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Studies on a case of aging in a gaseous particle detector

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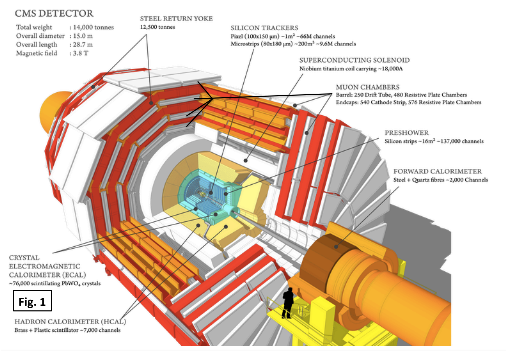
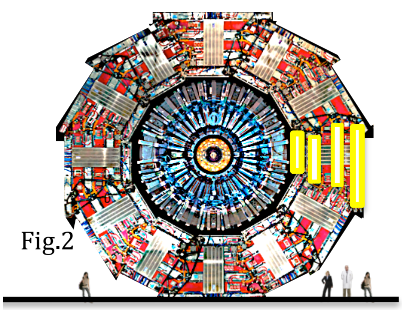
ABSTRACT

This note reports about activities and results obtained at the Legnaro National Laboratories of the INFN (LNL) and at the Physics and the Chemistry Departments of the University of Padova in a study of a case of aging in gaseous particle detectors. In September 2015 a first exposure of a spare CMS MU Barrel Drift Chamber (MB1 in the following) to an intense flow of ~700 keV photons showed clear signs of a fast loss of gain and of deterioration of the overall performance. After a cumulated irradiation equivalent to that expected to be collected at the end of the planned High Luminosity run of the CERN Large Hadron Collider (HL LHC) the loss of gain reached values larger than 70%.
The irradiation was performed at the Gamma Irradiation Facility (GIF) at CERN [1]. The study was triggered by the request of the CMS Technical Management for a check of the behavior of the CMS detector up to the integrated luminosity of 3000 fb^{-1}, equivalent to a radiation dose of 840 mG. The CMS MU Barrel Drift Chamber, designed in the late 1990 to stand the integrated luminosity of the foreseen ten years of LHC operation, are requested now to stand an integrated luminosity10 times larger. The CMS Detector is shown in fig.1) and 2) : the Muon chambers are visible inserted in the iron of the return yoke of the large central solenoid magnet.





A TRANVERSE SECTION OF CMS

The CMS yoke is equipped with four layers of Barrel Muon Chambers named MB1,MB2,MB3,MB4 starting from the innermost ones.. The 4 of a same sector are evidenced in yellow. The most exposed to the irradiation coming from the LHC interactions is the most internal layer , MB1 . The diameter of CMS measures 14 m.

Only about 10% of the Barrel chambers are expected to be exposed to such a large radiation flow but the large size of the loss was surprising and a strong hint to more accurate measurements and analysis. A study has been done in LNL and in the Departments of the University of Padova exploiting available instruments,

technologies and experience suitable for a deep analysis of the data to search for procedures to control and possibly mitigate the size of the phenomenon.
The search has not been conclusive yet, but the results of the new analysis and the collected information allows the planning of two crucial and hopefully conclusive checks at the GIF and in CMS during the year 2021

INDEX:

1) INTRODUCTION

Aging in wire gaseous particle detectors shows up as a loss of gain or by the appearance of anomalous behaviors (discharges) [2] due to the deposit of some compounds on the electrodes. The aging is triggered by the process of huge multiplication (O $10^4 \sim 10^5$) of the electrons made free in the gas of the detector by the passage of an ionizing particle. The multiplication process develops in the last few hundreds micron of the electrons path toward the anode electrode, a thin wire in this case, and is induced by the local very high electric field that allows the drifting electrons to gain enough energy to ionize the gas. This multiplication is needed to generate an electric signal large enough to allow the detection of the particle passage. Heavy molecules present in the gas can break in this very localized but high temperature plasma and fragments can reach and attach to the wires or cathodes. Aging may develop only when the detector is active i.e. with the High Voltage switched on.
Molecules of contaminant compounds can be present in the chosen gas mixture, can be introduced in the chamber along the gas pipes, or generated inside the chamber by materials present in their active gas volume. The last case seems to be at the origin of the phenomenon discussed in this note.
In the following Paragraph 2) contains a short description of the CMS DT Chambers, Paragraph 3) describes the first irradiation results. Paragraph 4) presents a couple of small detectors designed in LNL to integrate the big chambers set up at GIF. Paragraph 5) deals with the irradiation procedures and plans.
Paragraph 6) describes the tests and the analysis methodology. The outcome of the irradiations and the results of the studies on the nature of the aging deposits on the wires are presented in paragraphs 7) and 8). Summary, conclusions and some proposals are in Paragraph 9)

2) The CMS Barrel Muon Drift Tubes Chambers:
A medium size CMS Muon Barrel DT chamber is a device 250x250 cm^2 large, 30 cm thick and 1000 kg heavy [3].
It is composed by twelve layers of tubes: the tubes of a layer are obtained gluing two large aluminum plates to sandwich an array of parallel Aluminum I-Beams. (Fig.3)
A layer is made of 60~ 70 tubes with a cross section of ~4,2 X 1,2 cm^2 and ~ 250 cm long, with a volume of ~ 1 liter each. The 12 layers are grouped in three sets of four layers (Super Layers, SL) with independent electrical and gas circuits. They constitute a set of three fully independent detectors. The construction must guarantee that the absolute position of a wire in the reference frame of the SL is known within 100 micrometer. This figure represents about 40% of the nominal track position resolution.
The three SLs are built and tested individually and than glued together and to a thick Honeycomb plate to shape the final Chamber (Fig.4).

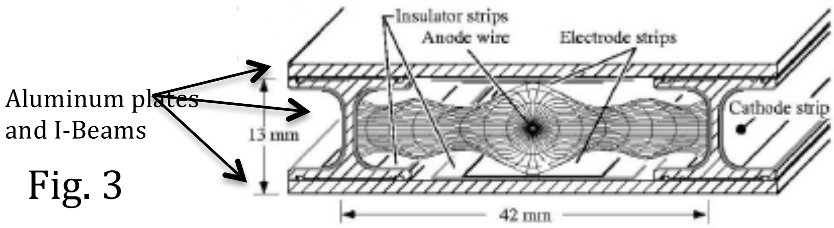


Fig. 3

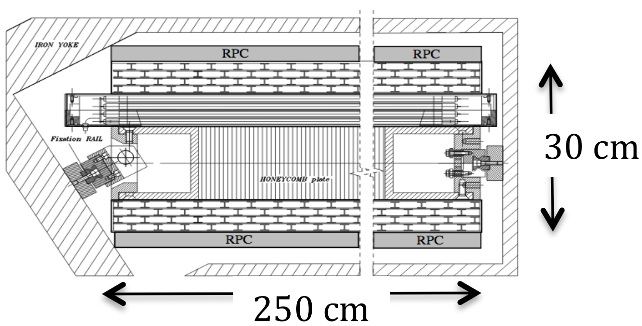


Fig. 4

Fig.3 A DT tube cross section: indicated are the cathode and electrode strips, the Al plates and I-Beams .
Fig.4 A DT chamber in position in the CMS iron yoke. One can see 2 SL with the wires along the LHC beam lines, perpendicular to the figure plane, and the third SL with the wires in the figure plane . In between, the honeycomb plate with supports attached to the iron yoke. Also shown are the RPC detectors.

The drift field in each tube is generated by the wire, that acts as anode, and by four electrodes acting as cathodes and made of thin aluminum strips glued two to the plates in front of the wires and two on the sides of the beams. Thin Mylar ribbons ensure the insulation of the electrodes from the grounded aluminum plates and I-Beams. The cathodes strips are glued to the Mylar ribbons and the Mylar ribbons to the Aluminum.
A suitable choice of the HV settings of this arrangement of electrodes (wire 3500~3600 Volt, central strips 1800 V, cathodes -1200 V) allows to obtain in the drift cell the electric field whose lines are visible in Fig. 3). It is homogenous enough to keep the drift velocity of the electrons within a window that allows a linear

relationship between the distance from the wire of the ionizing track and the time spent by an ionization electron to reach it.
A peculiarity of this design is that the Front End Amplifiers and the HV Distribution Boards and the related cabling are contained inside the active gas volume of each Super Layer. So the volume of each Super Layer contains materials that could release molecules of potentially dangerous compounds in the gas. This unusual arrangement was necessary to obtain the largest active area of the chamber compatible with the large dead regions generated by the structure of the Barrel Iron Yoke of CMS. The filling gas is a mixture of Argon and CO_2 in the ratio of 85/15. This mixture is known to be no contaminant.
All materials and parts were chosen after careful tests [4] which proved they were not generating a sizeable aging after a radiation dose equivalent to an integrated luminosity of 500 fb^{-1}, about twice the total integrated luminosity expected at the time of the project but an order of magnitude smaller compared to the HL LHC.

