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

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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

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

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

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

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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)

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

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 cm²) prototype (C) in low irradiation position ~ 1 MHz/cm²,
2 full size prototypes in high irradiation position: ~ 15 (chamber A) and ~ 20 MHz/cm² (chamber B)
Ar/CO2/CF4 (45/15/40) at gain ~ 6x10³ & 35 irradiation days

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)
Global aging test: results

Integrated charge:
detector C ~ 0.16 C/cm² ⇔ 1 LHCb y
detector B ~ 1.6 C/cm² ⇔ 8.5 LHCb y
detector A ~ 2.2 C/cm² ⇔ 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/cm², while Chamber A & B show ΔG/G ~ -10% after the same integrated charge!
Preliminary conclusions

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/cm² 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 (CF₄ 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 $\sim 3\text{MHz/cm}^2$ (expected LHCb rate $\sim 0.5\text{MHz/cm}^2$)

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<tr>
<th>Process</th>
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<td>$\sim 55%$</td>
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<td>Rate reduction</td>
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<td>$\sim 40%$</td>
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Chamber A (with the larger gain loss) shows ONLY a shift of the working point of $\sim15\text{V}$

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Test on aged chambers

Aged chambers exhibit NO rate capability loss up to $\sim 3\text{MHz/cm}^2$ (expected LHCb rate $\sim 0.5\text{MHz/cm}^2$)

- Chamber A: $\sim 55\%$
- Chamber B: $\sim 32\%$

- Gain reduction $\sim 30\%$
- Rate reduction $\sim 40\%$

- Total reduction $\sim 85\%$

Chamber A (with the larger gain loss) shows ONLY a shift of the working point of $\sim 15\text{V}$

To reproduce the Casaccia test results, a small chamber has been irradiated with X-rays (total current $\sim 2\ \mu\text{A}$ on $\sim 1\ \text{cm}^2$ irradiated spot) flushed with a low gas flow rate ($20\ \text{cc/cm}^2$)

- A current drop of $\sim 40\%$ for a $0.55\ \text{C/cm}^2$ integrated charge ($\sim 3\ \text{LHCb years}$) is found on the low gas flow measurement
SEM analysis & X-ray spectroscopy on aged chambers

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

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

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

RUN1&RUN2: particle flux & detector operation
439 days colliding beams @ $<\Phi> \sim 250 \text{ kHz/cm}^2$ @ $G \sim 4 \times 10^3$
$\rightarrow Q_{\text{int}} \sim 300 \text{ mC/cm}^2$

Average number of hits of physical FFE channel per cm$^2$ per trigger

GEM stations efficiency (25ns):
- After commissioning
- End of RUN2

$\Delta\text{Efficiency} < 1\%$
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 @ \( <\Phi> \sim 250 \text{ kHz/cm}^2 \) @ \( G \sim 4 \times 10^3 \)

\[ Q_{\text{int}} \sim 300 \text{ mC/cm}^2 \]

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

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GEM #1 RIGHT analysis

Bottom view

General aspect and areas under study on GEM#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

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.

Holes and Cu surface are homogenous

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

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GEM #3 RIGHT analysis

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

Micrometric particles are visible surrounding the holes.

The hole’s edges appear rougher as well as the Kapton into the holes.

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#3). The results are presented in wt. % and normalized.
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

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GEM Ch. LEFT analysis

S \sim 10\%

S < 1\%

S \sim 7\%

S \sim 10\%
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
The Casaccia high irradiation global test, with $\text{Ar/CO}_2/\text{CF}_4 = 45/15/40$, 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 $\text{Ar/CO}_2/\text{CF}_4 = 45/15/40$, the GEM detectors showed after a $Q_{\text{int}} \sim 300 \, \text{mC/cm}^2$:
- 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
  - $< 100 \, \text{nA}$ 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.
Many thanks for your attention
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.

<table>
<thead>
<tr>
<th>Aging Test</th>
<th>Gas Flow (cc/min)</th>
<th>Total Current (µA)</th>
<th>R = Current/gas flow (**)</th>
</tr>
</thead>
<tbody>
<tr>
<td>X-rays (*)</td>
<td>100</td>
<td>0.4 ± 0.8</td>
<td>0.004 ± 0.008</td>
</tr>
<tr>
<td>Casaccia C</td>
<td>350</td>
<td>36</td>
<td>0.10</td>
</tr>
<tr>
<td>Casaccia A, B</td>
<td>350</td>
<td>800 ± 1000</td>
<td>2.3 ± 2.9</td>
</tr>
<tr>
<td>LHCb M1R1</td>
<td>100</td>
<td>8</td>
<td>0.08</td>
</tr>
<tr>
<td>Sauli (Hamburg)</td>
<td>80</td>
<td>3 ± 12</td>
<td>0.04 ± 0.15 (no CF₄)</td>
</tr>
</tbody>
</table>

(*) 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 cm² irradiated spot) flushed with a low gas flow rate (20 cc/cm²)

A current drop of ~ 40% for a 0.55 C/cm² 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 CF₄ (40%) ⇒ Global Aging Test

A full size (20x24 cm²) prototype (C) in low irradiation position ~ 1 MHz/cm², and 2 full size prototypes in high irradiation position, ~ 15 MHz/cm² (chamber A) and ~ 20 MHz/cm² (chamber B)

Ar/CO₂/CF₄ (45/15/40) at reference Gain ~ 6x10³

monitored H₂O (± 1ppm), T (± 0.1°K), and external 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)
Station A15A1 (two GEM detectors called RIGHT & LEFT) has recently analysed after RUN1&RUN2 439 days colliding beams @ \(<\Phi>\sim 250\ kHz/cm^2\ @ G \sim 4\times10^3\) → \(Q_{int} \sim 300\ mC/cm^2\)
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/cm²):

- 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

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