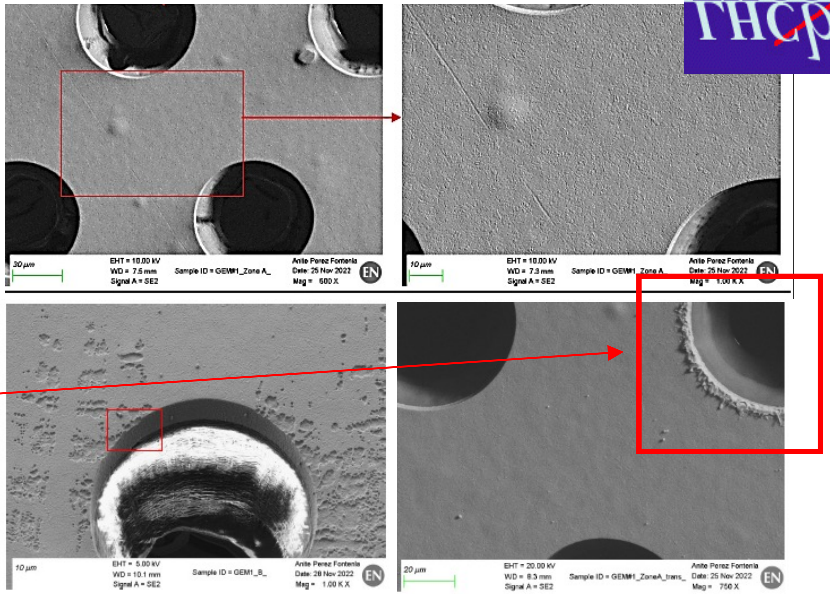
GEM #1 RIGHT analysis

Bottom view



General aspect and areas under study on GEM#1

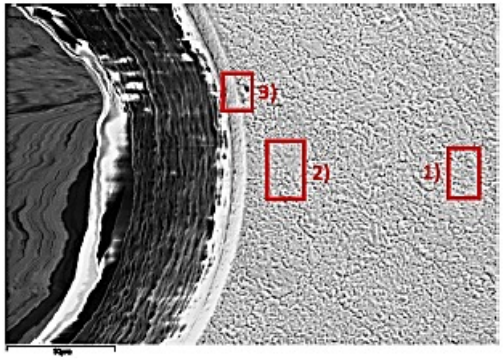
SEM images of the GEM#1 aspect on zone A.1

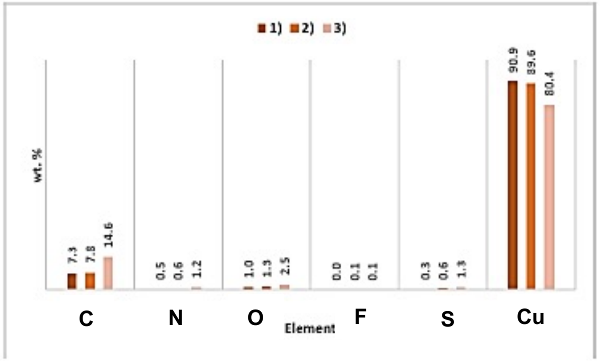


Holes and Cu surface are homogenous

Some Cu edges exhibit a molten aspect

Chemical analysis by EDS on the hole's surrounding areas pointed out **no residue** when approaching the hole's edge

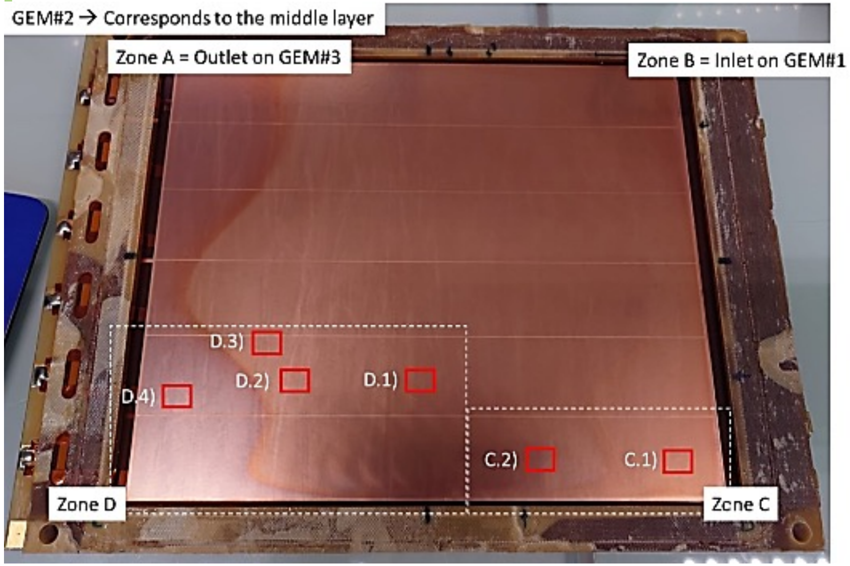




Elemental composition analysis by EDS on the hole surrounding area on zone B (GEM#1). The results are presented in wt. % and normalized

