



# STUDY of ECO-FRIENDLY GAS REPLACEMENTS FOR PARTICLE DETECTORS IN HIGH-ENERGY PHYSICS

AGING TEST STATION, PNPI, Gatchina

## I. Eco-friendly gas mixtures study:

- Analysis of the gas components
- Longevity resource problem
- Gas mixtures straw-test
- Results for CF3I gas mixtures
- Conclusion

## II. CF3I gas mixtures study with CSC prototype:

- CSC prototype
  - Test results for 1%, 5% and 10% CF3I containing gas mixtures
- N-pentane

## III. Si aging recovering method

- Short prehistory
- HERMES (DESY) MWPC curing from Si
- ATS straw multiple curing with 80%CF<sub>4</sub>+20%CO<sub>2</sub>

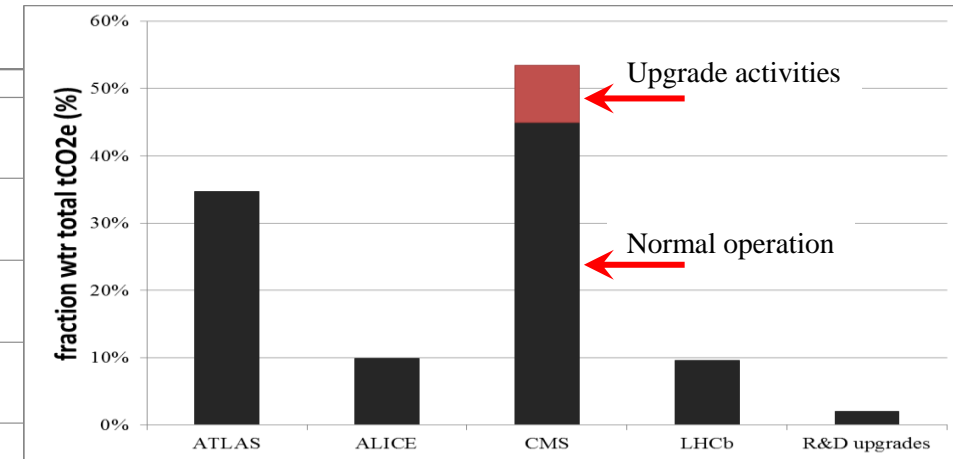
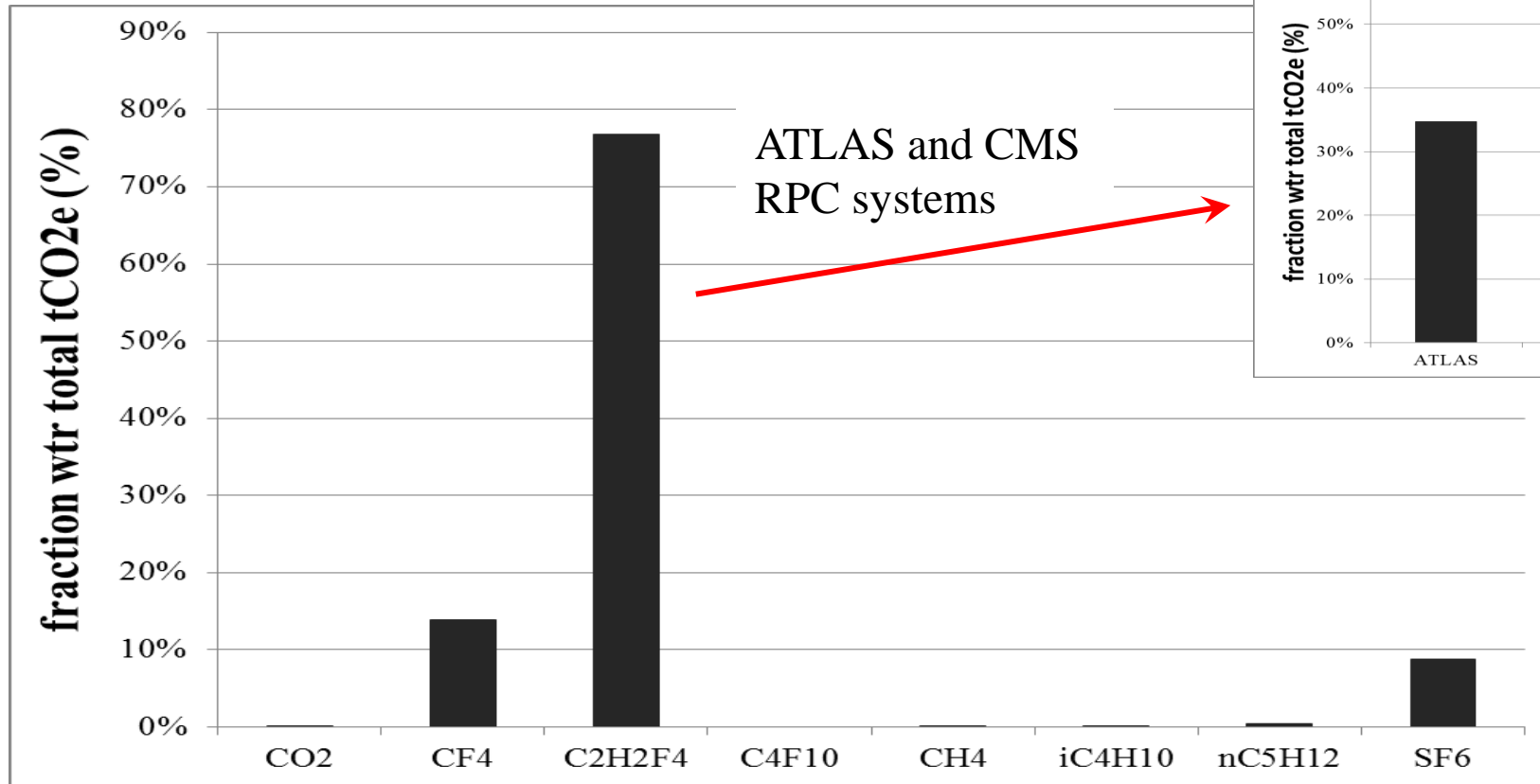
## IV Planning & Outlook

