Possible CF4 reduction or replacement in the gas mixture of the CMS Cathode Strip Chambers

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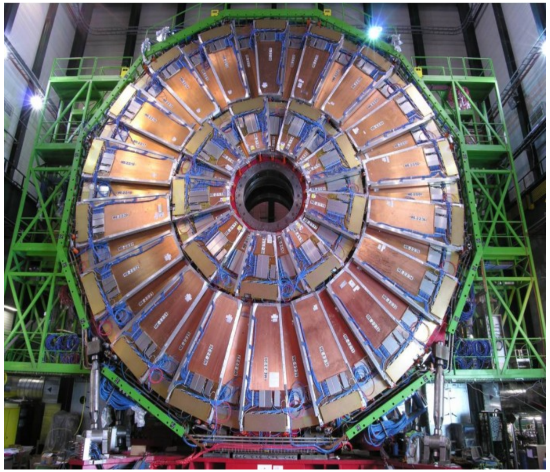
for the CMS CSC longevity team

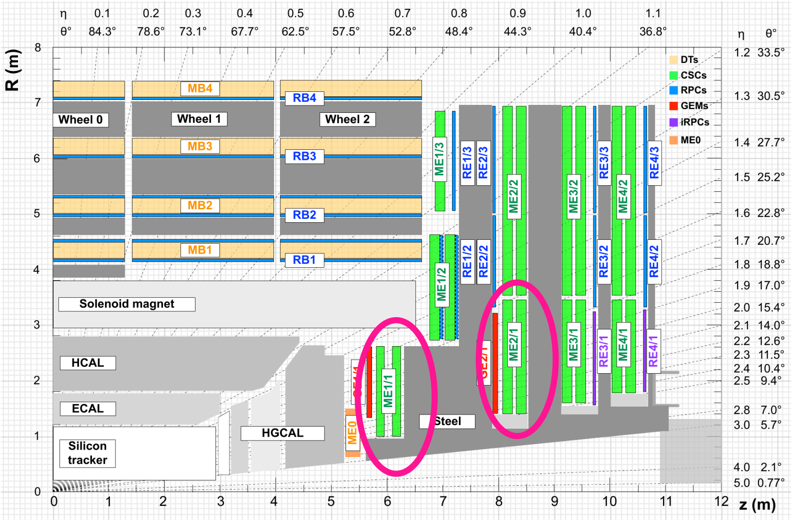
66th INFN ELOISATRON WORKSHOP: New gas mixtures for RPC and MRPC detectors
22.11.22

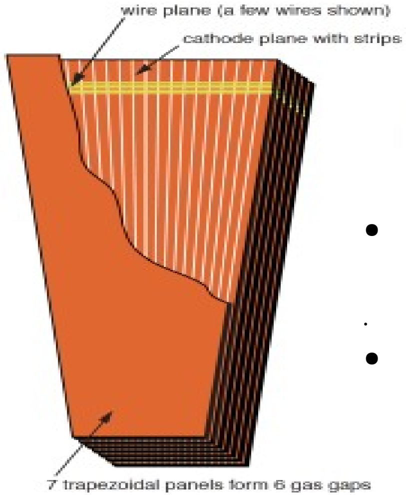


CMS CSC

- 6 layer **MWPC** with stripped cathode giving precise coordinate information across strips

- **ME1/1** and **ME2/1** chambers – in the most forward location, differ in material and construction





- **Cathode**: Cu-foil-coated glass-reinforced FR4 with strips milled into copper
- **Anode**: gold-plated tungsten wires, 50 μm in diameter (30 for ME1/1)

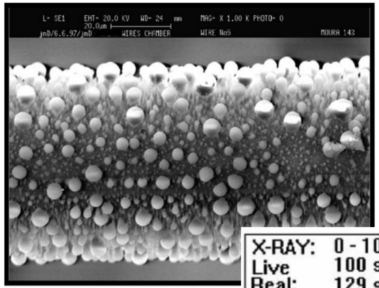
Prediction for **HL-LHC** operation for the most affected areas of ME1/1 and ME2/1 chambers: Accumulated charge (longevity - related equivalent operation time) of **0.20 and 0.13 C per cm** of anode wire

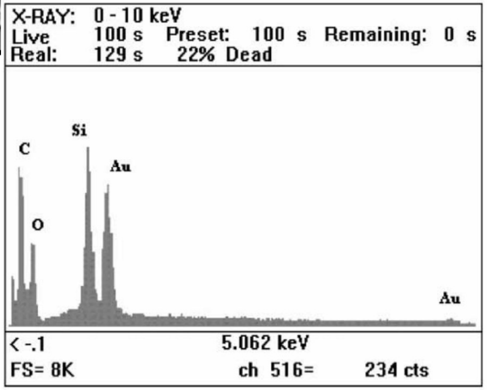
CSC gas mixture

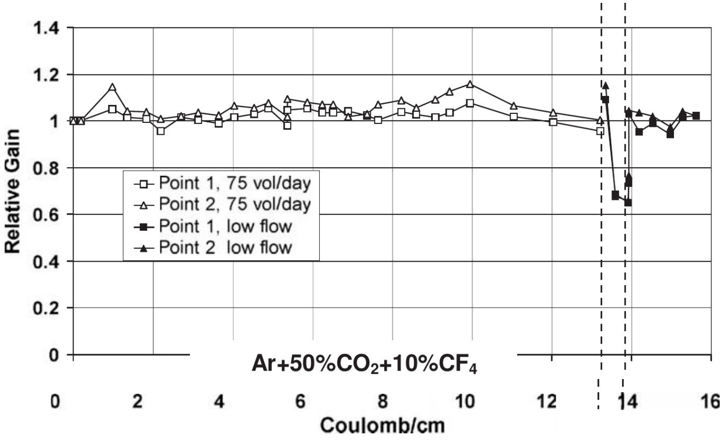
- **40% Ar + 50% CO2 + 10% CF4**

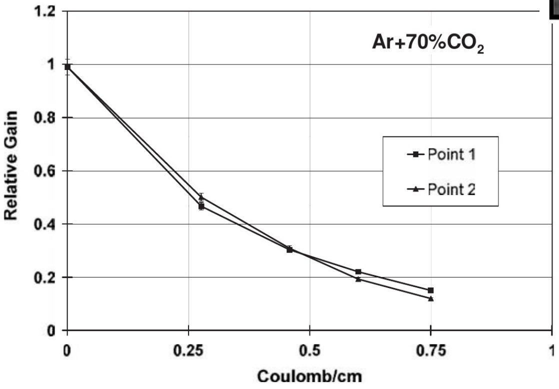
- The main purpose of CF4 in the gas mixture – protection against anode wire aging : Si + 4 F → SiF4
  (also breaking C-chains in polymer formation)

Early studies with first CSC prototypes (Si in contact to the gas volume - not the case in the final design) NIM A 488 (2002) 240–257









CSC gas supply

- Developed and supported by CERN EP-DT
  - CSC: 540 chambers ~66 m3 in total
  - Total flow ~6.6 m3/h
  - Close loop gas system
  - Replenishment rate: 10%
  - CF4 recuperation : 50-70%

details in Roberto Guida's talk

- **GWP(CF4)~7000**
- **Increasing price**
- **Availability on market is getting unstable**

Three ways to reduce or eliminate CF4 use or exhaust:

- **CF4 recuperation**: EP-DT – efficiency of the CF4 recuperation plant was increased from 30% to ~60% during LS2
- **CF4 reduction**:
  - lab studies with small prototypes ('miniCSC')
  - tests with full-scale production chamber at GIF++
- **Searches for CF4 substitutes**