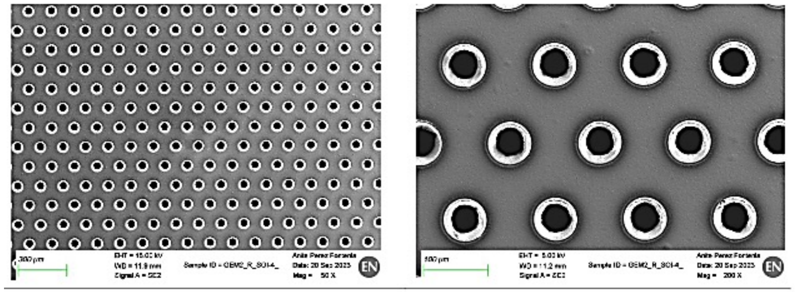
GEM #2 RIGHT analysis

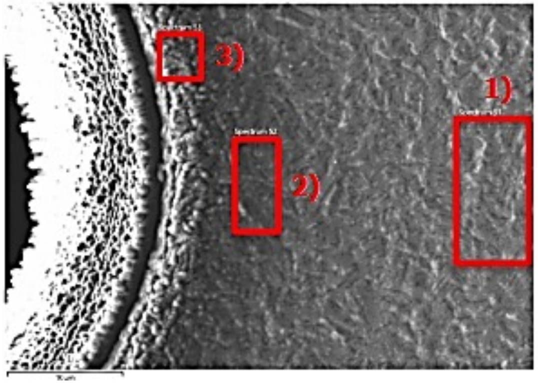
Bottom view

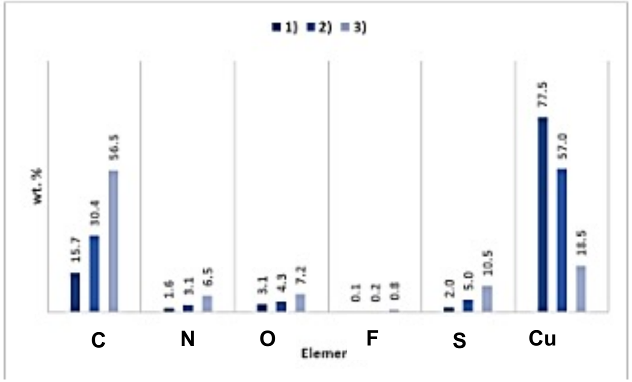


Holes and Cu surface are homogenous

SEM images of the GEM#2 aspect on zone D.4



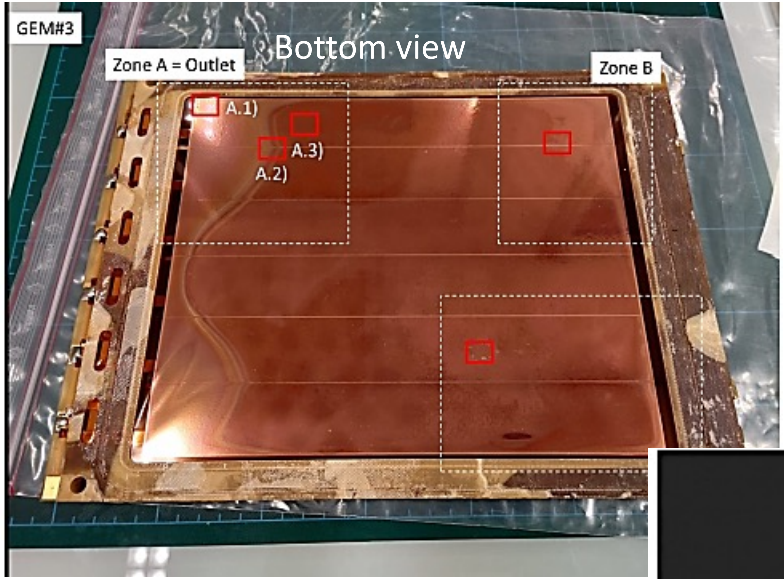




Elemental composition analysis by EDS on the hole surrounding area on zone D.4 (GEM#2). The results are presented in wt. % and normalized

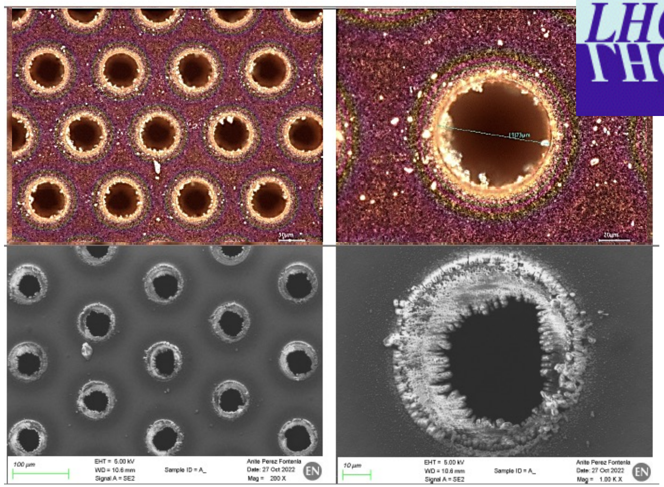
Chemical analysis by EDS on the hole's surrounding areas pointed out a small increasing content of C, N, **S (~10%)** when approaching the hole's edge

GEM #3 RIGHT analysis



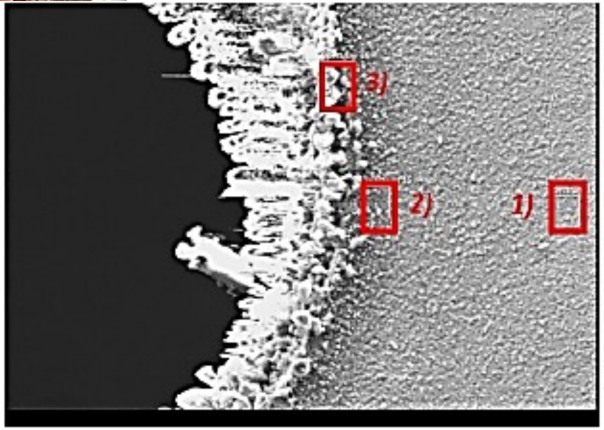
Micrometric particles are visible surrounding **the holes**

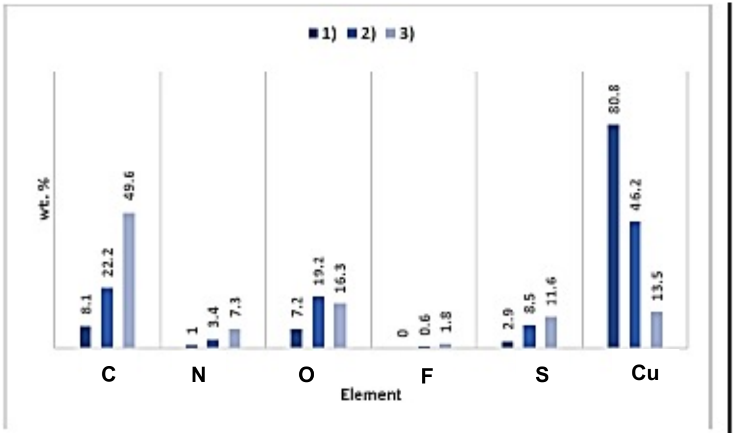
The **hole's edges** appear **rougher** as well as **the Kapton** into the holes