November, 29 2015

Г.Е. Гаврилов, семинар ОФВЭ

- Operational costs and Greenhouse Gas (GHG) emission
- Total emission:  $\sim 127100 \text{ tCO}_2\text{e}$
- Main contribution  $\text{C}_2\text{H}_2\text{F}_4$ (R134a),  $\text{CF}_4$ ,  $\text{SF}_6$

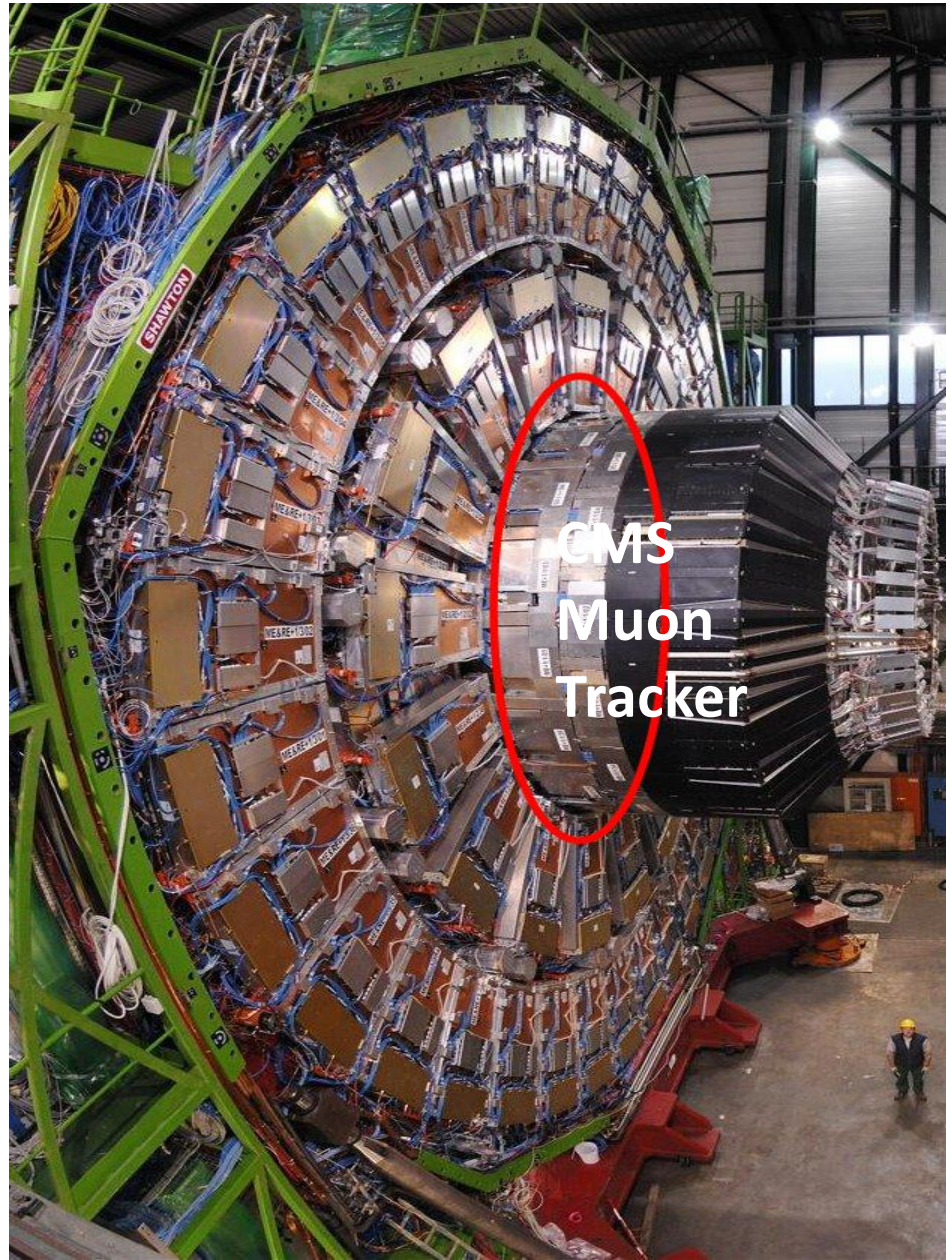
Gas emissions from particle detectors



# I. Исследование новых экологически безопасных газовых смесей Eco-friendly gas mixtures study:

## Экологическая проблема:

- 40%Ar+50%CO<sub>2</sub>+**10%CF<sub>4</sub>**
- Общий объем газа, поступающий в камеры CSC 180 м<sup>3</sup> в сутки
- Рециркуляция 90%
- Выброс **CF<sub>4</sub>** - 4380 тонн CO<sub>2</sub> в год в GWP единицах  
*Для сравнения выброс ТЭЦ 11-12 тыс. тонн CO<sub>2</sub> в год*
- Время жизни CO<sub>2</sub> в атмосфере 50-200 лет
- **Время жизни CF<sub>4</sub> в атмосфере 50 000 лет**



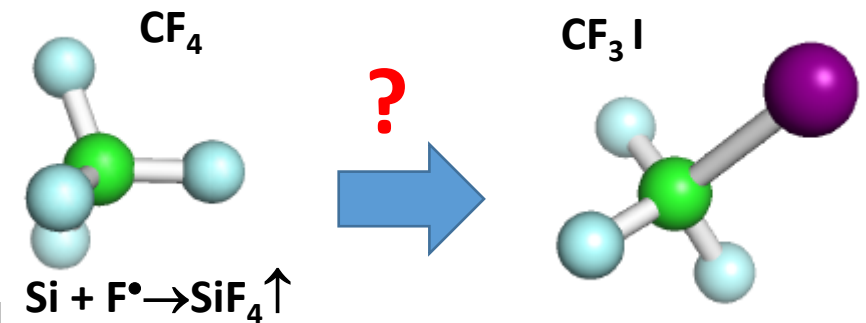
**1 kg CF<sub>4</sub>** в  
единицах  
GWP  
эквивалентен  
**7390 kg CO<sub>2</sub>**

**GWP (Global Warming Potential) –**  
единицы  
воздействия 1 kg  
CO<sub>2</sub> на атмосферу

Химическая формула	Индекс	GWP (Global-Warming Potential)	Время жизни в атмосфере, лет
CO <sub>2</sub>	R744	1	50-200
CF <sub>4</sub>	R14	7390	50 000
CF <sub>3</sub> I	R13I	0.4	<1
C <sub>2</sub> F <sub>6</sub>	R116	12200	10 000
C <sub>3</sub> F <sub>8</sub>	R218	8830	7 000
c-C <sub>4</sub> F <sub>8</sub>	RC318	10300	3 000

В таблице газы, широко применяемые в микроэлектронике для травления кремниевых микросхем все они, обнаружены в атмосфере обладают большим GWP