3) OUTCOME OF FIRST IRRADIATIONS (MB1 and MB2)

Two CMS Barrel Drift Tubes Chambers with a sensitive area as large of about 6 m^2 were exposed in accelerated tests of aging at the CERN GIF in order to study their behavior when operated at the High Luminosity LHC. High intensity irradiation generating a large rate of hits and high currents in the wires is considered necessary to produce in a reasonably short time the effects of the much longer operation at lower intensity in the long time of life of the experiment. The common assumption supporting that procedure is that the effect on the wires depends on the value of the integrated current and not on the time of integration. The observation that the actual figure of the currents in the chambers of the running CMS experiment at LHC are compatible up to 2x10^34/(cm^2sec) with a linear relationship with respect to the LHC Luminosity encourages their extrapolation to the values of 5x10^34/(cm^2sec) expected in the High Luminosity run. Under the assumption that aging depends on the currents and charge absorbed by the wires and not on the specific source of ionizing agents it is possible to represent the results at the GIF in function of the luminosity expected at HL LHC. The typical effect of the aging of the detector in the case under study was the coating of the wires by a thin layer of very high resistivity and non-solvable compounds; the effect appears to be not reversible.
A first chamber (MB1) was irradiated in summer 2015 and the irradiation of a second one (MB2) started two years later. In the GIF a set of absorbers allows to tune the intensity of the radiation within a very large set of values. The first chamber was exposed to a flow hundred times larger than the HL LHC expectation. A more prudent choice on the source attenuation was done for the MB2 irradiation: the absorbers were set to generate in the chamber wires a current about 10 times larger than that expected at High Luminosity LHC. In the second tests, on the MB2, only two layers (L1 and L4) of SL1 were active all the time until the cumulated charge reached the value expected at the end of the HL LHC run. The other two (L2 and L3 of SL1) were switched on time to time very shortly (to prevent any sizeable aging) to have a measure of the current at the given source rate to be compared to the one driven by the aging layers and for calibration purposes. During a calibration run all the four Layers of SL1 were kept active: the procedure consisted of an irradiation at various source intensities (source scan, from fractions to ten times the value expected at HL LHC) accompanied by an HV scan. The equivalent ~3000 fb^{-1} integrated luminosity

on MB2 L1 and L4 irradiation lasted 11~12 weeks with a calibration run every week. The total irradiation cumulated by the reference layers had to generate a gain loss smaller than 1%.
The high stability of the outcomes of the many Source and HV scans performed on those layers, fully irradiated but essentially never active ("virgin" in the following), indicates that "passive" irradiation does not affect the chamber performance. A "virgin" layer is than available for further unbiased aging test.

In both chambers a loss of gain of 70% or larger was observed [5] after the exposure to an integrated radiation dose equivalent to that expected at the end of the HL LHC run**.** The test was performed under a constant irradiation intensity, ten times higher than that expected at HL LHC. The gain loss of the aging layers is obtained as the fraction of decrease of the current per wire, as measured by the power supplies, with respect to the current driven by the reference layers. It is assumed that the current due to the irradiation is proportional to the radiation intensity, which is measured and recorded by a small ionization chamber (RAMSES) installed in a suitable position in front of the chamber.
The values of the RAMSES are regularly recorded together with the current driven by the aging layers and, at regular intervals of time, by the non-aging reference layers. Also regularly recorded are the atmospheric pressure and the air temperature. Those parameters allow at any time a reliable estimate of the current that the aging layer had to drive in absence of aging.
A list of the used Units and typical values is given below to help the reading of the results showed in Fig 5) and Fig.6).
Currents are always given in micro Ampere per wire (μA/wire) and the integrated radiation in milli Gray (mG) or in the equivalent CMS integrated Luminosity units (inverse femtobarn ($fb^{-1)}$)). The instantaneous dose is given in multiple of mG/hour or in the equivalent multiple of the basic LHC luminosity of 1 $x10^{34}/(cm^2$ sec)
The time of operation at High Luminosity is assumed by convention to be of $6x10^7$ sec. The integrated radiation dose at the end of the HL LHC run is 840 mG or 3000 fb-^{1}. (1fb^{-1} is equivalent to 0,28 mG). At the expected current of 0,045 μA/wire and at the nominal wire voltage of 3550 Volt at end of HL LHC the wires of the most exposed chambers are expected to have absorbed 10.8 mC/cm.
The nominal HL LHC luminosity of 5x10^34/(cm^2sec) is obtained at 0,05 mG/h.
At the time of writing the peak luminosity at the experiment is 1,5 $x10^{34}/(cm^2$ sec) equivalent to 0.017 mG/h.
In the running CMS experiment the luminosity reached 1,5X10^34/(cm^2sec) with a current of 0,014 μA/wire (0,01 mA/w in average) which is three times lower than the 0,046 μA/wire current /wire at 5x10^34//(cm^2sec) and 30 times lower of the current driven by the virgin layers during the accelerated aging process shown in Fig.5).

In fig 5) the abscissa is the amount of radiation integrated by a reference non aged wire in units of CMS Luminosity in fb^{-1}. The vertical axis is the loss of current of the aging wire with respect to the current of the reference wires. One can see that the loss increases quickly and reaches a value of about 80% at the 3000 fb^{-1} expected at the end of the HL run.

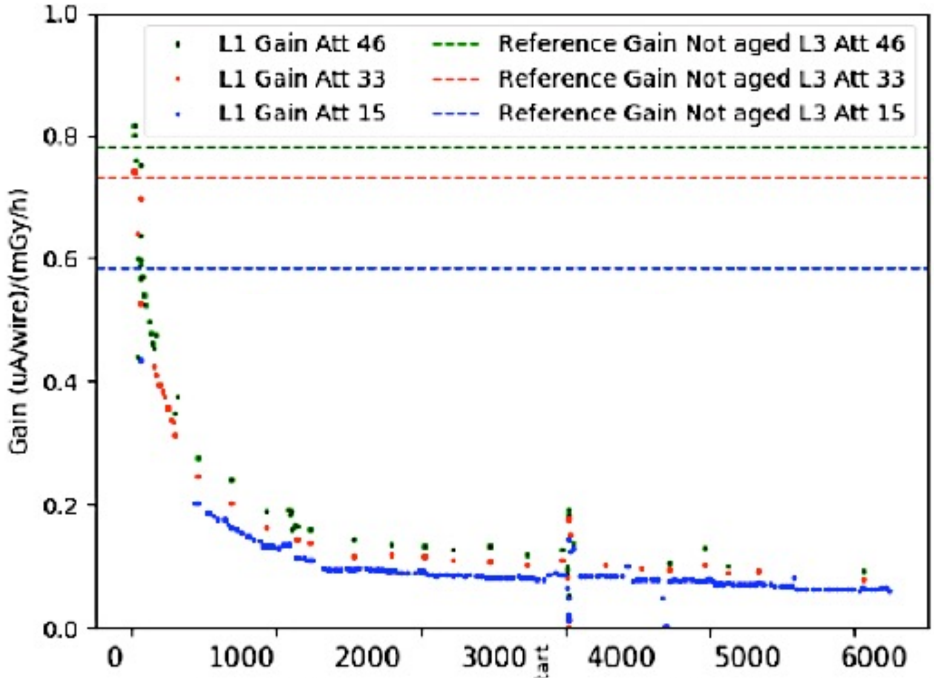


Fig.5 The loss of gain of the MB2 chamber at GIF versus the expected integrated Luminosity (in fb^{-1}) in HL LHC in the most exposed chambers of CMS. The total integrated Luminosity expected at the end of the HL LHC run is 3000 fb^{-1}

The losses in fig 5) have been measured at the unique and very high instantaneous radiation intensity chosen for the accelerated aging. This leaves open the possibility of a dependence of the loss on the intensity rate. The available source filters at the GIF allow measuring the loss of gain of the aged wires at a preselected set of source intensity, running from a figure below to above that of HL LHC. Those scans have been done at different values of the charge integrated during the process of accelerated irradiation. The Fig 6 A) shows the loss of gain of an aged wire with respect to the reference at some values of the integrated dose and in function of the intensity of the source. Naively one should expect the relative loss to be independent on the source intensity. The data show that this is not true and that a sizeable dependence exists in particular at intensities comparable to those expected at HL LHC. This phenomenon implies that the data shown in Fig 5) can't be used for an estimate of the current loss of an aged wire at intensities different from the unique one used during the accelerated test.

For high values of the instantaneous luminosity, or currents, the dependence of the loss of an aged wire on the source intensity is relatively weak but it quickly increases in the region of the HL LHC and below, the real interesting region. That region is the most critical for the measure due to the distortions introduced in the radiation field by the heavy filters introduced to attenuate the intensity of the GIF source. As a result lot of care is needed to extrapolate the results of an accelerated test at high current to the real physical case. An interesting and important effect is shown in Fig 6 B). The figure shows that the hit efficiency of an aged wire is much less sensitive to the instantaneous source intensity than the loss of gain. It can be seen that a loss of gain of 70% generates a loss of hit efficiency still sizeable, 30%, but smaller by more than a factor two.

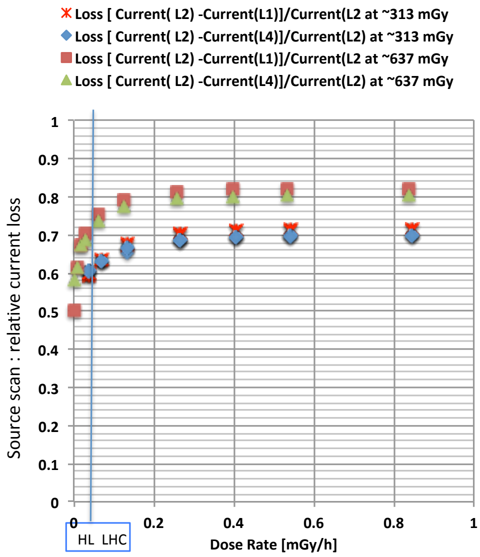


Fig.6 A

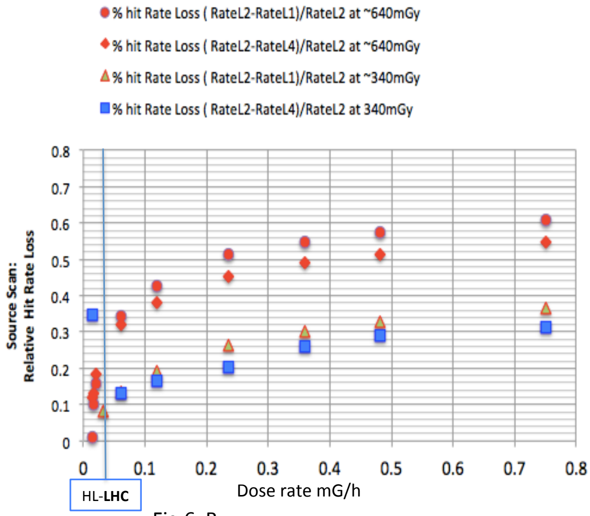


Fig.6 B

Fig A) shows that in the radiation intensity region of HL LHC the current loss of a wire after a cumulated dose of of 640 mGs (2300 fb^{-1}) is around 70% ,
Fig B) indicates that the hit rate loss is much smaller ,around 30%. This suggests that the hit inefficiency per wire might be within the same order of magnitude. A confirm can be found in fig 7.