OM and SEM images of the GEM#3 A.2 zone

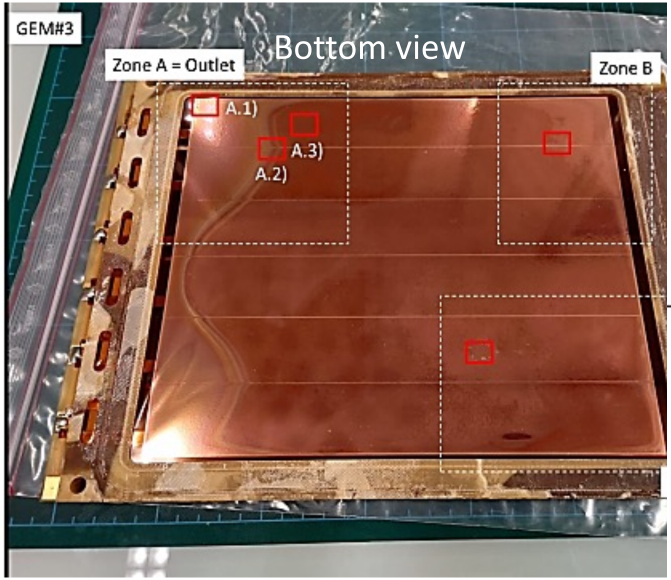
Chemical analysis by EDS on the hole's surrounding areas pointed out an increasing content of C,N, **F (<2%) and S (<12%)** when approaching the hole's edge

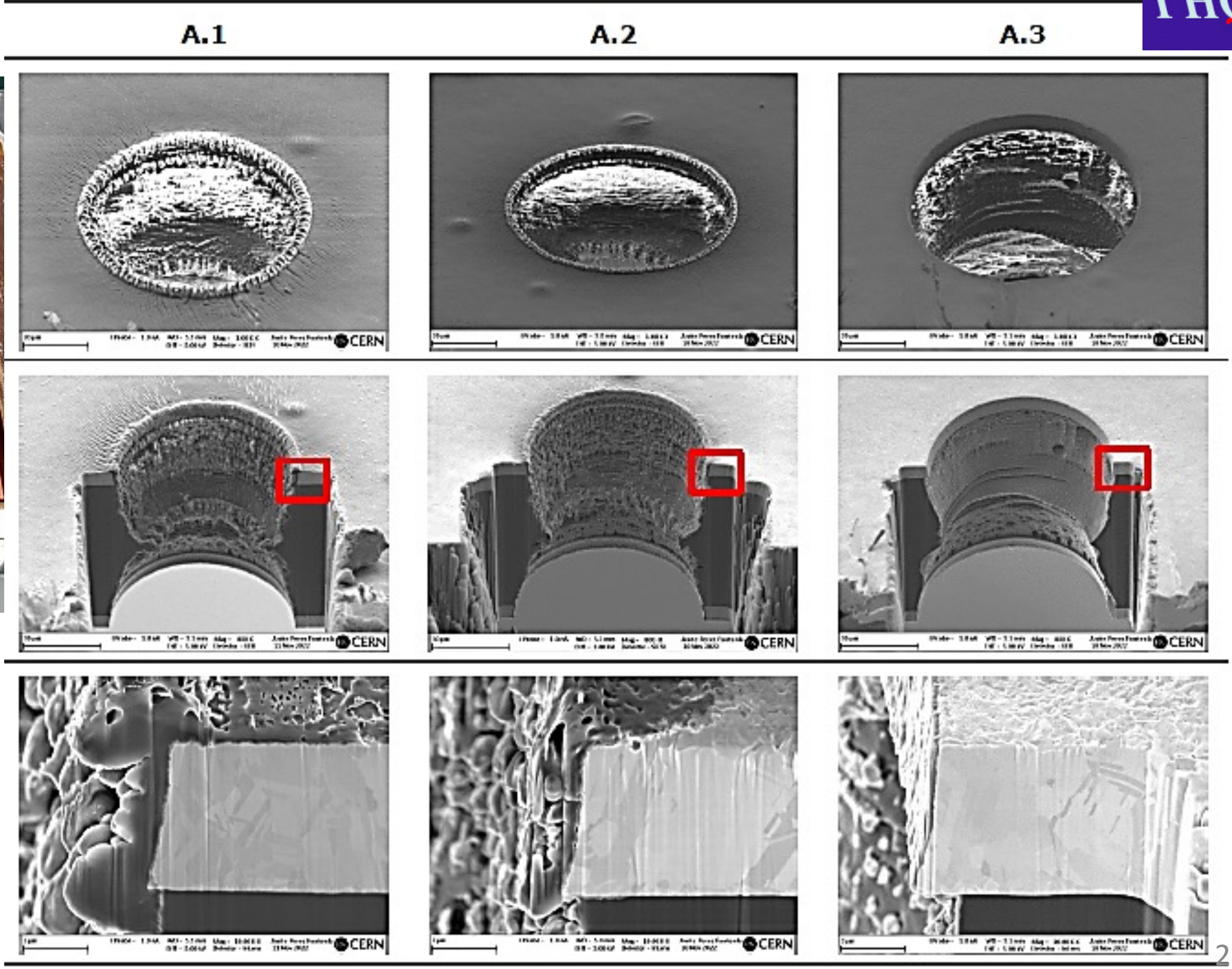




Elemental composition analysis by EDS on the hole surrounding area on zone B (GEM#1). The results are presented in wt. % and normalized