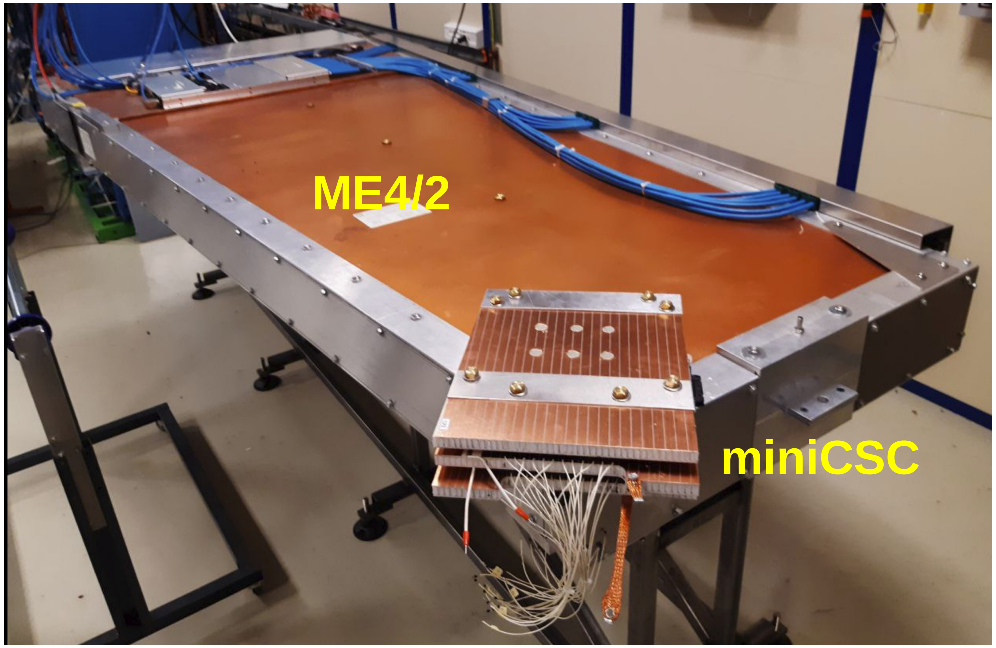
Any solution should preserve the CSC longevity in view of HL-LHC

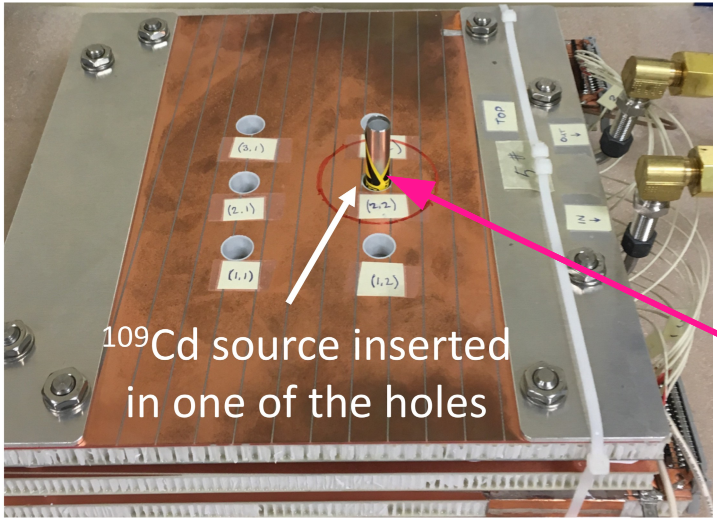
Reduction of CF4

Accelerated local irradiation of miniCSCs (ME2/1 type) with ^{90}Sr:

miniCSC:

- Specially built prototypes
- 2 layers 30x30 cm2
- Original material
- Original technology
- Scaled gas flow
- Open gas loop



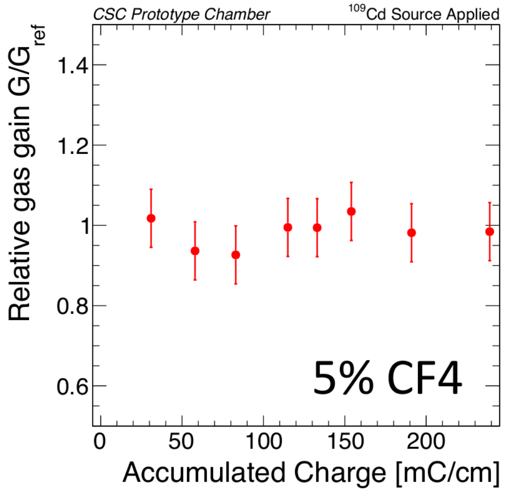


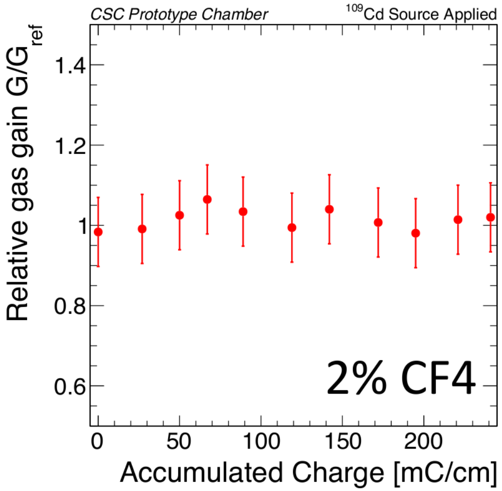
Irradiation (^{90}Sr) and control (^{109}Cd or ^{55}Fe) point

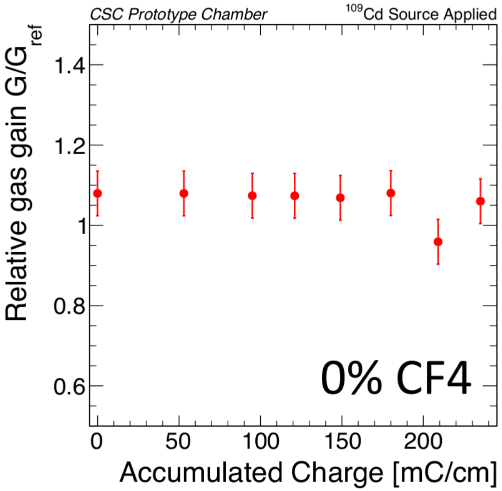
Reduction of CF4

Accelerated local irradiation of miniCSCs (ME2/1 type) with ^{90}Sr:

- 5, 2 and 0% CF4 - performed at 904 and GIF++ up to 0.24 C/cm (1.8 x $Q_{HL\text{-}LHC}$ for ME2/1)
- 10%CF4 was performed in PNPI up to 1.3 C/cm with high acceleration factor
- **no significant performance degradation was seen up in any of these longevity tests (gas gain, dark rate and current, interstrip resistance)**
- cathode and anode surfaces were investigated after the tests (CERN, University of Belgrade, Sarov)
- cathode surface modification is seen in all cases
- **anode depositions are clearly seen for 2 and 0 %CF4 even with a naked eye**