This is due to the still high gain of the wires and to the low threshold (20 mV) applied on the amplitude of the signals. In the experiment this might imply a competition with the local noise. Another representation of the same phenomenon is shown in fig 7).

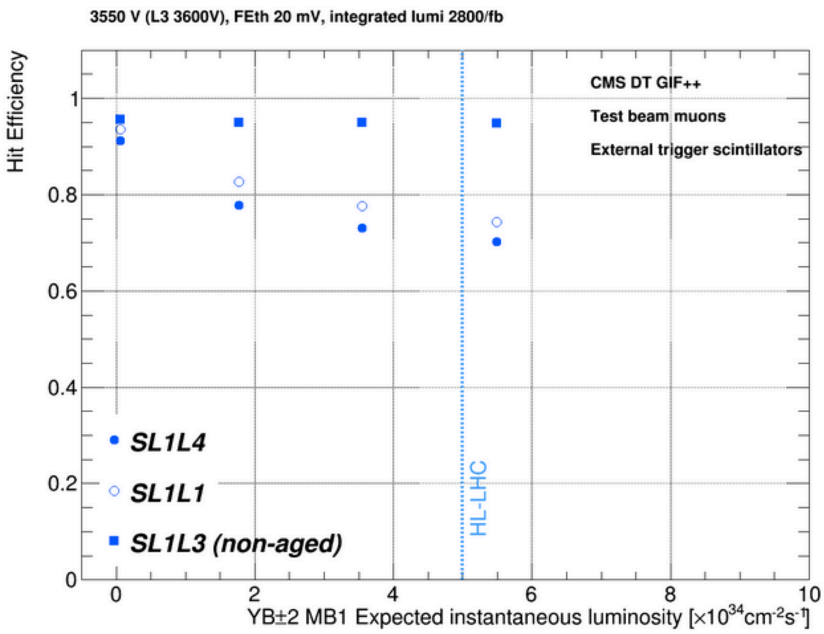


Fig. 7 The figure shows the Hit efficiency of aged and non aged wires in function of the instantaneous luminosity measured on the GIF Muon Beam. The Muons were triggered by a set of scintillators installed along the beam line and outside the irradiation area. The tracks were reconstructed through the signals detected by the " virgin" layers of the chamber. The amplifiers threshold was set at 20 mV for all the layers.
The test was performed after an integrated dose equivalent to 2800 fb^{-1}.

A natural explanation of the behavior of the relative loss of gain in a aged wire and in particular of its large variations at relatively low currents is that it is related to the composition and to the electrical properties of the material forming the coating on the wire surface. This idea was at the origin of the decision of concentrating some effort on the study of the coating development and on its components and properties.
The description of the methods used for those studies and of their results is the main subject of this Note. A second and decisive hint for this study can be found in Fig.14 in Paragraph 8).

4) ANCILLARY SMALL DETECTORS BUILT IN LNL

After the worrying result of the first test of the big MB1 Chamber and the difficulties encountered in the chamber operation in its handling and in the interpretation of the data, two types of small detectors were designed and built in LNL. They had to allow a better monitoring of the radiation intensity and to take complementary data for further calibrations and analysis in a better-controlled way. In the trials of the MB2 chamber at the GIF they were irradiated in the same time together with the big chamber in order to be exposed to the same irradiation and environment conditions. They are simple and small for an easy handling and opening for wire and field shaping electrodes analysis.
The first type of detector is a simple cylindrical single wire proportional tube with a 5 cm diameter and 50 cm long. This detector is named "Monotube " (MT in the following).
It was designed as simple as possible to be aging free: it ages if the gas at its input contains contaminant compounds generated or present in the gas from an upstream detectors or containers.
The second type of detector is a small mockup of a drift tube [6]: it consists of two tubes identical to those of the big DT Chamber but only 80 cm long (Fig.8).

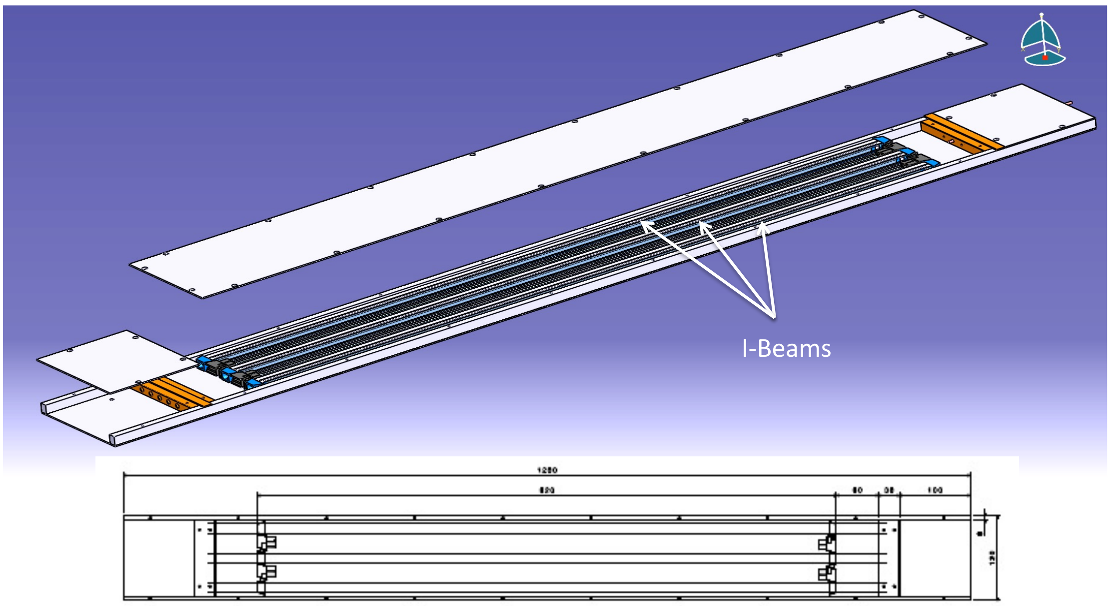


Fig. 8 A open bicell , shown are the three I-Beams that shape the two tubes , the removable cover and the absence of any electrical part in the gas volume.

They are referred as “Bicells” in the following. They were assembled using materials left back during the construction of the big chambers. The gas volume of each tube is 320 cm^3 to be compared to the 1000 cm^3 of a real DT tube and of a Monotube.
To exclude any possible gas contamination generated by non structural materials the electronics boards, cables and mechanical components for internal gas distribution etc… are not included in the Bicell active gas volume..
The wires installed in Monotubes and Bicells are the same as that in the CMS chambers: an a-magnetic gold plated chrome/nickel /cobalt wire 50 micron in diameter. The wires are from spools used for the CMS chamber assembly.

The simple structure of a Monotube suggested the implementation of a new way to produce electrons in the gas to study the development of an aging process. The usual assumption is that aging depends on the current due to the electron multiplication around the wire and not on the way those primary electrons are generated. The light of a LED of adequate wavelength put on a side of the aluminum tube of a MT can extract photo-electrons from the opposite aluminum wall. The electrons will drift to the wire and locally multiplied. Fig. 9) shows the case of a test on samples of the I-Beams. Here and in the following the word I-Beam indicates segment of I-Beam composed by the aluminum I-Beam plus the glued mylar and the aluminum ribbons.