Sulphur residual on the bottom of GEM #3 RIGHT





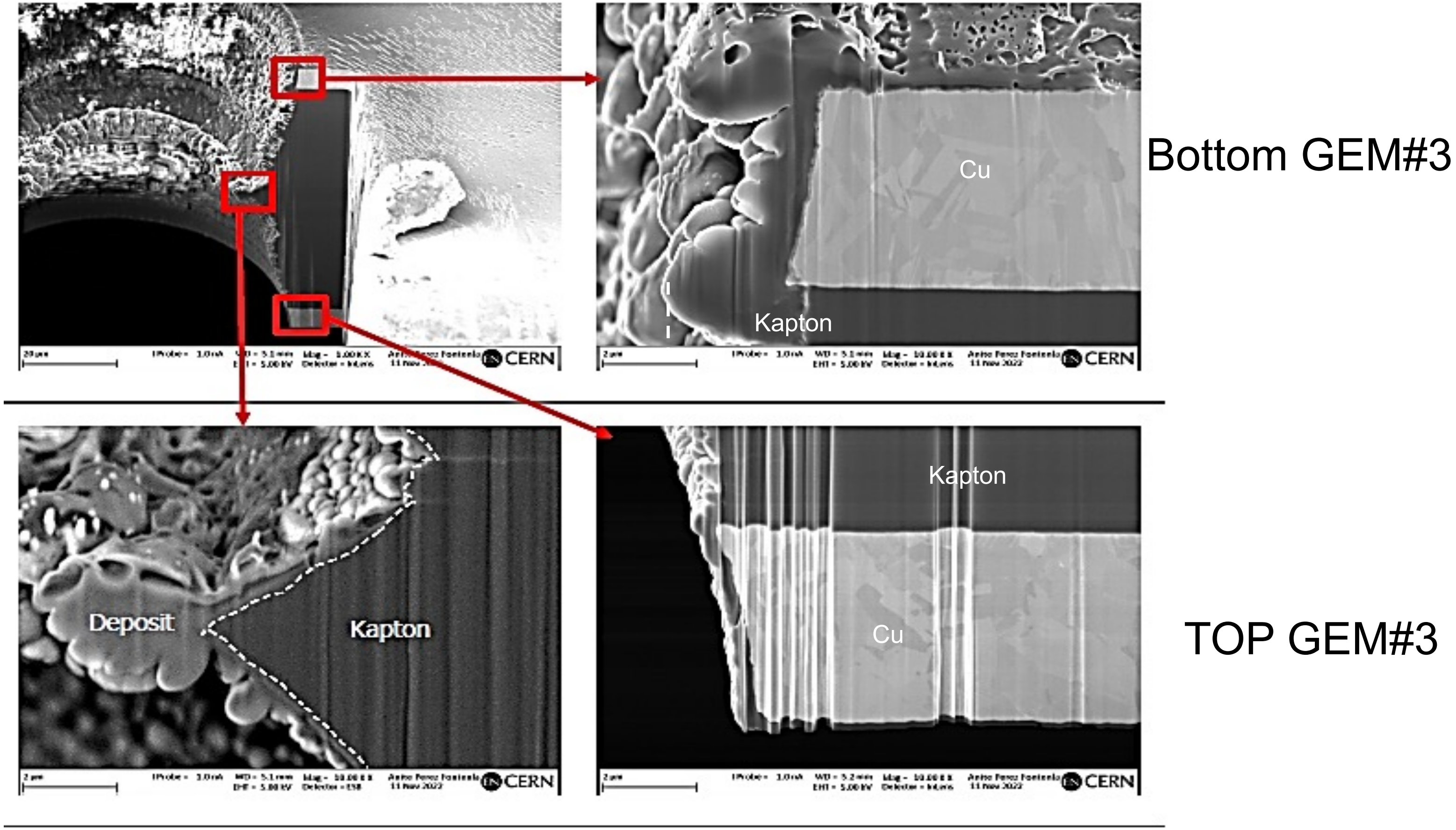
FIB-SEM inspection confirmed that **the residue** observed on the hole's edges **is also deposited into the hole internal surface** covering the Kapton and modifying the hole's geometry:

- A.1 ~ 2 µm,
- A.2 ~ 1 µm,
- A.3 nanometric

Sulphur residual in a hole of GEM#3

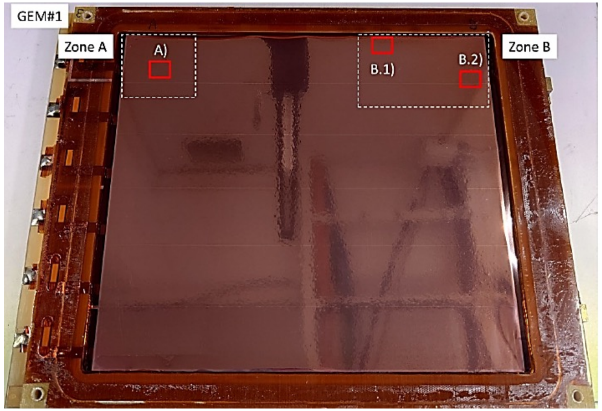
Zoom of A.1 hole inside the stained area of GEM#3

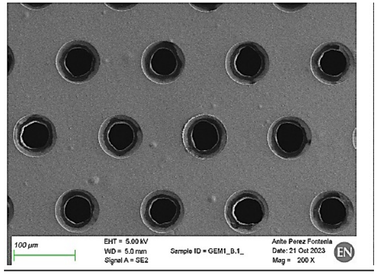
A **thinner deposit at the bottom** of the hole suggests a potential direction of the flow contamination

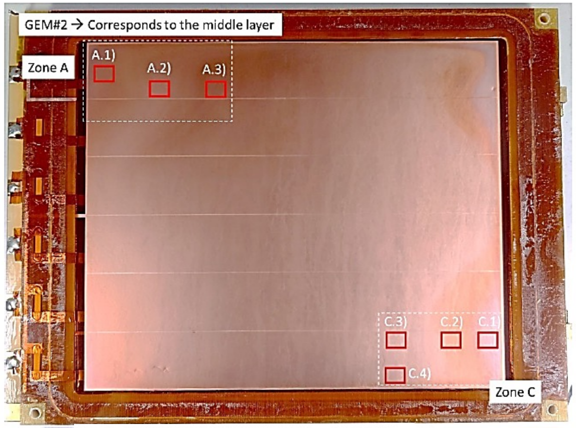


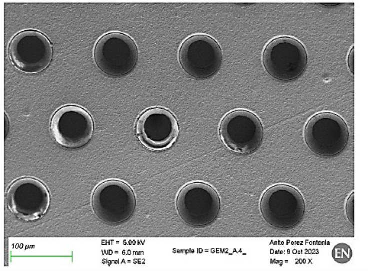
SEM images of the hole cross section on GEM#3 location A.1