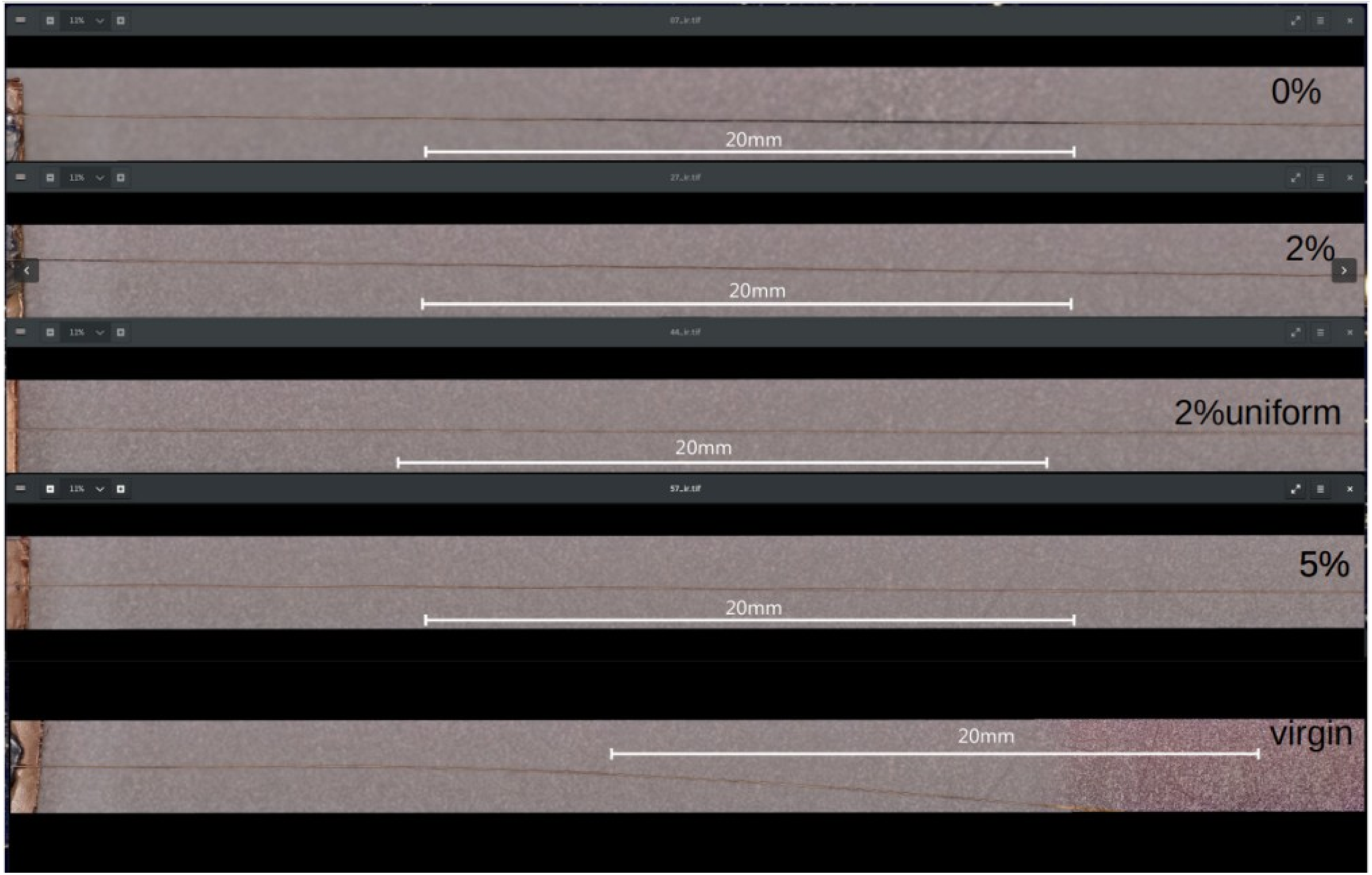






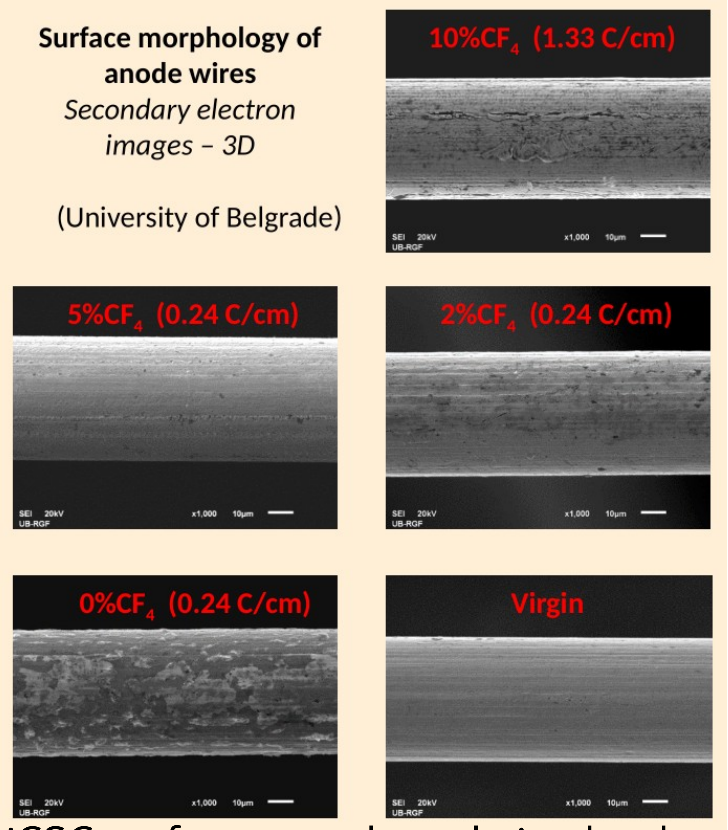
Reduction of CF4 – anode surface

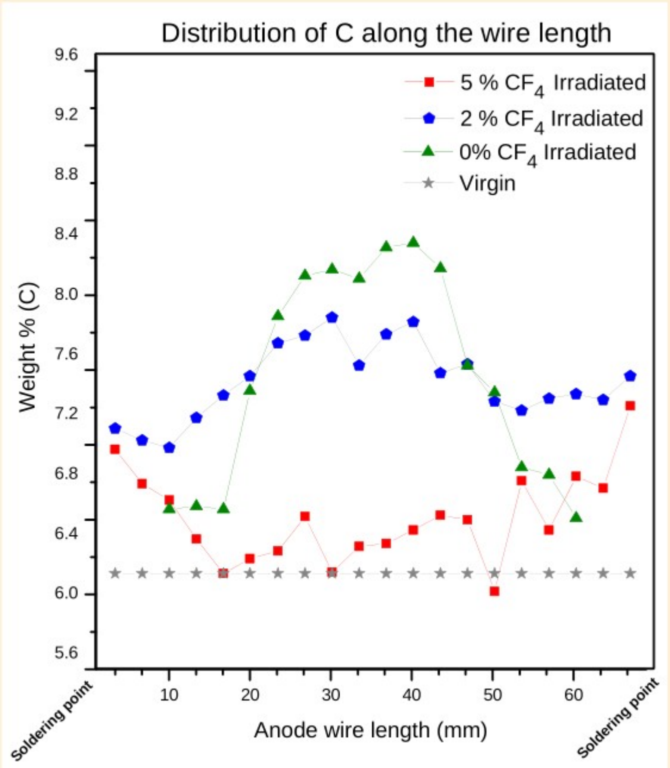
Optical microscopic view (CERN-MME-MM)



Reduction of CF4 – anode surface

SEM/EDS (University of Belgrade)