**Только CF<sub>3</sub>I не даёт вклада в парниковый эффект: GWP= 0.4 за 100 лет**



**Возможность травления Si фтороуглеродами**

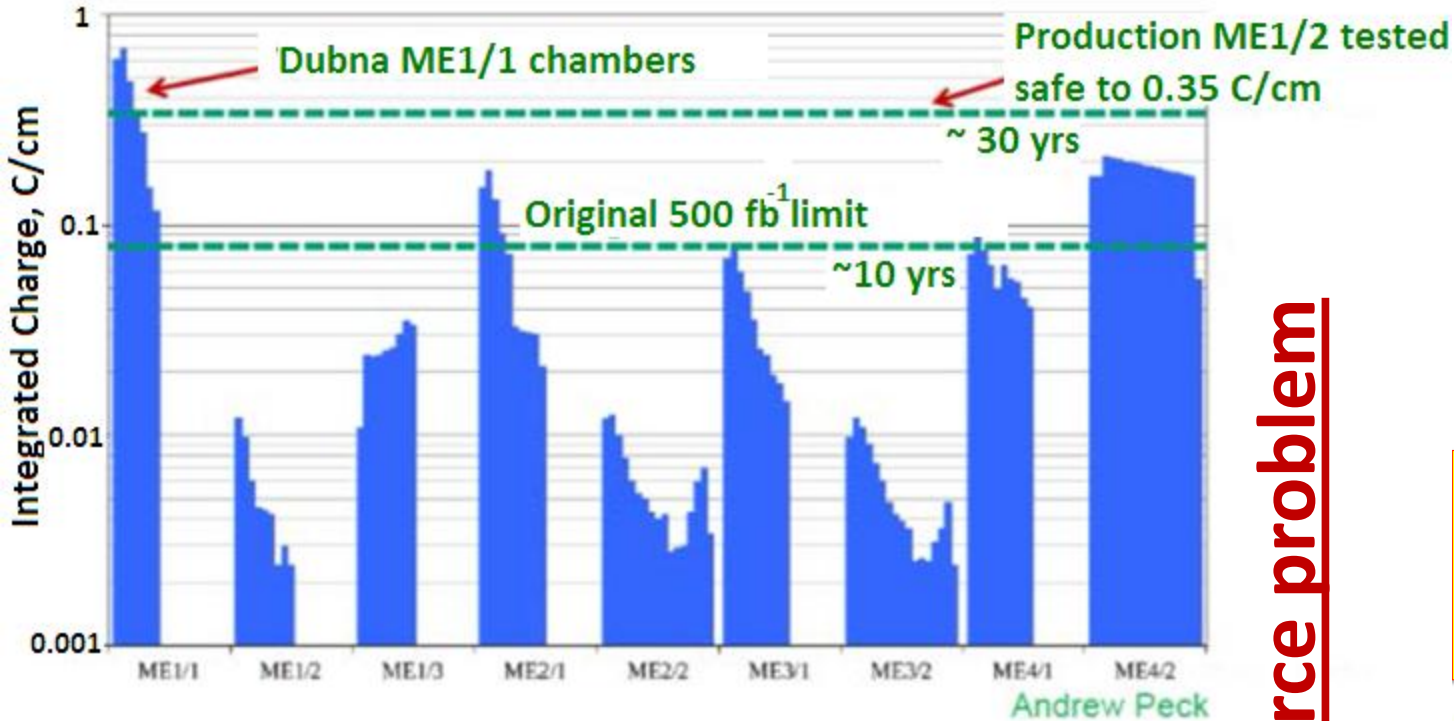
Рабочий газ	Параметр F/C	Характеристика процесса	
		без ионной бомбардировки (≤15 эВ)	с ионной бомбардировкой (≥100 эВ)
CF <sub>4</sub>	4	травление	травление
CF <sub>3</sub> I	3	травление	травление
C <sub>2</sub> F <sub>6</sub>	3	нет травления	травление
C <sub>3</sub> F <sub>8</sub>	2,67	полимеризация	травление
C <sub>2</sub> F <sub>4</sub>	2	полимеризация	полимеризация

D. Yangd, G. Chend et al. Properties of potential Eco-friendly gas replacements for particle detectors in High-Energy Physics, CERN-OPEN-2015-004, 02 May 2015.