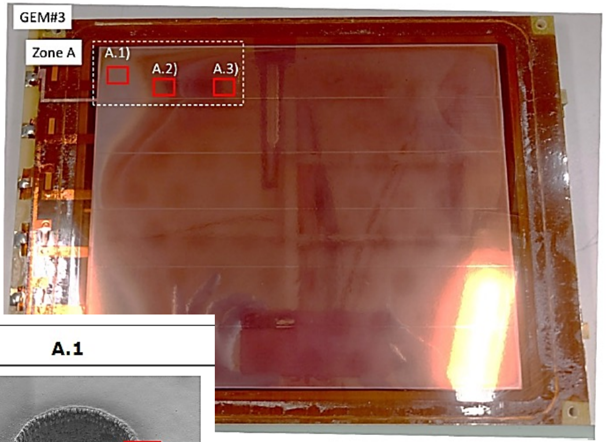
GEM Ch. LEFT analysis

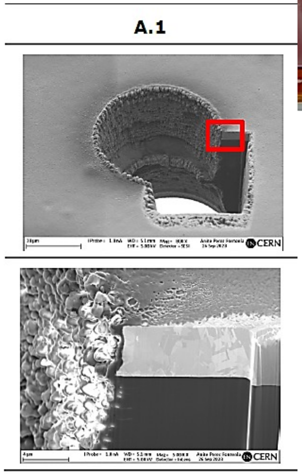


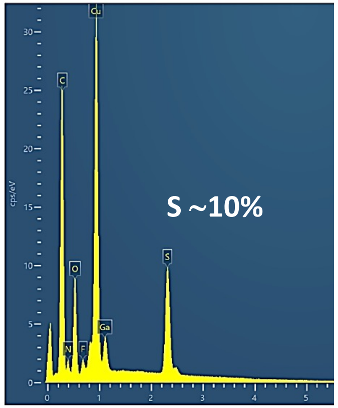




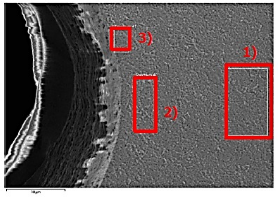


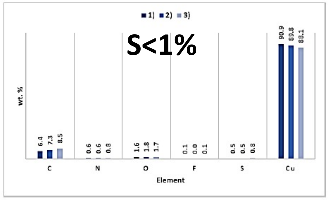




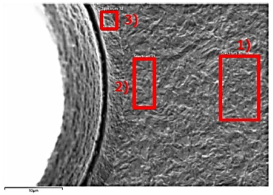


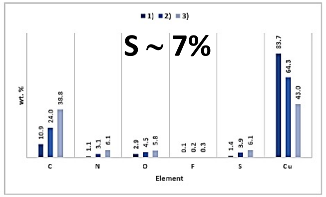
A



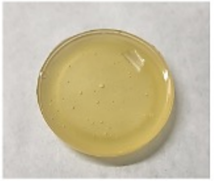


A.1





Search detection sulphur on ARALDITE 2012



Sample 1 –ARALDITE 2011

Sample 2 – ARALDITE 2012

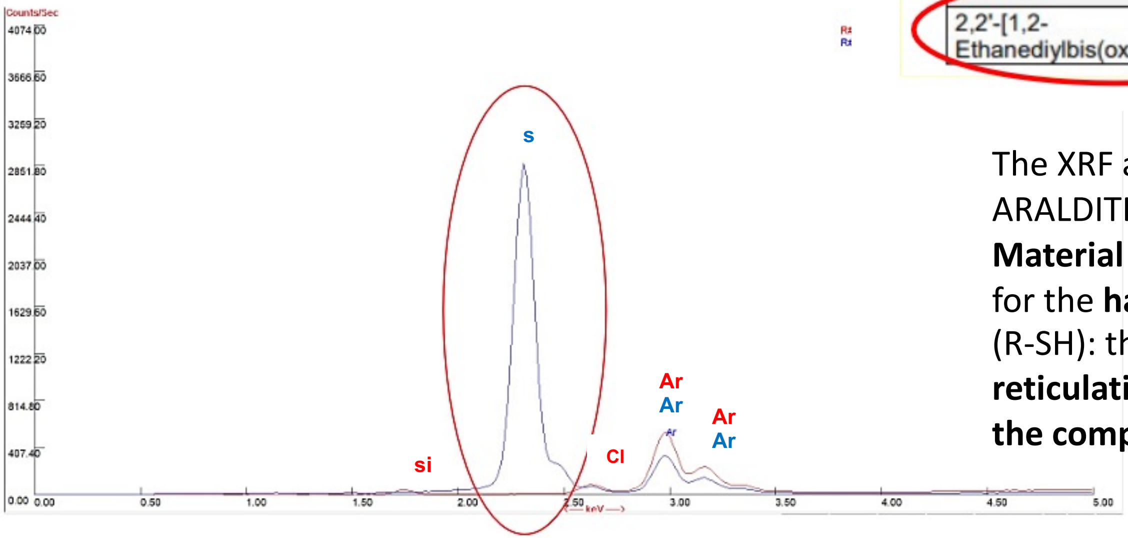
SECTION 3: Composition/information on ingredients

3.2 Mixtures

Hazardous components

Chemical name	CAS-No. EC-No. Index-No. Registration number	Classification	Concentration (% w/w)
2,2'-[1,2-Ethanediylbis(oxy)]bis(ethanethi	14970-87-7 239-044-2	Acute Tox. 4; H302 Acute Tox. 4; H332	>= 2.5 - < 10

- ARALDITE 2011
- ARALDITE 2012



The XRF analysis highlight presence of sulphur on the ARALDITE 2012 unlike sample 1 (*).