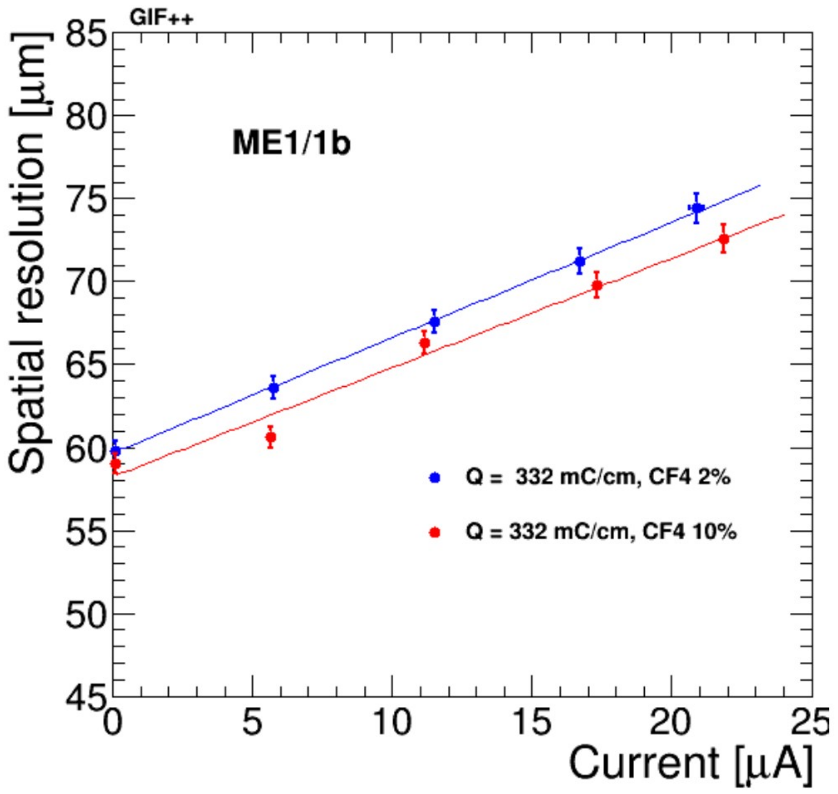
Though no miniCSC performance degradation has been seen in the longevity test – too low CF4% seems to be risky! 5%CF4 may be a good choice – also confirmed by the LHCb MUON operation experience => a longevity test with the full scale production CSCs are ongoing at GIF++ with 5%CF4

Performance with 2% CF4

Comparison of the CSC performance for muon detection has been done with ME1/1 and ME2/1 chambers during a GIF++ muon test beam

The measurements are done with different background levels up to the one comparable to LH-LHC

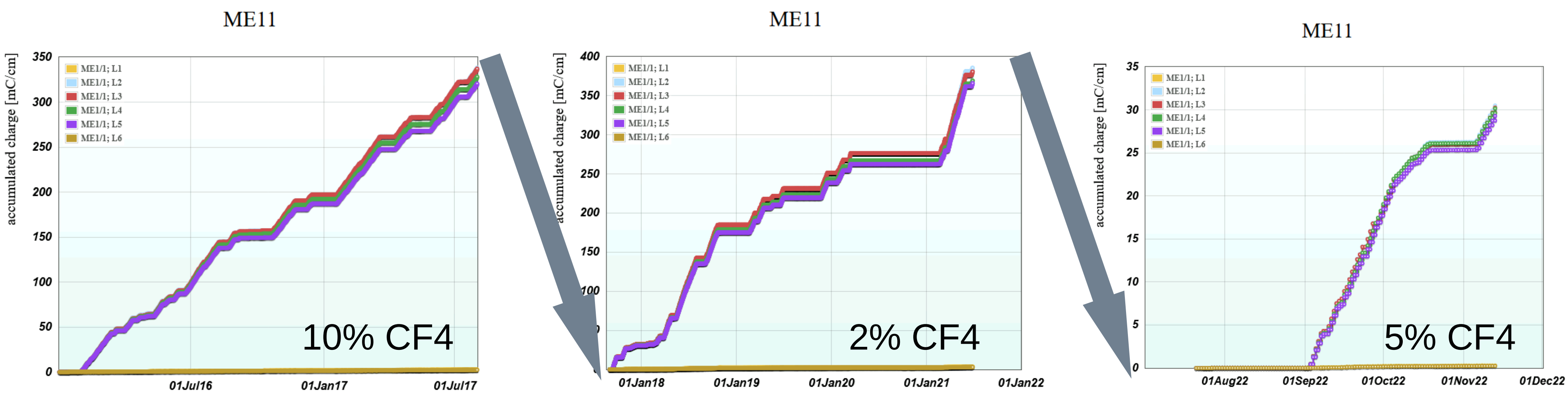
No significant difference between 2% and 10% CF4 gas mixtures was observed



Longevity tests at GIF++

- Started in 2016 with the nominal gas mixture and ME1/1 and ME2/1 CSCs
- Closed loop gas system with original materials and parts – thanks to CERN EP-DT
- The same gas flow and replenishment rate as for P5 chambers
- No recuperation
- O2, H2O monitoring (<10 ppm, <200 ppm)
- Original CSC electronics (RunII type)
- Two of six layers are preserved as reference
- Regular monitoring – relative gas gain as a current ratio, dark rate, strip-to-strip resistance
- Muon test beam measurements – relative gas gain as a muon peak position, resolution without and with background load, trigger/track/hit efficiency

Longevity tests at GIF++

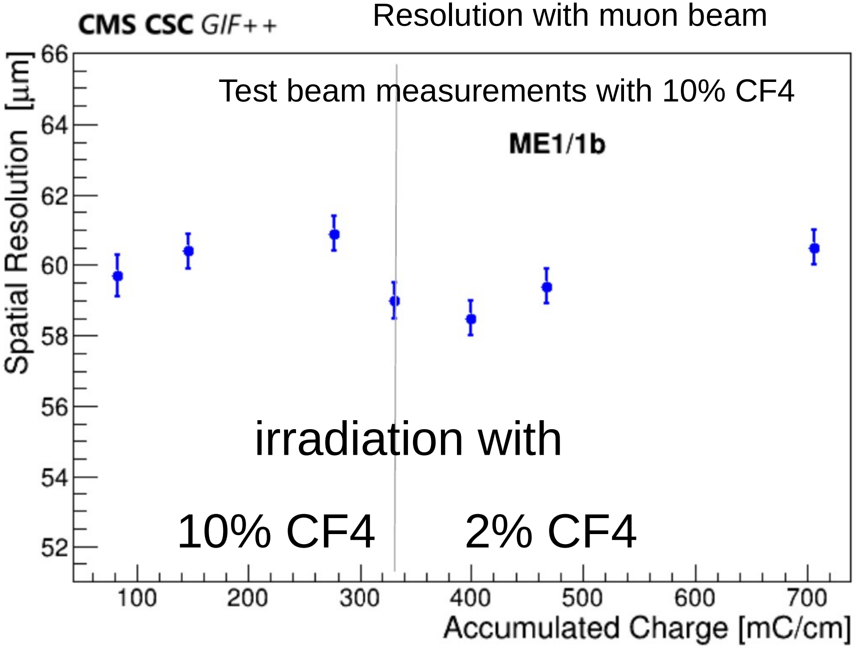


ME1/1:
Q(HL-LHC) ~ 0.20 C/cm
Q(10%CF4) ~ 0.33 C/cm
Q(2%CF4) ~ 0.37 C/cm
Q(5%CF4) ~ 0.03 C/cm, ongoing