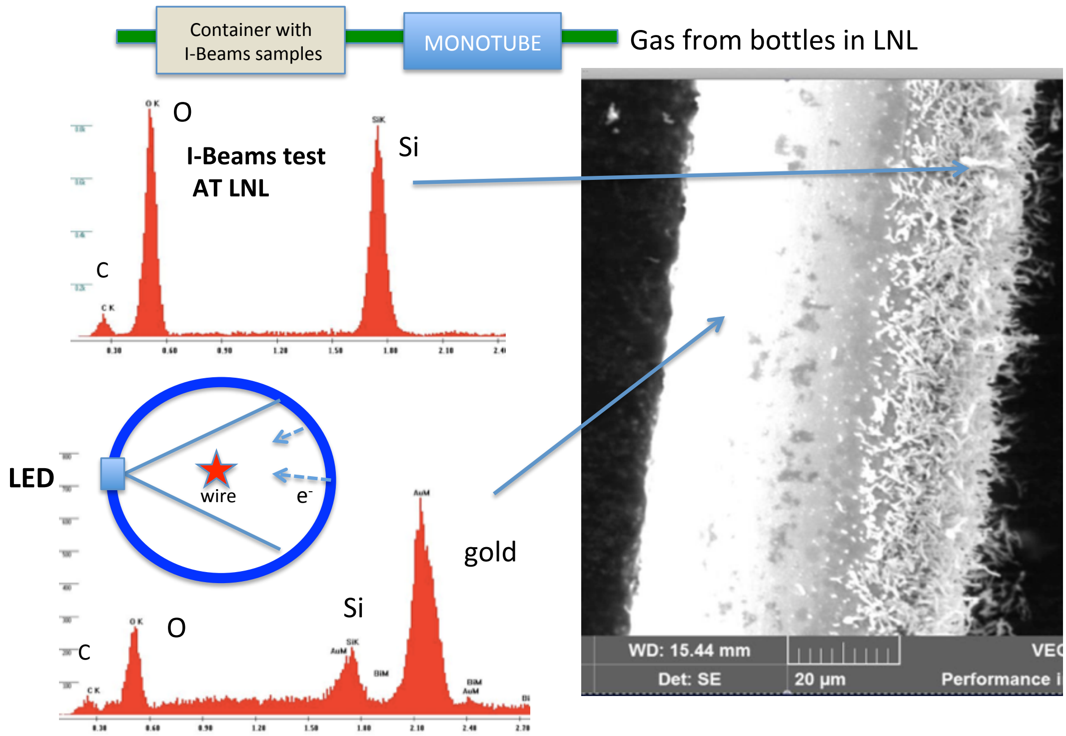


Fig 9 Example of aging induced in a Monotube wire by the multipication of the photo electrons Extrcted by a LED ligth. Note the different depth of the coating on the two faces of the wire, indicated by the arrows.In the worst case the coating is thick enough to mask the gold plating.

Due to the limited size of the light spot on the tube wall the current from the wire is localized along few centimeter of wire length and mainly on the side of the wire looking to the region illuminated by the LED. But it was found to be enough to generate a local process of aging in presence of pollutants.
The variation of gain due to an aging process is obtained at regular time intervals switching of the LED and with the help of a small Cesium source.

This technique allowed many qualitative tests to be done in LNL without use of very intense radioactive source. Unfortunately it does not allow to obtain reliable quantitative figures because of the limited area of the phenomenon and of the difficulties in the control of the rate of extracted electrons.
The first application of the method was the study of the properties of the gas coming out from the series of two big spare CMS MB3 type chambers installed on the Muon Spectrograph apparatus active in Legnaro. The gas source is a set of premixed bottles of standard Ar/CO2 85/15 gas mixture.
Monotubes flushed with the gas coming out of those MB3 Chambers were found to age indicating that the gas from the chambers was contaminated. This was not a surprise after the result of the MB1 irradiation at CERN. But we will see later that this result has some intriguing differences with respect to the GIF findings.
The LED technique allowed to be able to repeat a systematic search of the outgassing properties of the different materials present in the gas volume of the DT Chambers. The set up consisted of a clean container filled with samples of the parts to be analyzed followed by a downstream analyzing Monotube (top part of Fig 9). In these tests the MB3 chambers were skipped and the gas was taken directly from the bottles. In LNL the containers with the material samples were not under irradiation. To check for a possible effect of the radiation on the outgassing properties the tests were repeated at the GIF having the samples and the analyzer MT under irradiation. To guarantee the independence from possible local contamination in the GIF area and to allow a comparison with LNL result the filling gas was not from the standard GIF distribution rack but from an independent premixed bottle as at LNL.
The two experiments agreed on the finding that only the samples of the I-Beams equipped with the glued electrodes contaminate the gas.
In summary the first tests in LNL showed that effects of the presence of pollutant molecules in the gas volume of the MB chambers start to show as the integrated charge increases up to the HL LHC expectations, and that the responsible source might be the materials constituting the I-Beams .

5) The irradiation schemes and procedures at GIF and LNL

The magnet coil and the huge iron barrel yoke of the CMS experiment protect the DT Chambers against the radiation from the interaction point of the two proton beams of the LHC. The Barrel Chambers were not expected and not designed to stand a very high rate of particles.
So in the tests at GIF they require a low radiation intensity when compared to the requests of other users. The competition and the unavoidable sharing among the large number of users with different set ups and different and typically much higher radiation intensity requirements, made the tests discontinuous with very long pauses, up to one year long.
The time sequence of the tests and operation in LNL and GIF is shown below**.**

2015 August-November: first MB1 irradiation up and beyond the HL LHC levels. Monotubes were used to check and quantify the effect of the presence of water or O_2 .
2016 September 2nd test MB1 : irradiation of 8 new wires in MB1: they aged in the same way as the old ones that they substituted.
2016 Construction of the Bicells in LNL
2016 December to June 2017 irradiation at GIF of the first Bicell
2017 August to September irradiation at GIF of 4 Bicells

2016 -2017 Long test of materials outgassing in Legnaro
2017 October to March 2018 first irradiation of the MB2 chamber. Two Bicells and a set of monitoring MT installed in front of the Chamber were irradiated in the same time.
2018 November to Jan 2019 substitution and irradiation of 8 new wires in MB2
2019 February first short irradiation of a set of virgin wires of the Layer 2 of the MB2 SL1
Jan 2019 to May 2019. Repetition under irradiation at the GIF of the study of the outgassing properties of the samples of materials present in the DT Chambers.

The material effects of the aging cannot generally be removed, so in an aging device the measures are not repeatable. This is possible only by substituting some wires with a set of virgin ones (feasible but difficult in those chambers in the reduced space available at the GIF) or exploiting the fact that the aging phenomenon develops only on active wires. The MB1 irradiations were done at a relatively "very" high intensity, a factor 100 larger than the expected figure at HL LHC, and unfortunately also with all the 12 layers active. All of them aged. Two years later in the MB2 irradiation only few layers, two layers, or, when feasible, only a fraction of one of the 12 available layers of the chamber were kept active and the source intensity reduced by a factor ten. The non-active layers were preserved to make very short calibration runs. As mentioned in Paragraph 3) only the first group of four layers, those of the SL 1 of MB2, was used up to now: the first and the fourth Layer of the SL1 were active all the time until the cumulated dose reached the value expected at the end of the HL LHC run. The Layers 2 and 3 were switched-on very shortly (few minutes), time to time, for calibration purposes.
In a second GIF run (one year later) eight wires of the aged Layer 1 of MB2 SL1 were substituted with new ones (New Wires) and irradiated for about 80% of the expected HL LHC dose. Later on also a fraction of Layer 2 was irradiated for a shorter time. Some of the New Wires were extracted and studied in Legnaro and at the Padova University with different technologies. The behavior of the New Wires and the outcome of their analysis are unexpectedly different from those of the "old" MB2 wires in the same L1 layer. (Fig 10 and 11).

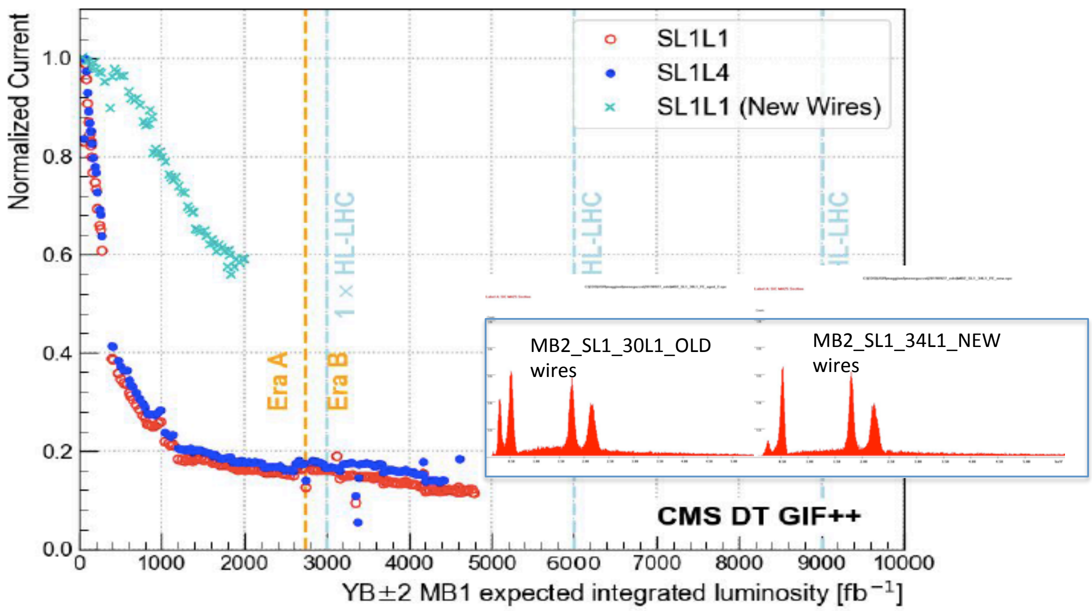


Fig. 10 After 2000 fb^{-1} the L1 New Wires , in green, loose 42% of the gain, the old wires loose 84%. The insert shows the eds spectra of old and New Wires (see also fig. 9)