Material Safety Data Sheet shows that the molecule 2.2' used for the **hardener** was composed with thiol chemical function (R-SH): the sulphur can come from it because during the **reticulation polymerization the sulphur remains present in the compound's skeleton**

(*) https://edms.cern.ch/document/2962125/1

Summary & Outlook

The Casaccia high irradiation global test, with **Ar/CO2/CF4=45/15/40**, was understood :
- the **F-etching** observed is correlated with **low gas flow**
- **F-etching effects** mainly on the **third GEM** with fluorine deposits near the copper holes bottom edge

With Ar/CO2/CF4=45/15/40, the GEM detectors showed after a **Q_{int}~300 mC/cm^2**:
- **sulphur** residual deposit mainly on the **third GEM**, probably due to the **outgassing of the ARALDITE 2012;**
- **no effect of F-etching on GEM**;

→ **No major performance loss** observed during LHCb operation
→ **< 100 nA dark current** recorded on GEM#3

The work on the GEMs chambers **is not conclusive** but **has just begun:**

→ Investigation of the **chemistry of the sulphur** in the **detector operation**
→ Look at possible **effects of sulphur** on other detector components (anode & cathode)
→ **Analyzing other detectors installed @ LHCB is foreseen**

We are very grateful to **Anité and Stefano** (EN-MME-MM CERN Group) for their **excellent work**

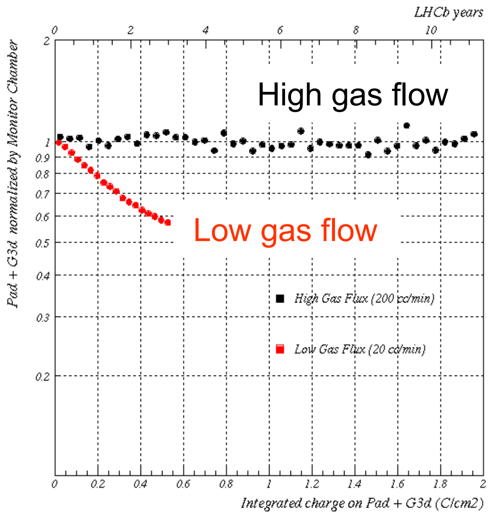
Aging summary

This table summarizes the detector anode current normalized to detector total gas flow.

It is clear from the table that heavily irradiated chambers at Casaccia were operated with a low gas flow to total_detector current ratio, which might have been the cause of the observed aging behavior.

Aging Test	Gas Flow (cc/min)	Total Current (□A)	R=Current/gas flow (**)
X-rays (*)	100	0.4 □ 0.8	0.004 □ 0.008
Casaccia C	350	36	0.10
Casaccia A , B	350	800 □ 1000	2.3 □ 2.9
LHCb M1R1	100	8	0.08
Sauli (Hamburg)	80	3 □ 12	0.04 □ 0.15 (no CF_4)

(*) no global test.
(**) might also depends on irradiation spot and chamber volume.



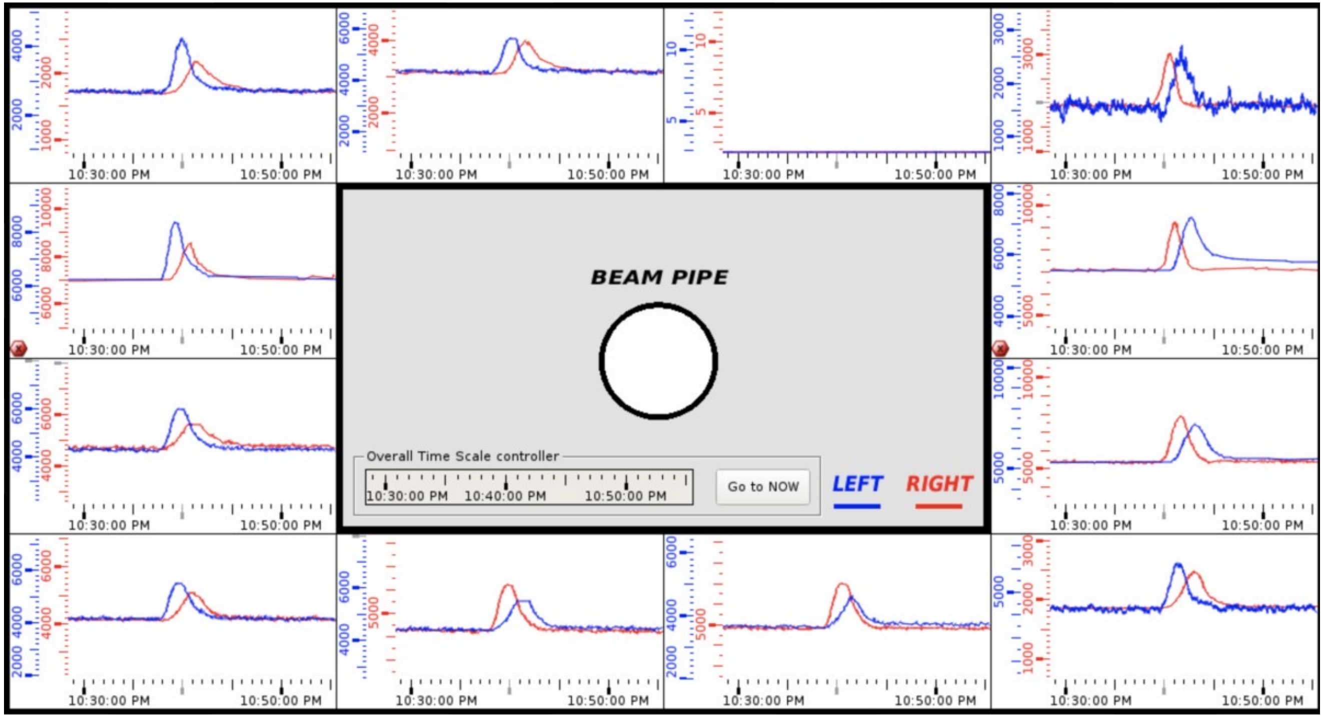
To reproduce the Casaccia test results, a small chamber has been irradiated with X-rays (total current ≅ 2 µA on ≅ 1 cm^2 irradiated spot) flushed with a low gas flow rate (20 cc/cm)