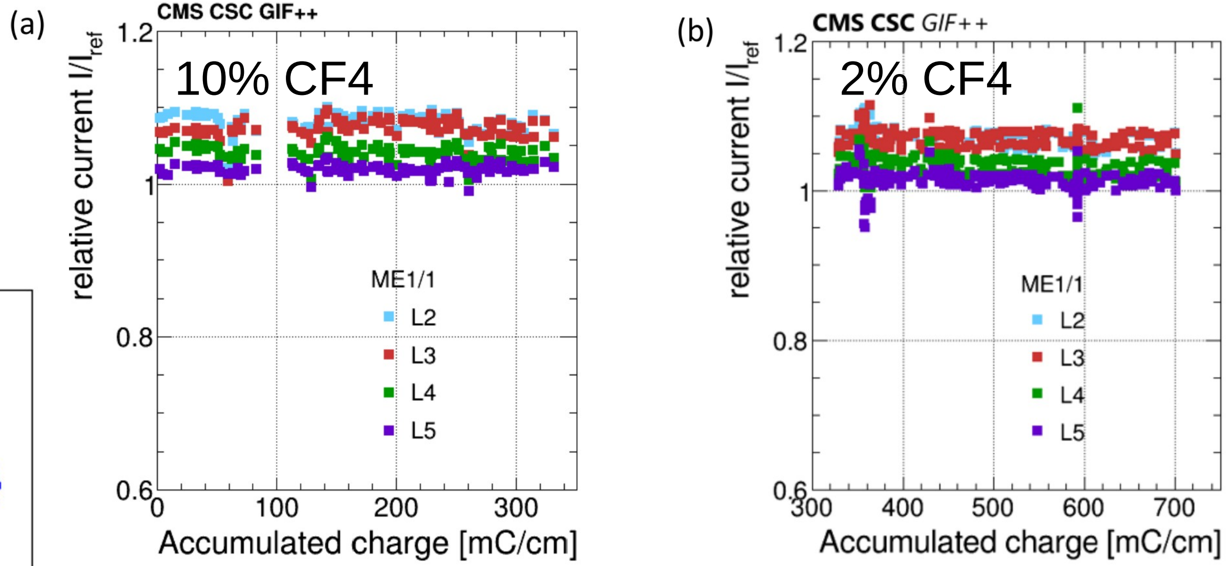
ME2/1:
Q(HL-LHC) ~ 0.13 C/cm
Q(10%CF4) ~ 0.33 C/cm
Q(2%CF4) = 0 C/cm
Q(5%CF4) ~ 0.04 C/cm, ongoing

Longevity tests at GIF++

Resolution with muon beam



Relative gas gain



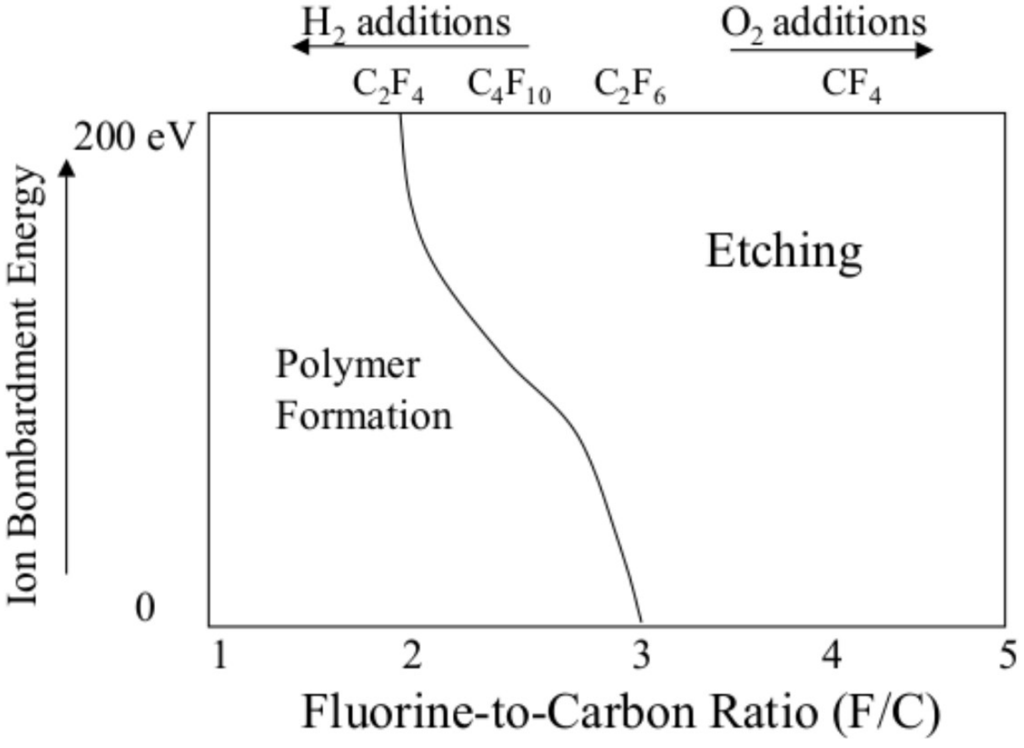
- No significant performance degradation observed so far
- Irradiation ongoing with 5% CF4 – the most probable candidate for future CSC operation

Searches for CF4 replacement

Reasonable low GWP candidates:

- Compounds (gases) **containing F** with
  - relatively short molecular chain (<5)
  - low GWP (<500)
- These candidates should be
  - not hazardous (toxic, flammable, etc.)
  - have reasonable boiling point
- **F/C ratio plays significant role for Si etching**

  => relatively high F/C ratio



Philip D. Rack University of Tennessee

Searches for CF4 replacement

- Most of (hydro)fluorocarbons with relatively short chains and relatively low GWP (<500) are toxic or flammable...

  (exception) – CF3I – tried - too electronegative

- Molecules with long chain may tend to polymerize, i.e. may cause anode/cathode ageing

- **GWP(CF4) = 7390**
- all perfluorated gas compounds except CF3I have larger GWP
- only HFC/HCFC/HFE with GWP < 500 are listed
- Fluorinated alcohols not included due to -OH group
- **Flammability** (may be different in different systems)**:**
  - **F**=flammable, **HiF**=highly flammable, **ExtF**=extremely flammable

			GWP	F:C ratio	remarks
	Carbonyl fluoride	COF2	1	2:1+O	extremely tioxic
	Trifluoroiodomethane	CF3I	0.4	**3:1**	tried, **electronegative**
Halon-1202	CAS: 75-61-6	CBr2F2	231	**2:1**	Irritant; bp=26°C;
Halon-2311	CAS: 151-67-7	C2HBrClF3	41	3:2+Cl	Irritant, toxic. bp=53°C;
HFC-41	Fluoromethane	CH3F	92	1:1	F
HFC-143	1,1,2-Trifluoroethane	CHF2-CH2F	353?	3:2	ExtF
HFC-152	1,2-Difluoroethane	CH2F-CH2F	53	2:2	toxic; bp=31°C
HFC-152a	1,1-Difluoroethane	CHF2-CH3	124	2:2	ExtF; toxic??
HFC-161	Fluoroethane	CH2F-CH3	12	1:2	ExtF
HFC-263fb	1,1,1-Trifluoropropane	CH3-CH2-CF3	76	3:3	F
HFC-272ca	2,2-Difluoropropane	CH3-CF2-CH3	144	2:3	

HFC (single bonds)

Increasing chain length

HFO (a double bond)