Antti Nuottajärvi, Tarmo Suppala, Plasma Etching and Integration with Nanoprocessing, [Ion Beams in Nanoscience and Technology](#) Part of the series [Particle Acceleration and Detection](#) pp 251-263, Nanoscience Center, University of Jyväskylä

Григорьев Ф.И. Плазмохимическое и ионно-химическое травление в технологии микроэлектроники: Учебное пособие / Московский государственный институт электроники и математики. М. 2003. 48 с.





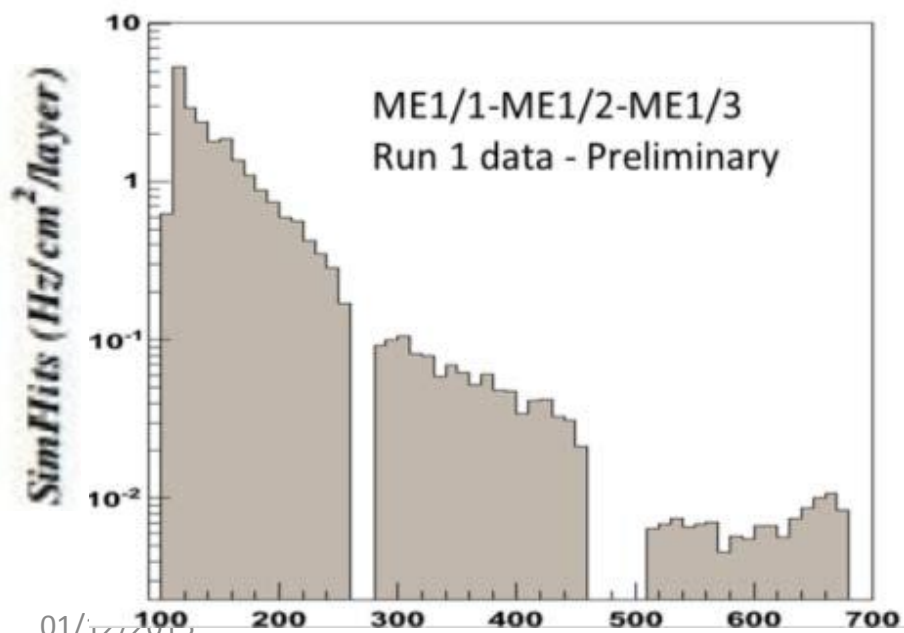
## Currents observed in Run 1 and Run 2

- ME1/1:  $I \approx 2 \mu\text{A per } 10^{34} \text{ cm}^{-2}\text{s}^{-1}$
- ME2/1 (HV#1):  $I \approx 5 \mu\text{A per } 10^{34} \text{ cm}^{-2}\text{s}^{-1}$
- Current per lumi is about the same at 8 and 13 TeV