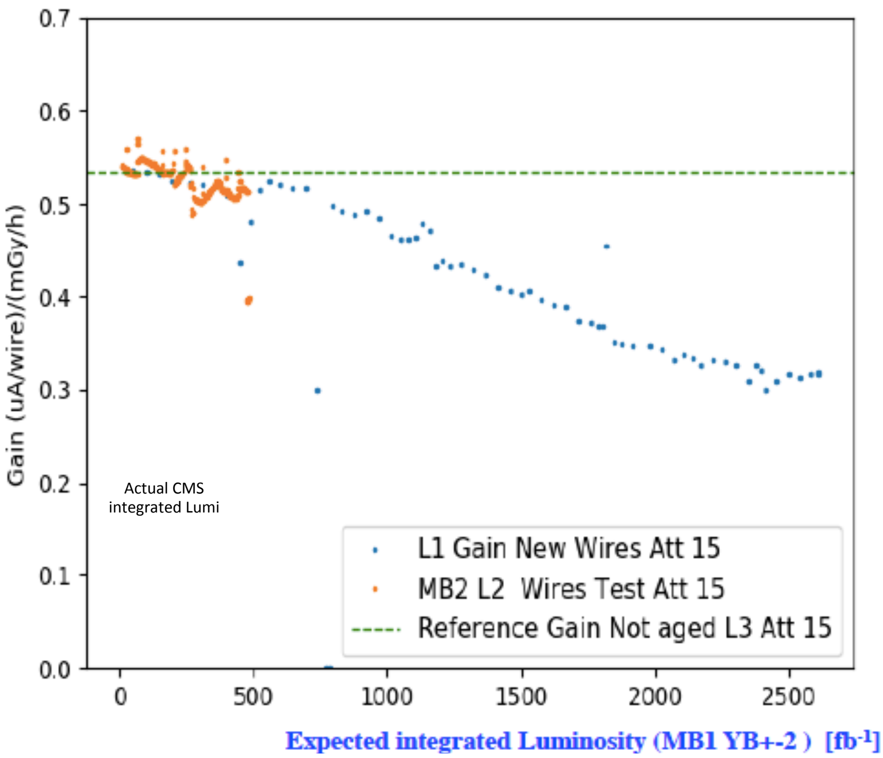


Fig. 11 Enlarged view of the variation of gain of the New Wires of the loss of gain of the MB2 New Wires of L1 during the irradiation from Oct.2018 to Jan 2019 and , In red, the behavior of the wires of the L2 virgin layer in the short two weeks of irradiation in January.

6) SAMPLES AND MATERIAL STUDIES AND ANALYSIS TECHNIQUES

At the end of every irradiation campaign some wires from the irradiated big chambers, from the Monotubes and from the Bicells were extracted and analyzed with various instruments in Legnaro or/and at the Padova University Departments.

The wires analyzed were from :
A)- MB1 chamber old wires
B)- MB1 New Wires
C)- MB2 wires from Layers 1 and 4 irradiated to a cumulated dose equal or larger than HL LHC .
D)- MB2 New Wires in layer 1 irradiated up to about half the High Luminosity dose.
E) wires from Bicells and MTs active in front of MB2 during the full run.
F)- Wires from Bicells and Monotubes irradiated independently from MB chambers in 2016/17
G)- Wires from the Monotubes used in the LNL and GIF tests of the outgassing properties of all the materials present in the gas volume of the DT Chamber
A special attention was dedicated to the study of the properties of the cathodes materials and glues.
Several techniques and instruments were used in the analysis; they are listed below:

Electronic microscope visualization of the wire surface
EDS: Energy-Dispersive X-ray Spectroscopy: to identify atomic species present on the coating surface
GCMS: Gas Chromatograph coupled to a Mass Spectrometer for outgassing composition analysis.
SIMS: Secondary Ions Mass Spectrometer for the in depth analysis of the coating
FTIR ATR : Fourier Transform Infrared Spectroscopy in Attenuated Total Reflection mode : to identify the nature of the compounds present on the wire and electrodes surface and glues.

7) RESULTS 1

A),C) - In spite of the very different intensity of the source the irradiations of the big chambers MB1 in A) and MB2 in C) gave substantially the same result: a loss of gain of about 87% after the 3000 fb^{-1} of High Luminosity LHC (10,8 mC/cm). (fig. 10) .
B) - The irradiation of eight new wires of MB1 that were substituted to 8 aged wires, gave the same result as in the first MB1 irradiation A).
D) - Remarkably different was the behavior of the New Wires of MB2 irradiated in the test D). The gain loss of the new wires after 2500 fb^{-1} is 42%.while old wires lost already 80%. (fig.10 and 11) . It is important to note that, as far it is known, the conditions of irradiation of the New Wires in D) were the same as for the "old" MB2 wires in C).
E) Bicells irradiated together with MB2 during C) lost`16% after 12 mC/cm to be compared to the 80% loss of MB2 irradiated in the same time and conditions**.**
F) Bicells irradiated separately at GIF in F) in 2016/17 lost ~ 20 % of their gain after 20 mC/cm.
G) wires from the tests of materials in LNL and GIF seem to indicate that aging comes only from materials or adhesives present in the I-Beams

The short irradiation of the reference L2 wires seems to follow the same behavior as the New Wires. (fig. 11)
The large difference observed in the losses of gain in the different tests is coupled systematically with evident features in the pictures and in the EDS spectra of the wires. All the EDS X ray spectra from MB, MB2, from the New Wires, from the Bicells and MTs show two characteristic peaks originating from Silicon and Oxygen. Only the wires from MB1 and MB2 during tests A), B) and C) show the huge Carbon peak at the left end of the spectra. The Carbon peak is absent in the spectra of the New Wires of MB2 and in the wires of the Bicells. (fig 12 and 13).
In the next section it is shown that this different behavior is confirmed by the SIMS and FTIR studies .

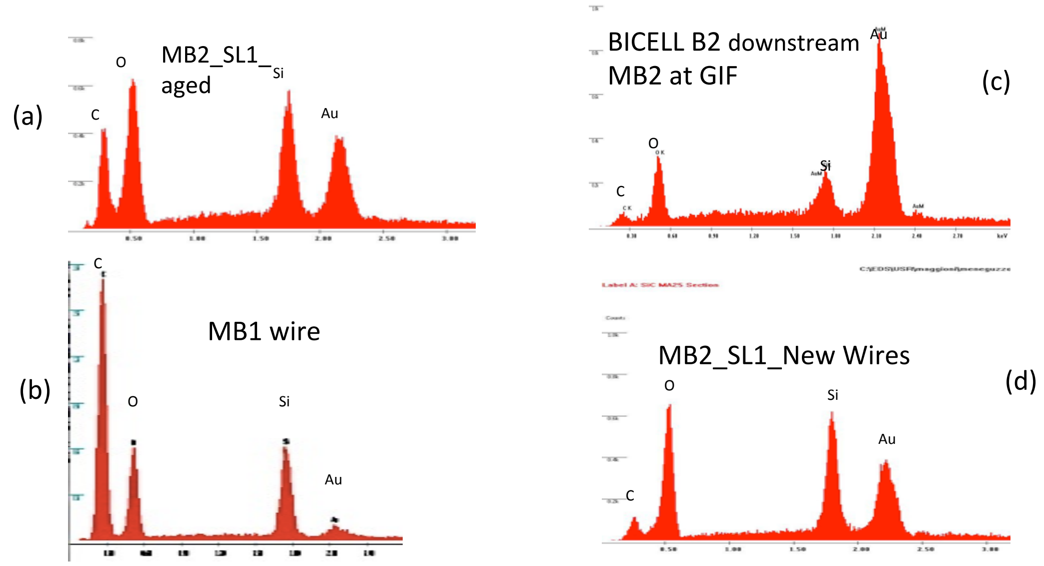


FIG 12 Left up (a) and down(b) plots are the eds result for the MB2 and MB1 irradiated wires. To be noted the large Carbon peak which is absent , or at background level in the right up (c) and down (d) plots showing a wire from a Bicell irradiated together with MB2 and one of the new Wires in MB2.

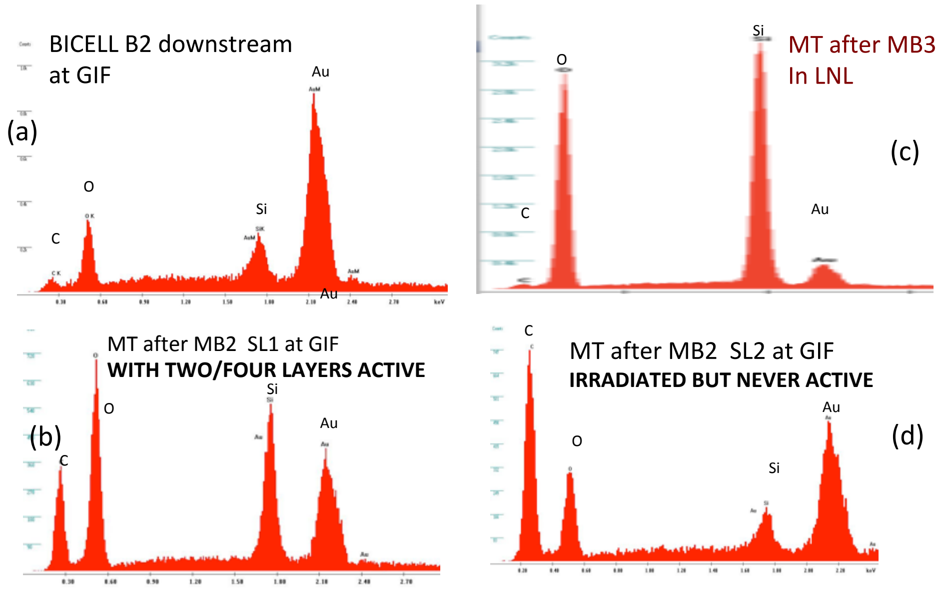


Fig.13 The eds image in (a) and (c) are caracterized by the absence of the Carbon signal which is well present in (b) and (d). Comparison of (b) to (a) of fig 9) shows that MT reproduce well the eds in the upstream chamber. (d) was expected to be very similar to the MB3 case (c).

The above results can be summarized in two important findings. The first is related to the observation of aging in the Bicells. Being the Front End and HV distribution boards and ancillary services outside their gas volume the aging can depend only on materials or adhesives present in the I-Beams. The EDS of the coatings of their wires exhibit the typical couple of Si and Oxygen peaks. This "universal cathode effect" is confirmed by its presence in all the MB1 and MB2 wire analysis.