			GWP	F:C ratio	remarks
HFC-1132a	Vinylidene fluoride	CH2=CF2	<1	2:2	ExtF, toxic
HFC-1141	Vinyl fluoride	CH2=CHF	<1	1:2	ExtF
HFC-1225ye	1,2,3,3,3-Pentafluoropropene	CF3CF=CHF	<1	5:3	Irritant
HFC-1234yf	2,3,3,3-Tetrafluoroprop-1-ene	CH2=CF-CF3	4	4:3	F
HFC-1234ze(E)	trans-1,3,3,3-Tetrafluoroprop-1-ene	CHF=CH-CF3 (E)	7	**4:3**	
HFC-1243zf	3,3,3-Trifluoropropene	CF3CH=CH2	<1	3:3	F, toxic
HFC-1336mzz(Z)	cis-1,1,1,4,4,4-Hexafluorobut-2-ene (CAS:692-49-9)	CF3-CH=CH-CF3 (Z)	9	**6:4**	no hazards, pb=33°C;
HFC-1336mzz(E)	trans-1,1,1,4,4,4-Hexafluorobut-2-ene (CAS:66711-86-2)	CF3-CH=CH-CF3 (E)	18	**6:4**	**no hazards, pb=8°C;**
HFC-1345zfc	3,3,4,4,4-Pentafluorobut-1-ene	C2F5-CH=CH2	<1	5:4	F
HCFC-1224yd(Z)	cis-1-Chloro-2,3,3,3-tetrafluoroprop-1-ene	CHCl=CF-CF3 (Z)	1	4:3+**Cl**	no hazard info; Ashai Glass (Japan)
HCFC-1233xf	2-Chloro-3,3,3-trifluoroprop-1-ene	CH2=CCl-CF	1	1:3+**Cl**	F, irritant
HCFC-1233zd (E)	trans-1-Chloro-3,3,3-trifluoroprop-1-ene	CHCl=CH-CF3 (E)	4.5	3:3+**Cl**	no hasards; **ATL-RPC**
HCFE-235da2	Isoflurane	CHF2-O-CHCl-CF3	350	5:3+**Cl**+O	toxic

HCFC

Increasing chain length

HFE

HFE-143m		CF3-O-CH3	750	**3:2+O**	**JINST13 P03012; bp= -24°C**
HFE-236fa	CAS: 20193-67-3	CF3-CH2-O-CF3	487	**6:3+O**	bp=62°C
HFE-245fa1	CAS: 84011-15-4	CHF2-CH2-O-CF3	286	**5:3+O**	bp=26°C
HFE-245mc		CF3-CF2-O-CH3	622	**5:3+O**	**JINST13 P03012 ; bp=5°C**
HFE-254cb2	CAS: 425-88-7	CH3-O-CF2-CHF2	359	4:3+O	HiF; irritant
HFE-263fb2	CAS: 460-43-5	CF3-CH2-O-CH3	11	3:3+O	no hazard info; bp=31°C;
HFE-338mmz1	CAS: 26103-08-2	(CF3)2CH-O-CHF2	380	**8:4+O**	bp=40°C;
HFE-347mcf2	CAS: 171182-95-9	CHF2-CH2-O-CF2-CF3	374	**7:4+O**	bp=50°C;
HFE-347mmy1	CAS: 22052-84-2	(CF3)2CF-O-CH3	343	**7:4+O**	Irritant; bp=29°C;
HFE-347mmz1	(Sevoflurane)	CH2F-O-CH(CF3)2	216	**7:4+O**	bp=56°C;
HFE-356mec3	CAS: 382-34-3	CH3-O-CF2-CHF-CF3	101	6:4+O	F, Irritant; bp=53°C;
HFE-356mm1	CAS: 13171-18-1	(CF3)2CH-O-CH3	27	6:4+O	F?. Irritant?; bp=50°C;
HFE-356pcc3	CAS: 160620-20-2	CH3-O-CF2-CF2-CHF2	110	6:4+O	bp=68°C;
HFE-356pcf2	CAS: 50807-77-7	CHF2-CH2-O-CF2-CHF2	265	6:4+O	bp=74°C;
HFE-365mcf3	CAS: 378-16-5	CF3-CF2-CH2-O-CH3	11	5:4+O	F; bp=46°C;

Increasing chain length

Searches for CF4 replacement

- Not so many choices…
- The two ethers from the JINST publication have GWP slightly above the cut (500) but still ~10 times better than CF4
- Molecules with long chain may tend to polymerize, i.e. may cause anode/cathode ageing
- **HFO-1234ze** – does contain F, but **F:C** ratio is not optimal **(4:3)**
- **HFO-1336mzz(E)** - preliminary looks reasonable - better **F:C** ratio **(6:4) ==> availability??!**
  - …and longer molecular chain
- Hydrochlorofluerocarbons (HCFC) – HCFC-1233zd(E) – poor **F:C** ratio **(3:3)** and **chlorine** containing
  ==> of low interests for CSC
- Hydroflueroethers (HFE, R-O-R') – **HFE-245fa1 (5:3)** and **HFE-143m (3:2)**
  - contain oxygen
  - listed in JINST13 P03012 as being of potential interest for gas detectors

  ==> availability??!

- Other possibilities?…

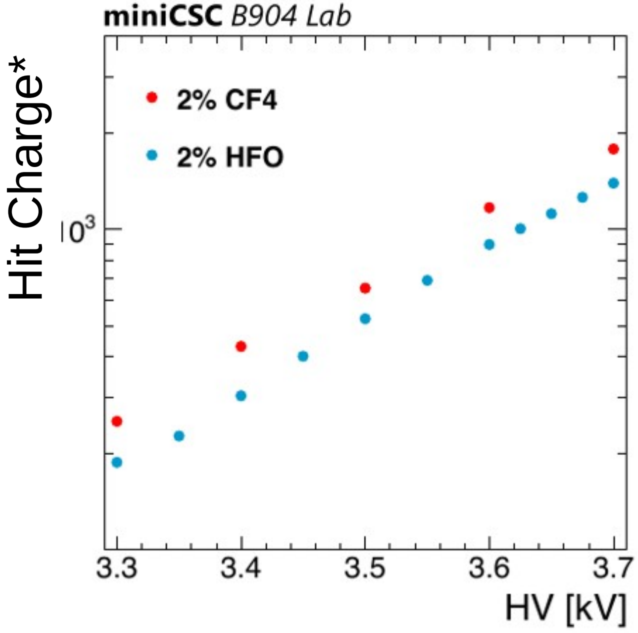
Searches for CF_4 replacement

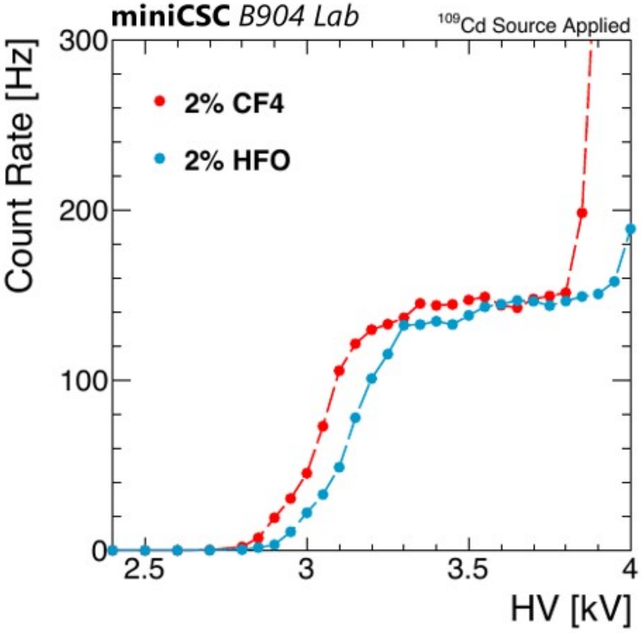
- HFO1234ze studies ongoing – see next slides
- Extremely limited availability and high price for other candidates – still searching for them
- Easier solutions? Adding O_2 to very small admixtures of CF_4? To be studied…
- (Semi-)liquid ethers?