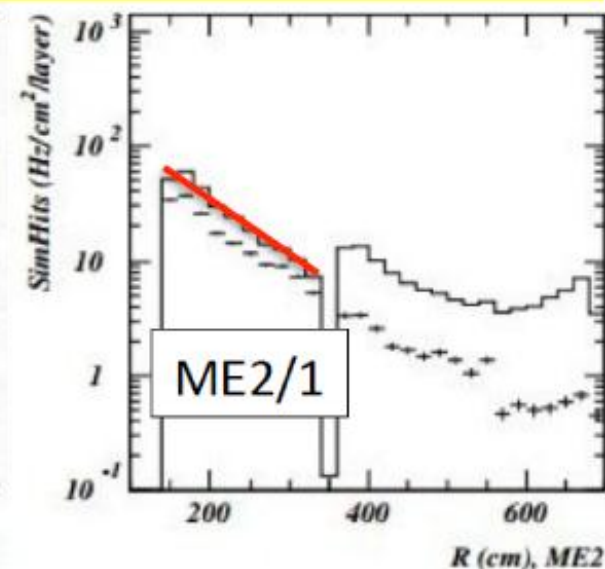
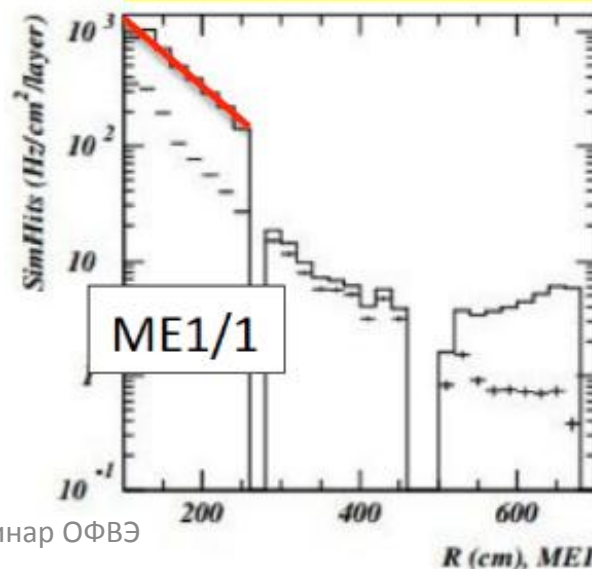
## Extrapolation toward HL LHC $L = 5 \times 10^{34} \text{ cm}^{-2}\text{s}^{-1}$

- ME1/1:  $I_{\text{HL-LHC}} \approx 10\text{-}15 \mu\text{A}$
- ME2/1 (HV#1):  $I_{\text{HL-LHC}} \approx 25 \mu\text{A}$

**Longevity resource problem**



## CMS NOTE 2002/007: 14 TeV, $10^{34} \text{ cm}^{-2}\text{s}^{-1}$



# What is the main origin of background in the CSC?

The major source of background radiation in the CMS region away from the beam pipe ( $R > 50\text{cm}$ ) comes from neutrons with energies up to few hundred MeV (testing with reactor-n not exhaustive).

- Background hits in the CSC are mostly generated by Compton electrons produced by 0.1-1 MeV photons irradiated by neutron-capturing nuclei.
- The avalanche total deposited charge is typically 3-4 times larger than that released by minimum ionizing particles ( $\sim 1\text{pC}$  at the CSC operating voltage of 3.6 KV).

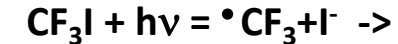
Neutrons (and protons) with energy above 20 MeV are also the major source of malfunctioning (SEU) or incremental damage to electronics components caused by hadronic interactions in the silicon

# Gas components - candidates for CSC

## Electron Interactions Cross Section with Plasma Processing Gases

From aging point of view:

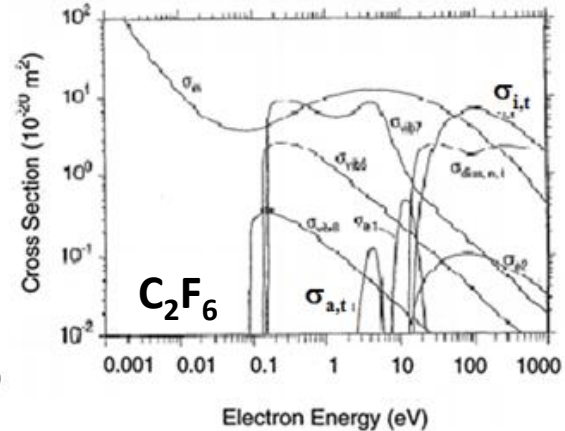
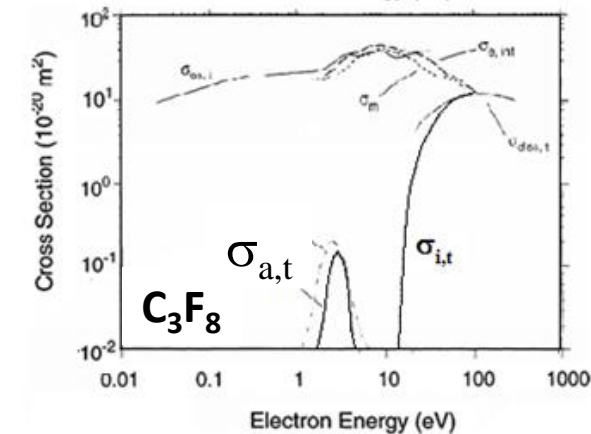
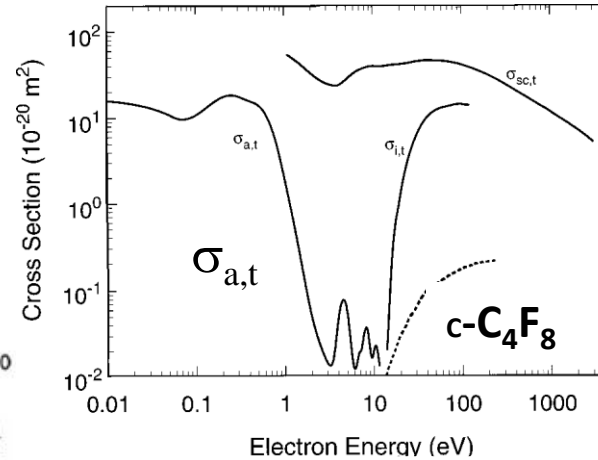
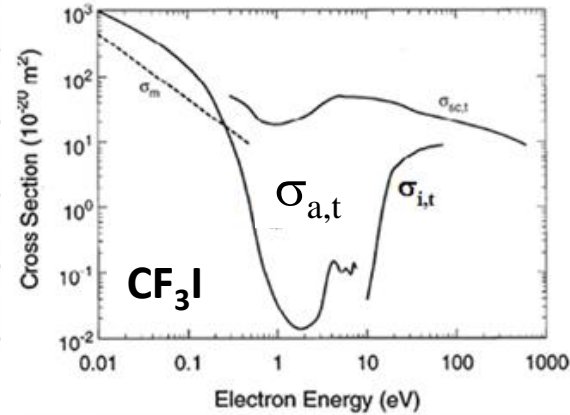
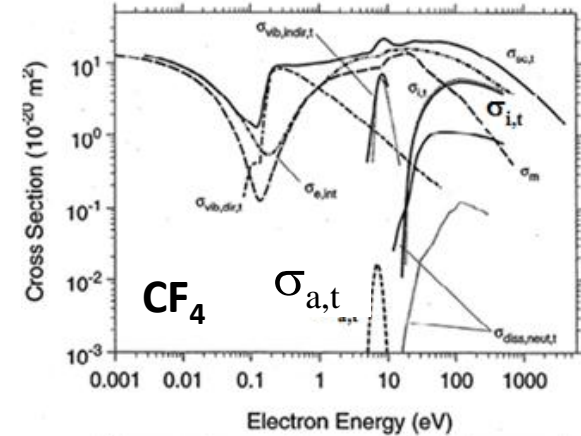
**CF<sub>3</sub>I is capable to substitute CF<sub>4</sub> because**



**Si etching by CF<sub>3</sub>I is possible because the binding energies of C-I (2.4 eV) and C=C (2.8 eV) that is << C-F (5.6 eV)**

Daiki Nakayama et al., Highly selective silicon nitride etching to Si and SiO<sub>2</sub> for a gate sidewall spacer using a CF<sub>3</sub>I/O<sub>2</sub>/H<sub>2</sub> neutral beam, [Journal of Physics D: Applied Physics](#)

Samukawa, Seiji et al., Environmentally harmonized CF<sub>3</sub>I plasma for low-damage and highly selective low-k etching, [Journal of Applied Physics](#); Mar2008, Vol. 103 Issue 5, p053310

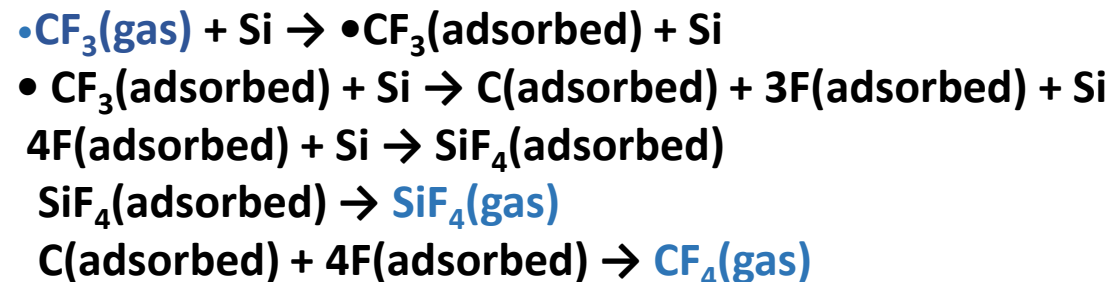


### Gas mixture to be studied

Gas mixture to be studied	$\langle dE/dx \rangle$ , MeV·g/cm <sup>3</sup>	X <sub>0</sub> , g/cm <sup>2</sup>	N <sub>p</sub> , cm <sup>-1</sup>
40%Ar+50%CO <sub>2</sub> +10%CF <sub>4</sub>	1.67	29.21	36.72
40%Ar+55%CO <sub>2</sub> +5%CF <sub>3</sub> I	1.667	28.31	45.97
40%Ar+59%CO <sub>2</sub> +1%CF <sub>3</sub> I	1.67	29.29	36.43
40%Ar+50%CO <sub>2</sub> +10%CF <sub>3</sub> I	1.64	27.07	57.56