The second finding is that no Carbon signal is clearly evident in the New MB2 Wires and in the Bicells : the lack of this signal comes together with a much milder aging compared to the MB1 or MB2 wires .

However ambiguities and contradictions showed also up.

In test E) a Bicell was present downstream to MB2 flushed with the OR of the gas output lines of the three SLs and three independent gas lines from the three SLs were feeding each a dedicated Monotube. An intriguing finding was the presence of the Carbon signal in the three Monotubes and its absence in the Bicell wires. (fig 13 shows SL 1 and SL2 but SL3 has the same face).

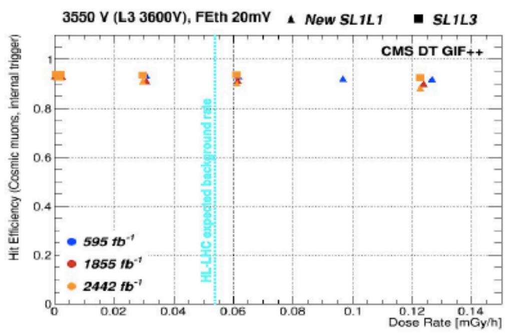
It worth's noting that the presence of Carbon in the MT downstream SL1 might be justified by its two active and aging layers but, on the contrary, the SL2 and SL3 were never switched on, they are supposed to be "virgin" layers. As mentioned in section 4) in a test in LNL a Monotube downstream two MB3 "virgin" spare chambers, and fed by their gas output, aged showing the Si and O_2 peaks but with no sign of Carbon. (fig 13) This is apparently in contradiction with the behavior of the Monotubes downstream the MB2 Super Layers SL2 and SL3. The only difference is that the MB3 were never exposed to an intense irradiation as happened , but "passively", to SL2 and SL3 But, as reported in Paragraph 3), the stable behavior of the reference L2 during the many calibration runs seems to exclude any effect from a "passive" irradiation. .

The MB3 behavior agrees however with the no Carbon outcome in the New Wires of MB2.

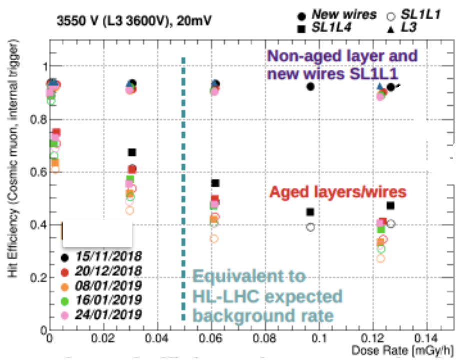
In synthesis it seems that the DT chambers age with time due to two different processes. The EDS spectra indicate that one, present in all tests, is characterized by the deposit on the wires of Silicon compounds and a second, responsible for a much severe aging, contains together with Silicon also deposits of Carbon compounds.

8) RESULTS 2

An effort to understand better those spectra and to obtain some deeper information exploiting other analysis techniques has an important justification related to the observation that a loss of current of 70% translates in a loss of hit efficiency around 30% while a loss of current around 30 ~ 40% , as suggested by the New Wires performance (Fig.10 and 11) might still affects a bit the time and space resolution but would have negligible effects on the hits efficiency and so on the overall performance of the chambers.
This hypothesis is confirmed by the analysis of the hit efficiency of the New Wires compared to that of a virgin layer shown in Fig 14). In spite of a loss of gain of ~ 40% the New Wires have the same detection efficiency of the virgin wires.



Hit efficiency for cosmic Muons in function
of the instantaneous dose for the
New Wires of the SL1 L1. The efficiency of the "virgin"
L3 is shown by comparison
There are three series of data at different
integrated dose.
The performance of the New Wires is not
affected also in presence of a high background.



Hit efficiency for cosmic muons of the New Wires
compared to the Old ones in function of the
Instantaneous dose and at increasing integrated
dose.The efficiency for Old and New is measured
separately.

Fig.14

The first target of the new analysis was the Silicon based glue present in the Cathodes: The search was done by a group of the Dipartimento di Scienze Chimiche of the University of Padova .
About 0,5 g of glue were separated with different solvents (acetone, esano, dichloromethane , ethyl-acetate) from the adesive Mylar tapeand analyzed with a Gas Chromatograph followed by a Mass Spectrometer.
The chromatograms of the different extractions show very similar profiles
The peacks are due to the presence of Siloxanes, of several molecular weight, that caracterize the glue present on the tape.
Remarked is the presence of phtalates coming with good probablity from the plastic support. (Fig 15). Phtalates are Carbon compounds caracterized by the C=O links typical of the ester groups. This was the first clear hint indicating a different origin of the two types of contamination. No information from the wire coating was obtained with the same procedures: the coating appeared to be not solvable at all.

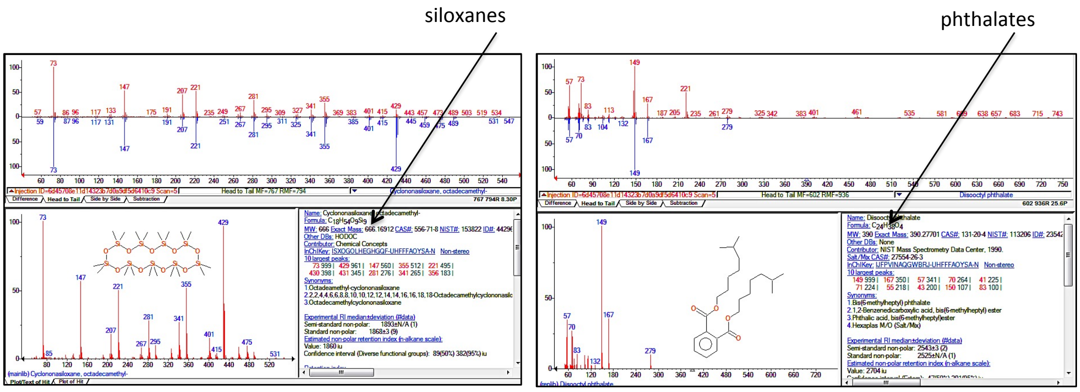
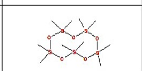
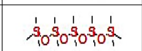
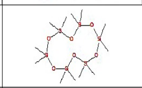


FIG 15

Results of the I-Beams glue done with a GCMS apparatus at the Dipartimento di Chimica of the University in Padova.
in About 0,5 g of adhesive tape were extracted with different solvents and analyzed with a GC-MS. The chromatograms of the different extractions show very similar profiles
The peaks are due to the presence of Siloxanes, of several molecular weight that caracterize the glue present on the tape.
We remark the presence of phthalates coming with good probablity from the plastic support,

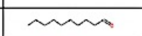
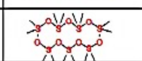
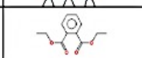
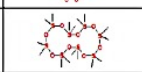
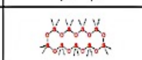
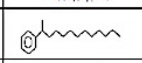
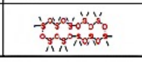
The result is compatible with an early analysis done by courtesy by the firm S.Gobain that produced the Aluminum/Mylar strips. They separated mechanically a small sample of glue from the mylar face of the ribbon and heated it up to 300 degrees in steps of 50 degrees..
Outgassing vapor was analyzed using a Gas Chromatograph followed by a Mass Spectrometer. They found that Silicon compounds, siloxanes, were outgassed at low temperatures but that phthalates showed out only at a temperature of 150 degrees. (Fig. 16) The temperature in a chamber is uniform and around 20 degrees with exception of few local very small area due to the few active electronics parts that can reach 40 degrees.

Volatiles found in adhesive at 100 ° C

RT (min)	Compound	Structure	% Match	CAS #
12.06	Cyclopentasiloxane, decamethyl-		84	541-02-6
12.56	Pentasiloxane, dodecamethyl-		81	
14.54	Cyclohexasiloxane, dodecamethyl-		84	

Results from S.Gobain H −Old
(with permission)

Figure 3: GCMS chromatogram of adhesive in h-old silicone tape at 150°C

RT (min)	Compound	Structure	% Match	CAS #
13.19	Decanal		91	112-31-2
16.71	Cycloheptasiloxane, tetradecamethyl-		95	107-50-6
18.23	Diethyl Phthalate		98	84-66-2
18.66	Cyclooctasiloxane, hexadecamethyl-		89	556-68-3
20.35	Cyclononasiloxane, octadecamethyl-		89	556-71-8
21.76	Benzene, (1-methyldodecyl)-		87	4534-53-6
21.85	Cyclodecasiloxane, eicosamethyl-		91	18772-36-6

GCMS chromatograms from US S Gobain of a sample of glue mechanically detached from the Mylar support.
No volatiles are detected at 50ºC .
Shown are the findings at 100 and 150ºC
Phthalates compounds show up at 150ºC.

FIG 16

Phthalate

The results suggested the possibility of a time dependence of the deposition of the different polluting compounds. The composition of the coating along its depth was studied searching for non-homogeneities.