- Meanwhile – development in CMS b904 CSC lab
  - miniCSC production – 6 new prototypes
  - dynamic mixing gas system based on EP-DT design – 4 input channels

HFO1234ze

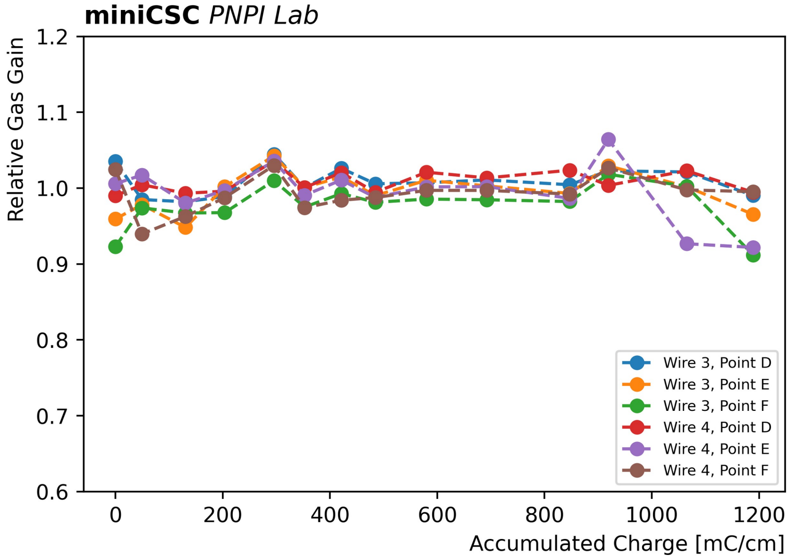
- Quick performance test with miniCSC comparing 2%CF4 and 2%HFO1234ze mixtures
- Just 100V increase in working voltage, good efficiency, reasonable plateau length

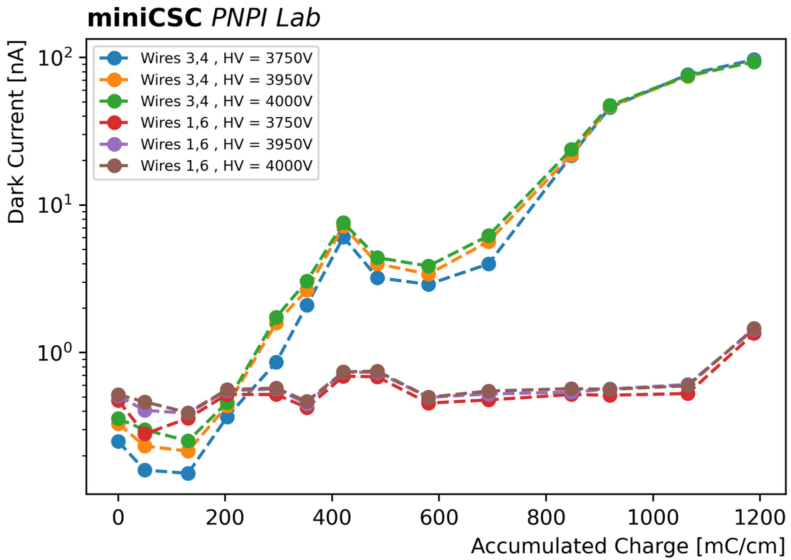




HFO1234ze

- First very accelerated longevity test – local ^{90}Sr irradiation, open loop, no O2/H2O monitoring
- No gas gain reduction up to 1C/cm but **significant increase in dark current** during irradiation



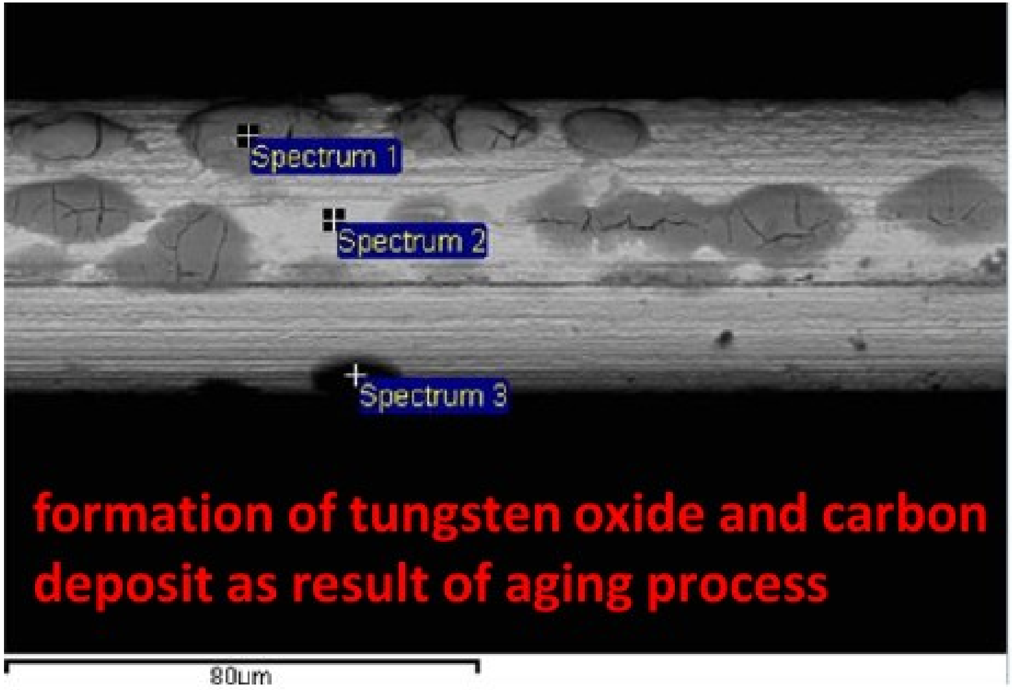


Assuming the extra-currents coming from the irradiation spot: **10 nA from ~2 cm** of irradiated wire **scaled to 200 m of ME2/1s1 wires give 100 uA…**

Test to be repeated in more controllable conditions

HFO1234ze

- EDS/SEM analysis: in contrast to Ar/CO2 and Ar/CO2/CF4 gas mixtures we see significant modification of the anode wire surface with tungsten oxide on the wire surface



All results in weight%

Spectrum	In stats.	C	N	O	F	Al	Si	Cl	W	Au	Total
Spectrum 1	Yes	4.86	0.00	24.42				0.00	69.83	0.88	100.00
Spectrum 2	Yes	5.61	4.75	1.13				0.00	4.57	83.95	100.00
Spectrum 3	Yes	60.95	5.87	3.85	5.86	0.71	0.21	0.69	0.37	21.47	100.00

Irradiated electrode analysis

- Close collaboration with **University of Belgrade**
- Available methods:
- Moreover, we are discussing possible theoretical/MC considerations of chemical plasma processes – potentially useful but require additional manpower

Deposit was analyzed using non-destructive techniques

- ***Scanning Electron Microscopy (SEM)***

produces 3D images of a sample by scanning the surface with a focused beam of electrons which interact with atoms in the sample, thus giving information about the surface morphology of the material