A current drop of ~ 40% for a 0.55 C/cm^2 integrated charge (~3 LHCb years) is found on the low gas flow measurement

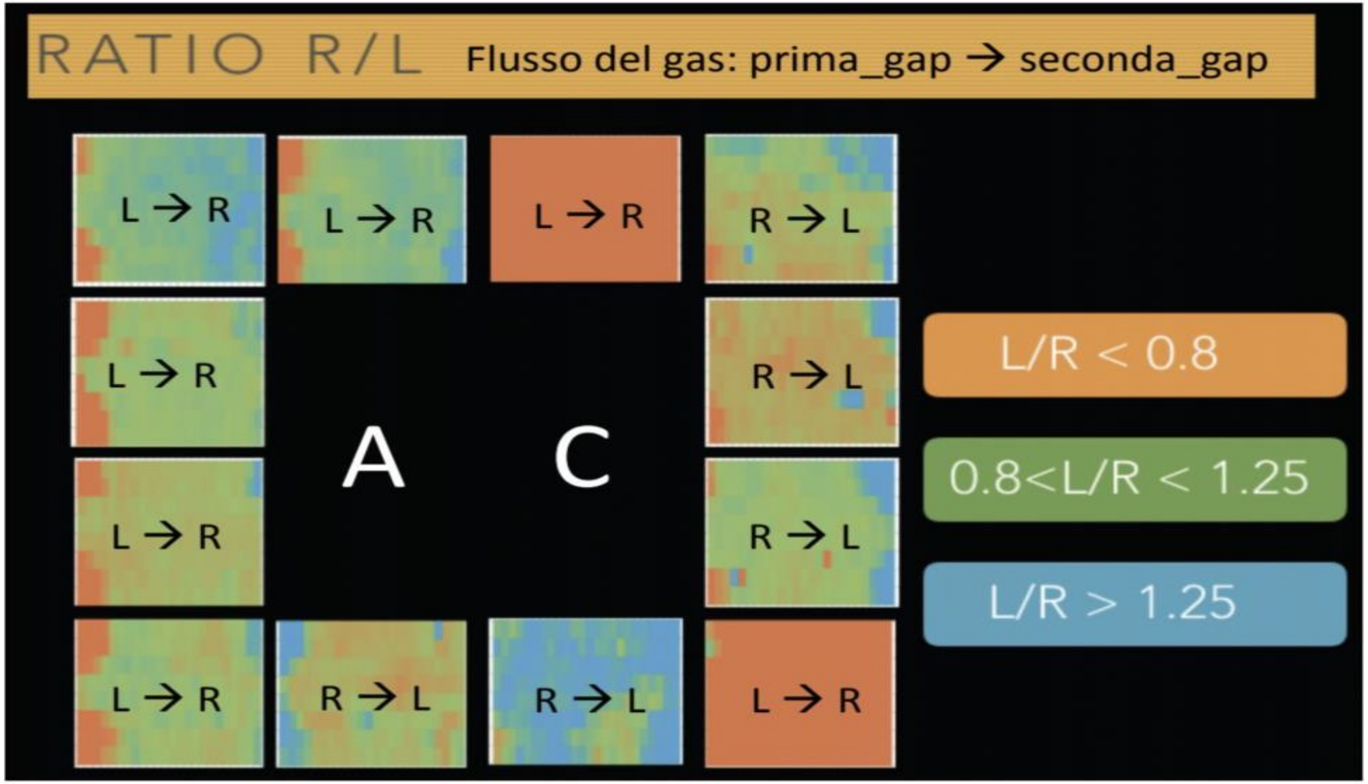
NO current drop is observed on the high gas flow measurement

How we used the gas instabilities

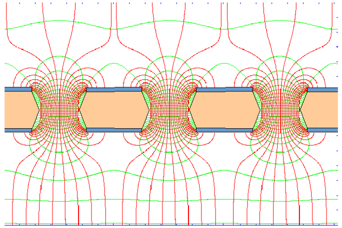
- The bad gas mixture reached the GEMs after approximately 30’
- Since the two gaps on each GEM detector are connected in series, the bad gas mixture entered one gap before the other
- The current increased first in the gap where the mixture enters the GEM chamber



Profiting of gas instabilities



Global aging test: set-up



⇒ to check the compatibility between the construction materials (detector and gas system) and the gas mixture

⇒ large amount of CF_4 (40%) ⇒ Global Aging Test

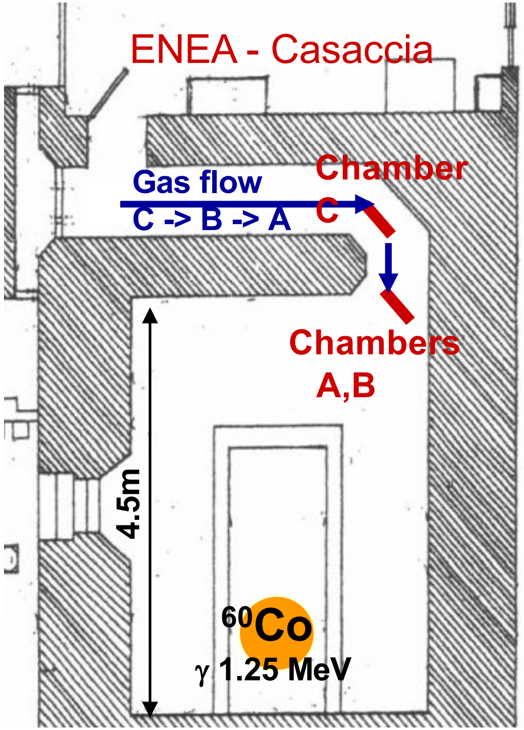
A full size (20x24 cm^2) prototype (C) in low irradiation position ~ 1 MHz/cm^2, and 2 full size prototypes in high irradiation position, ~ 15 MHz/cm^2 (chamber A) and ~ 20 MHz/cm^2 (chamber B)