The analyzing instrument was a Secondary Ions Mass Spectrometer (SIMS), operated by colleagues of the Istituto di Fisica della Materia of the Padova Physics Department. In a SIMS, an energetic Oxygen molecular beam can be made to sweep the surface of the wire to generate the release of ions that are then injected in a Mass Spectrometer. The sweeping is repeated many times slowly erasing the coating and extracting ions at increasing depths.
Discontinuities in time during the process might indicate that the deposition of different compounds is not simultaneous, e.g. the possibility that the Carbon compounds were deposited as first in the initial phase of the aging process or vice versa.
The test was performed on samples of a virgin wire, on a MB1 wire and on a Bicell wire. (fig. 17,18 and 19, the last includes also a background test) (quot prof. Carnera)

Rate
In log scale

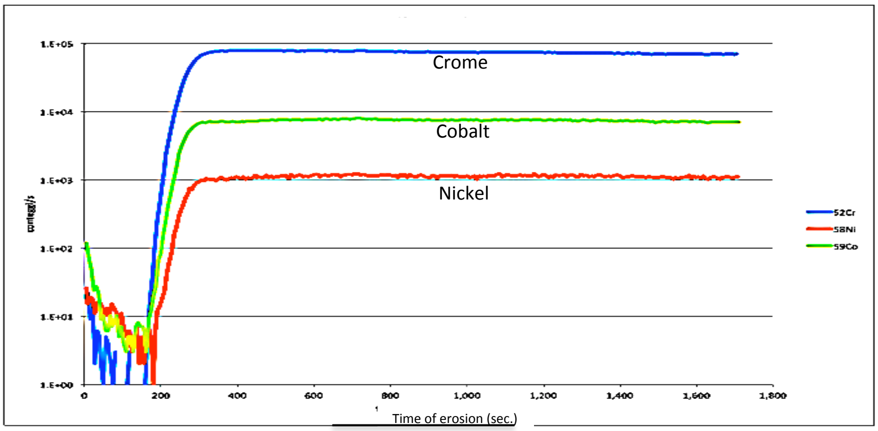


Fig.17

SIMS analysis of a virgin wire done at the Dipartimento di Fisica dell' Univesita' in Padova . The thin gold coating of the wire is not detected: a too small number of atoms are extracted and very few of them are ionized.
Than the beam reaches the anode wire, showing its composition..

10^4

10^3

10^2

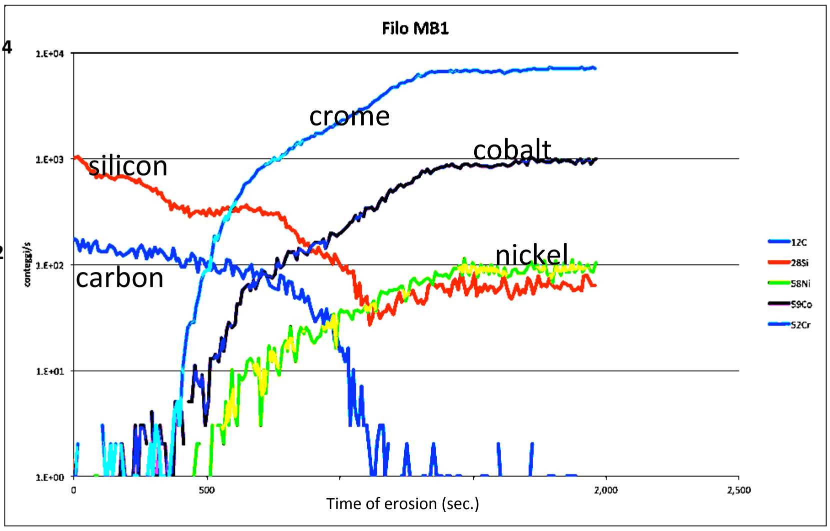


Fig. 18

SIMS analysis of an aged MB1 wire : at the beginning only C and Si are visible , after a while the coating start to be thin enough to allow the beam to reach the wire extracting heavy ions from it. When the coating has been completely erased, Si and C drop by order of magnitude.
A fraction of silicon remains, extracted from the supporting plate.

NEIGHBOUR BACKGROUND

SIMS analysis of a wire of BICELL 1 and background response.

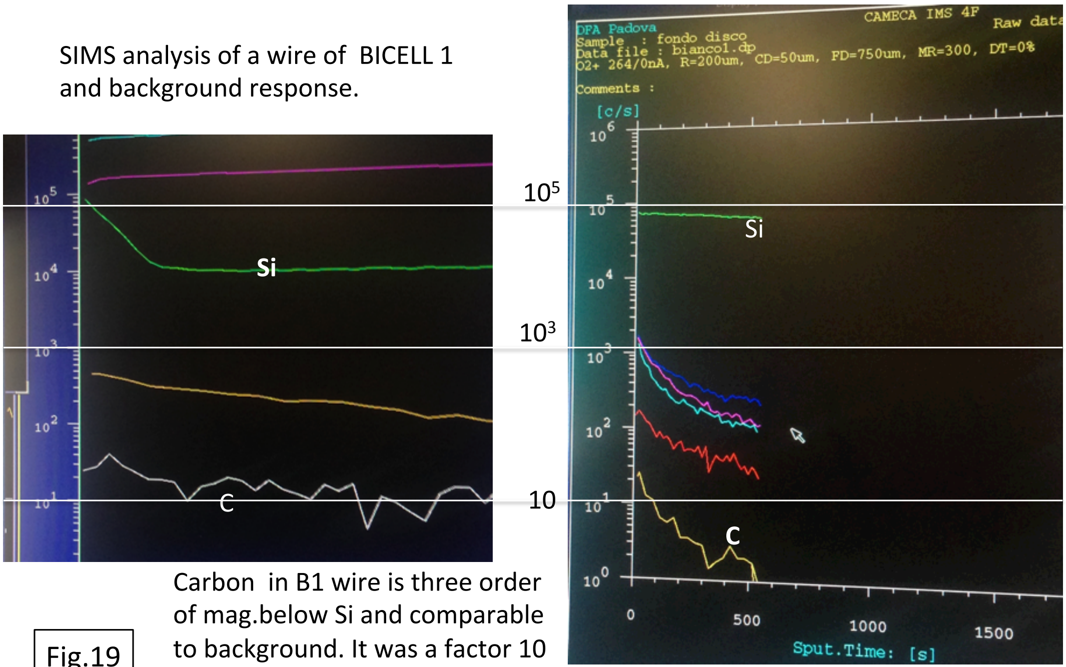


Fig.19

Carbon in B1 wire is three order of mag.below Si and comparable to background. It was a factor 10 below Si in MB1 (previous slide) .

The result shows that the coating is homogenous and highly resistive and confirmed the presence of Carbon in the MB1 wires and its absence in the Bicell wire. A very high resistivity was also observed during some EDS measurements and is likely to explain the large dependence of the detection efficiency of a aged wire on the intensity of the irradiation.

The “final” confirm of the intriguing phenomenology revealed by the EDS analysis was obtained with the Infra Red technique available in LNL. With some care and adjustments the FTIR (quote Carturan) could analyze both the 50 micron wires and the glue deposited on the surface of the Mylar strips of the Cathodes.

In case of the test A), B) and C) of the big Chambers the FTIR confirmed the presence on the aged wires of Silicon compounds (siloxanes) and of two groups of carbon compounds (phtalates and methyl groups). But it also confirmed the absence of Carbon compounds in the Bicell and on the NEW MB2 Wires.

They indicate a low presence of Carbon compounds (methyl groups) on the Cathode glue compatible with the outcome of the gas spectrometer analysis from S Gobain. (fig 20 and 21).

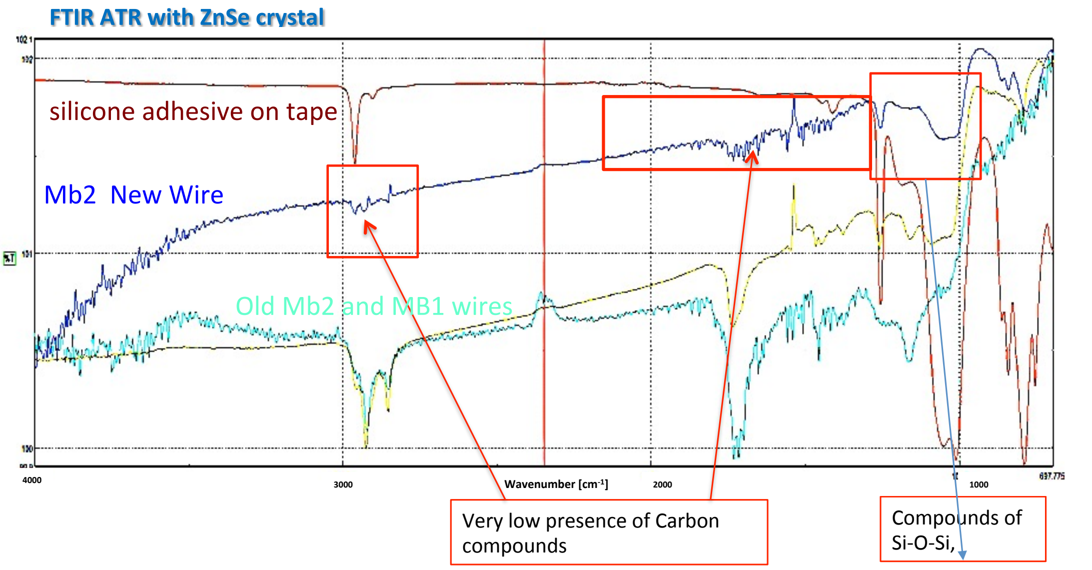


FIG 20

Results from I-Beams glue and from MB1,MB2 and MB2 New Wires obtained with the FTIR ATR analysis apparatus in LNL in Legnaro.In evidence the lack of Carbon compounds in the New Wire and their presence in the MB1 and MB2 "old" wires.