- ***Energy Dispersive X-ray Spectroscopy (EDS)***

characteristic X-rays produced by the interaction of electrons with the sample are used to map the distribution and estimate the abundance of elements in the sample, i.e., elemental analysis of the material surface (penetration depth 0.5 - 2 µm, detection of elements Z≥5, detection limit: ~ 0.1 wt%)

- ***X-Ray Diffraction Analysis (XRD)*** - *Long range order in crystal structures:*

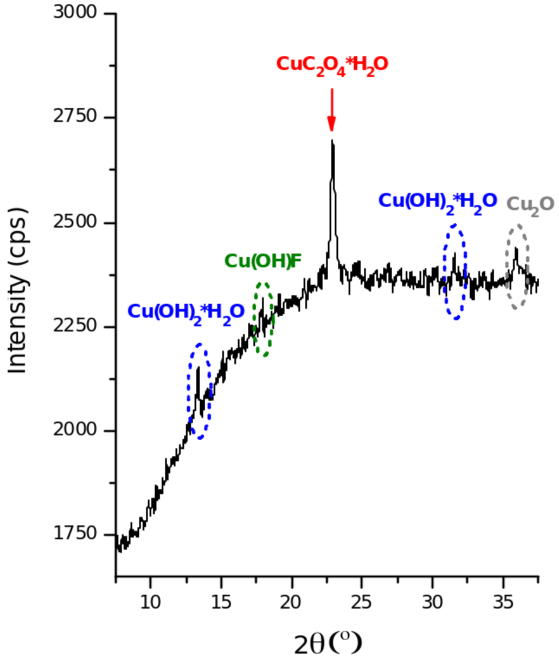
measures the angle of the beam scattered from crystal planes thus giving the information on periodic atomic arrangements in a given material, which is used for identification of crystal material (analyzed area: ~15x15mm, penetration depth < 15 µm, detection limit: 1%)

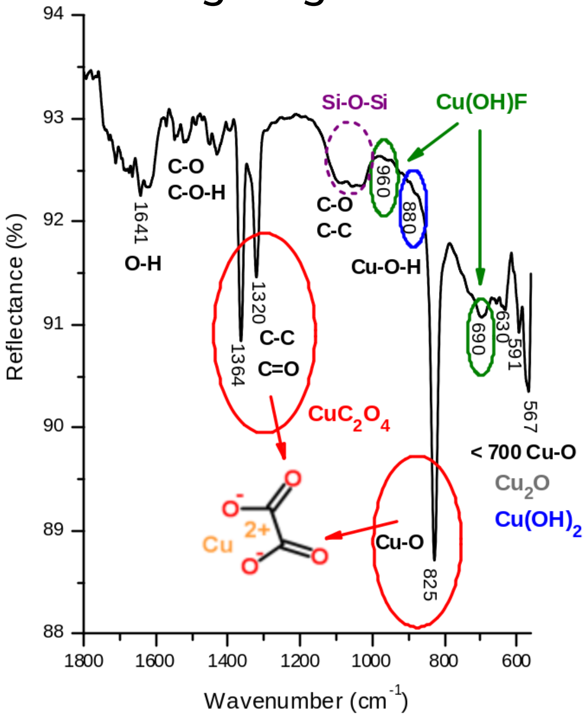
- ***Fourier Transform Infrared Spectroscopy (FTIR)*** - *Middle range order in crystal and amorphous structures:*

measures interaction of infrared radiation with chemical bonds within a material thus giving information on functional groups within a molecule and energy of chemical bonds (penetration depth 0.5 - 2 µm, detection limit: ~0.1 wt%)

Irradiated electrode analysis

University of Belgrade – just an example of ongoing studies – **work in progress!!**





- **Dominant crystal phase is $CuC_2O_4*H_2O$**
- **Crystal phases $Cu(OH)_2*H_2O$, Cu(OH)F, Cu_2O are present in lower amount**
- **Si-O-Si bonds and C-C/C-H bonds indicate presence of amorphous polymers**

Next steps

- Irradiation at GIF++ with 5% CF_4 – ongoing
- 904 lab – finalizing the setup development – dynamic gas mixing system + set of miniCSC

  => many thanks to CERN EP-DT gas group (gas system) and to CMS mechanical workshop (miniCSC)
  - Local irradiation with controllable conditions (O_2/H_2O monitoring)
  - Scheduled performance and longevity tests with HFO1234ze, recuperated CF_4, CF_4+O_2
- Searching for alternative gases of interest on marked (time-consuming..)
- Work on irradiated material analysis together with Belgrade
- …lack of manpower… Some of topics may be common for CMS CSC and the RPC EcoGas (material analysis, market search, experience exchange)? Will be happy to combine!

backup

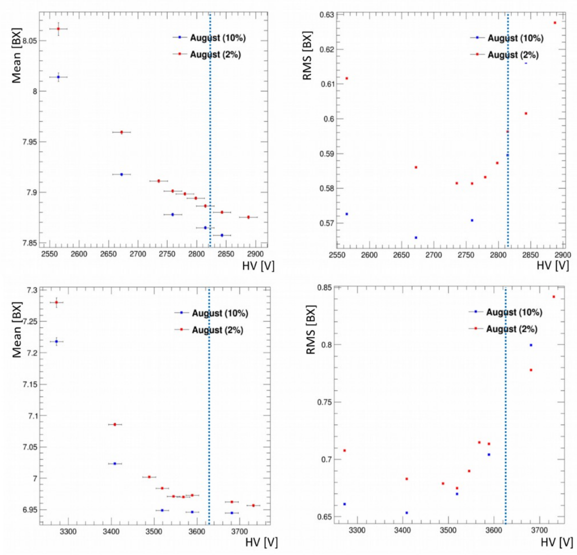
Update on comparison of 10% and 2% CF4 mix

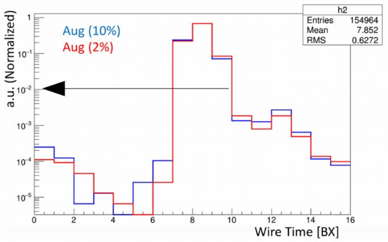
Wire hit time distribution:

40%Ar+50%CO2+10%CF4 vs 40%Ar+58%CO2+2%CF4

work in progress
B.Joshi (UF)

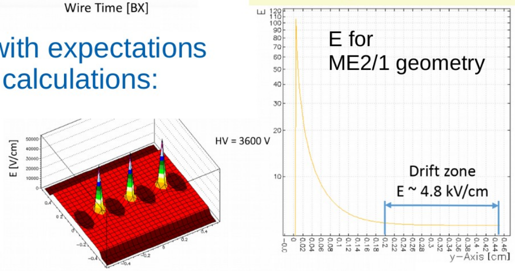
Mean and RMS of the distribution

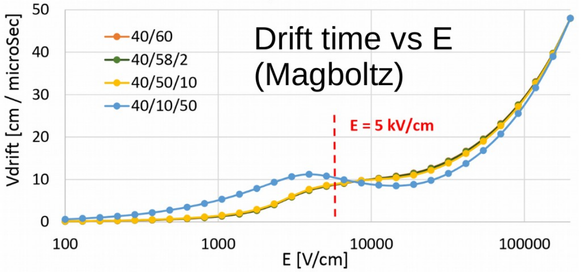




In agreement with expectations from Magboltz calculations:

S. Nasybulin (PNPI)





No noticeable difference in signal timing for 2% and 10% CF4 mixtures