$Ar/CO_2/CF_4$ (45/15/40) at reference Gain ~ 6x10^3

monitored H_2O (± 1ppm), T (± 0.1°K), and external P (± 0.1mbar)

gas flows: C -> B -> A -> T/H_2O Probe -> Out initially Φ_{gas}= 200 cc/min, then Φ_{gas}= 350 cc/min

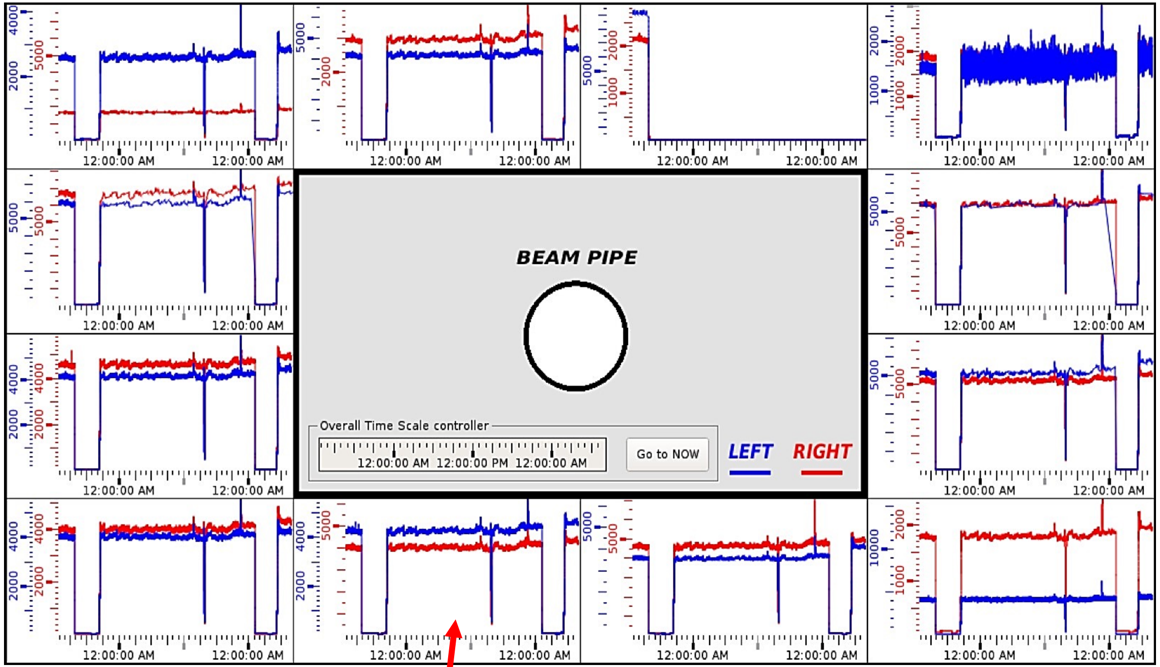
gas inlet line ⇒ stainless-steel tubes
exhaust gas line ⇒ polypropylene tubes (not hygroscopic)

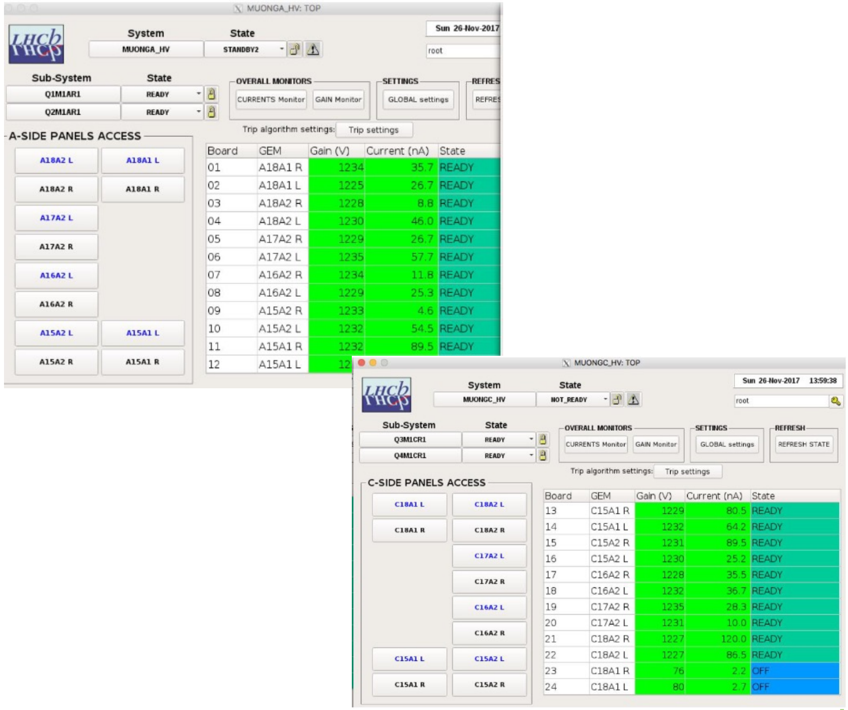


GEM detectors operation @ LHCb

Current up to 5 μA with beam collision

HV GEM Monitor





Station A15A1 (two GEM detectors called RIGHT & LEFT) has recently analysed after RUN1&RUN2

439 days colliding beams @ <Φ>~ 250 kHz/cm² @ G ~ 4*10³

→ Q_{int}~300 mC/cm²

Summary & Outlook

The Casaccia high irradiation global test, with **Ar/CO2/CF4=45/15/40**, was understood :
- the **F-etching** observed is correlated with **low gas flow**
- **F-etching effects** mainly on the **third GEM** with fluorine deposits near the copper holes bottom edge

→ Detectors, even after a severe irradiation in bad conditions, exhibit good time and efficiency performance

→ Further tests have shown that **no F-etching** occur if **the gas flow is properly set**

With Ar/CO2/CF4=45/15/40, the GEM detectors showed after RUN1 and RUN2 (**Q_{int}~300 mC/cm^2**):
- **sulphur** residual deposit mainly on the **third GEM**, probably due to the **outgassing of the ARALDITE 2012;**
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