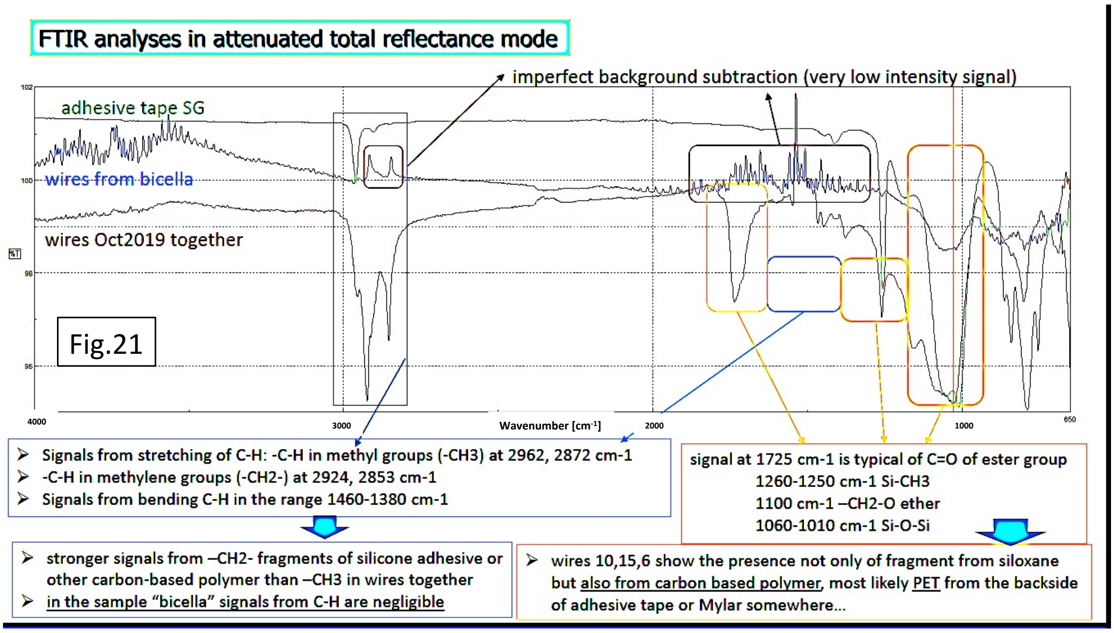


Fig.21

But they also agree with the indication of the GCMS tests in Padova that phtalates might be generated under irradiation by some PET parts present in the chamber (e.g Mylar…). It should however notice that no Carbon and only Si and O were observed on the MT aged wires downstream the material samples under irradiation at the GIF.
A last EDS analysis was recently performed on the cathodes.
The cathodes are made by a thin Al strip glued to a thick Mylar ribbon that is in turn glued to the Aluminum of the plates and of the I-Beams.
The result is shown in Fig 22 and suggests that Carbon compounds (phtalates) are present in the Mylar and that they might show up under an intense irradiation.

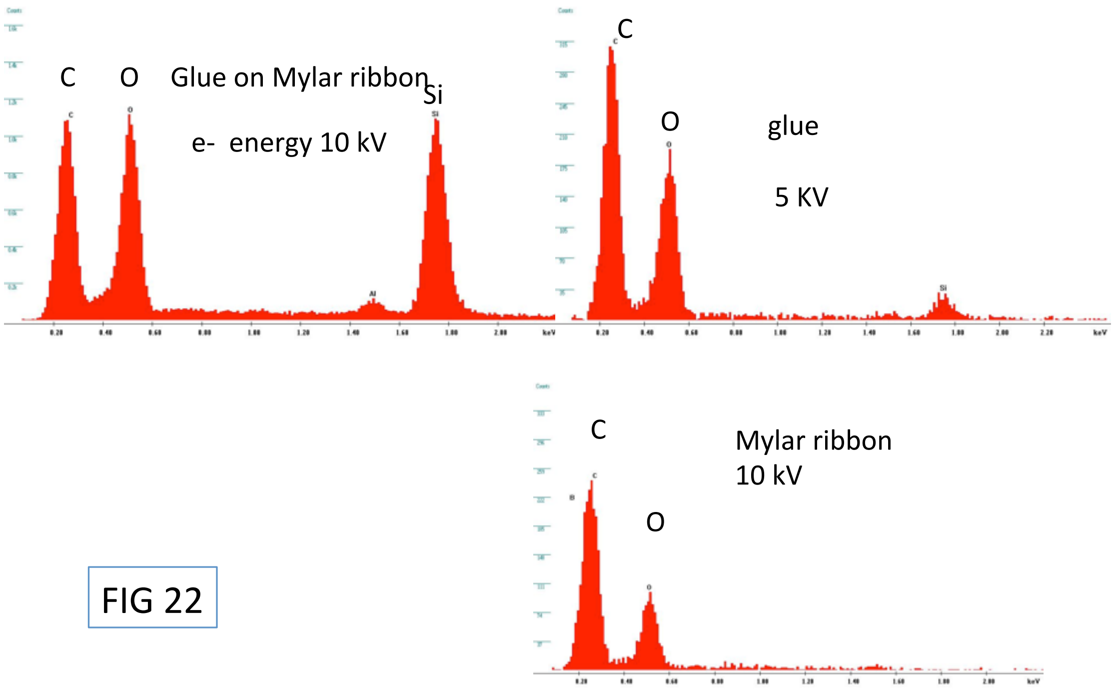


FIG 22

9) SUMMARY and CONCLUSIONS

The most important conclusion is that the barrel DT chambers of CMS will age with time.
This aging seems to have two origins: an established origin is in the glue on the cathodes. It gives the characteristic Si and O_2 peaks due to the siloxanes present in all the EDS spectra . The absence of a clear Carbon signal in some of the spectra indicates a second possible origin from a different source of contamination .The carbon signal is absent in the New Wires and in the Bicells. Even under the assumption that the absence of Carbon on the New Wires of the MB2 chamber is due to some mistake or to some and non recorded anomaly during the irradiation, one should in any case understand the origin of its systematic absence in the Bicells.
The crucial observation is its absence in the Bicell downstream MB2 in C) when it was simultaneously present in the downstream Monotubes.
This absence might be explained by the different rate of gas renewal in the Bicells tubes with respect to that in the DT chambers tubes and in the MTs.
The volume of a Bicell tube is three times smaller than that of the other two detectors. In spite of the fact that care was taken to operate all detectors at the same gas flow rate per tube, the different tube volume implies a three times faster gas renewal in the Bicell. Under the assumption that the outgassing rate does not depend on the gas flow, the above translates in a three times lower impurity concentration in the Bicells. This might explain the non-detection of a possibly weak Carbon signal and the non-cancellation of the “strong” firm of the Silicon compounds.
The nominal gas flow in the DT is of one cc per minute and per tube, an irradiation at 3 cc/minute resulting in the absence of a Carbon signal in the wires of a layer of a virgin Super Layer might clarify the contradiction with the Bicell and possibly indicate a way for a milder aging.
One should also look to the simultaneous appearance of Carbon in a MB chamber at nominal flow and in a Bicell operated at a very low gas flow. A figure of

0,3cc/minute for a Bicell is challenging but not impossible. If the results from the New Wires are due to a non detected accident, a positive outcome of this double test would be a credible result.
It will not however explain the lack of Carbon in the material tests in Legnaro and at GIF and in the MB3 in Legnaro.
So the second mandatory activity is the continuation of the irradiation of MB2 New Wires in SL1and of the wires of the Layer 2 in the MB2 SL1. A confirm of the observed mild aging behavior would imply that an improvement of the gas quality was introduced after the end of the MB2 irradiation and the start of those on the New Wires. There is no clear indication that the gas feeding the chambers in the first tests was not clean but this interval of time coincides with an intervention on the GIF gas system needed to seal a leakage detected in a Dewar.
The confirmation of the behavior of the New Wires would probably imply that no Carbon will be present in the new flow tests of Bicell and MB2 that will be hopefully filled now with the "new" cleaner gas. This will also agree with the outcome of the material tests and with the results on MB3 in LNL
The case of a "bad" behavior of the New Wires and of the L2 wires should be considered in the light of the outcome of the High Flow test. A contextual positive answer from the High Flow test (i.e. no Carbon and gentle aging) opens a road to mitigate/control the aging.
Fig. 6) shows that the gain, and so the hit efficiency, of an aged wire depends on the current i.e. on the intensity of the radiation. The high resistivity of the coating is probably the major responsible of this behavior but there are also indications that the space charge due to the many slow moving positive ions generated by an intense radiation induces deformations in the field of the drift cell. These two effects begin to show up at intensities below those expected at HL LHC They should be better investigated with frequent, improved and careful source scans at intensities that spans an interval centered on the expected HL.
An improvement on the precision of the measure of the currents will be probably needed. These studies could help a lot in the understanding of the discrepancy between the GIF and CMS figures and allow a better extrapolation from the accelerated aging at GIF and the real aging in the experiment.

9) AKNOWLDGEMENTS

The analysis in LNL ran in parallel with the chamber irradiation at CERN GIF.
The set up and run at CERN was, and still is, a very heavy and delicate job from the organizational and technical aspects. Organization requires specific agreements with the CERN GIF responsibles but mainly with the colleagues from other experiments and groups active in the site.
The technical difficulties come from the hostile radiation environment and the organization and operation of the many types of different services required by the many users. The limited size of the group of available people from the DT group, committed in the same time in the important operation of the CMS detector at P5, was also a non negligible headache. This complicated activity was firmly driven by the CMS DT Project Manager Ignacio Redondo with help from his CIEMAT colleagues, of Isidro Gonzalez and the Oviedo Team, of the Aachen group with Hans Reither and colleagues. An important contribution to specific issues came also from several of the Authors of this Note.
A special recognition is due to D. Dattola (INFN To) for his outstanding and crucial contribution to all the phases of the experiment.

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