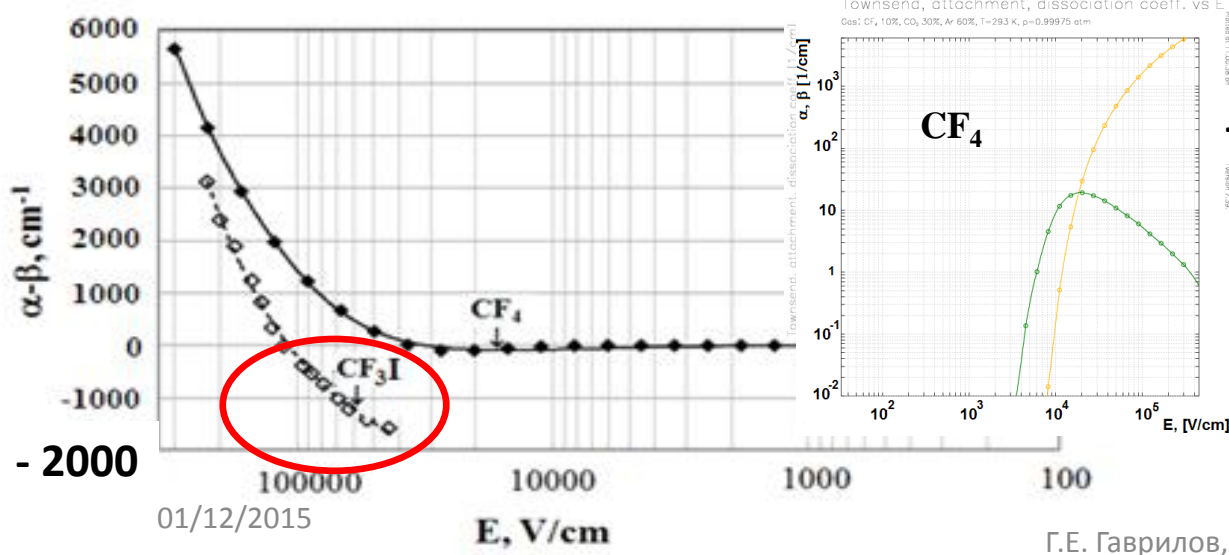
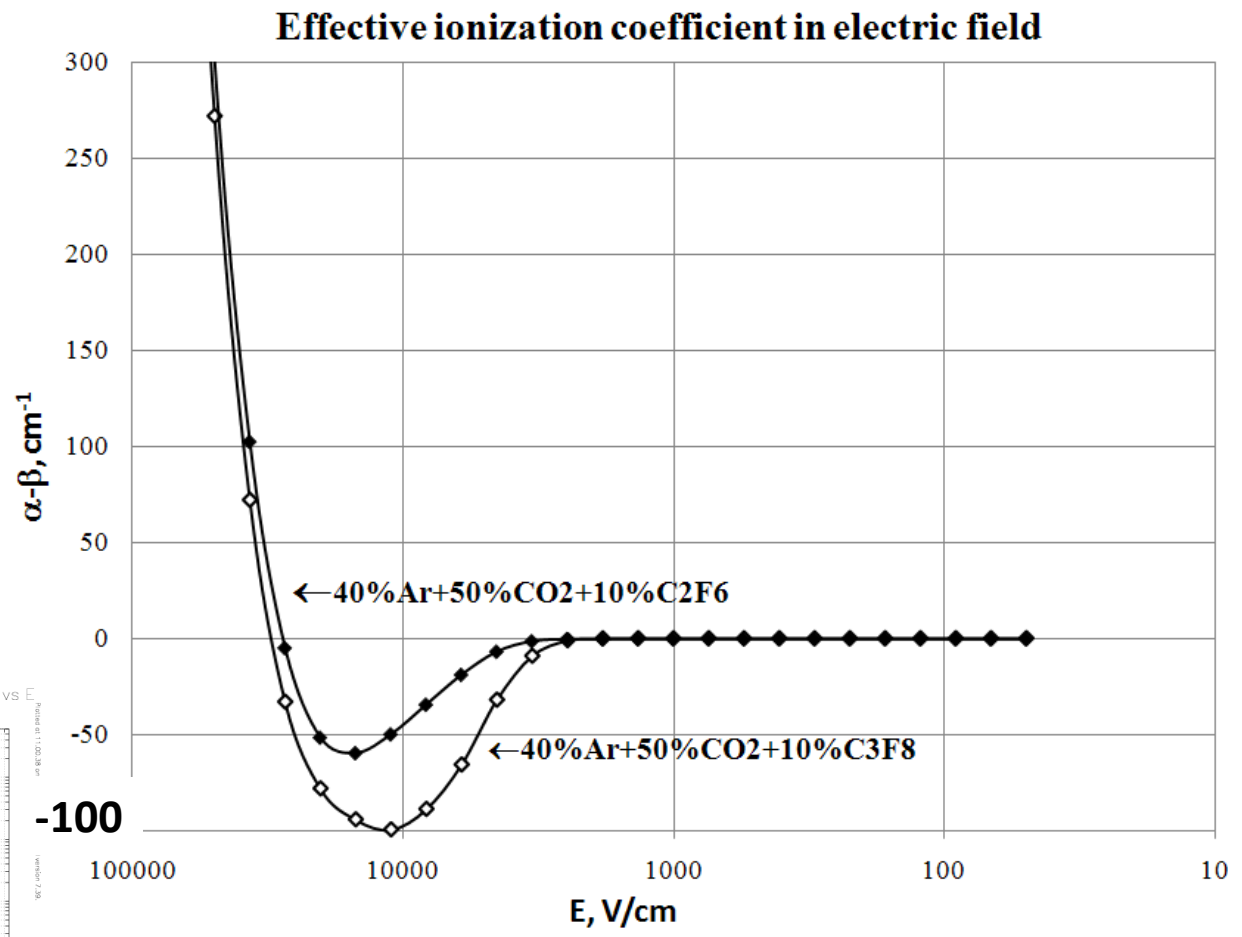
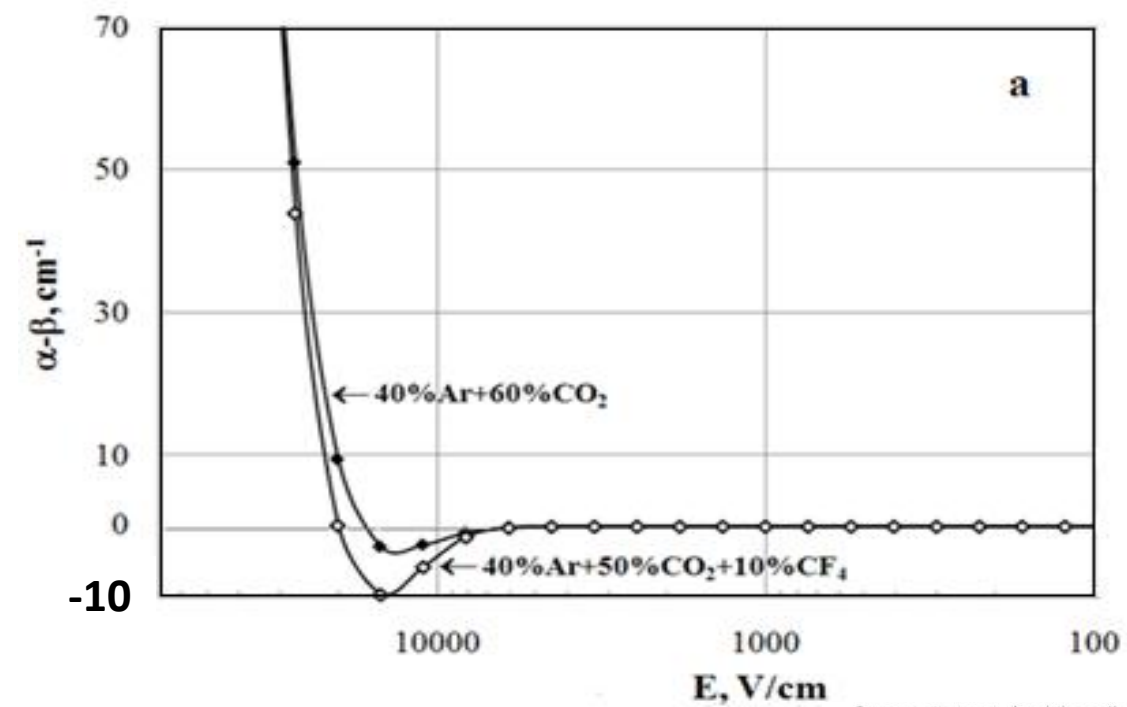
S.R. [Hunter](#), J.G. [Carter](#), L.G. [Christophorou](#),  
Electron motion in the gases CF<sub>4</sub>, C<sub>2</sub>F<sub>6</sub>, C<sub>3</sub>F<sub>8</sub>, and n-C<sub>4</sub>F<sub>10</sub>,  
[Phys Rev A](#). 1988 Jul 1; 38(1): 58-69.

Si etching with CF<sub>3</sub>I



chemical adsorption  
dissociation  
chemical reaction  
desorption

# GARFIELD simulation of $(\alpha-\beta)$ – effective ionization and data from publications



**Attachment for electrons  $E \sim 0.01-0.5$  eV in CF<sub>3</sub>I is 200 high than in CF<sub>4</sub>**



## Gas components - candidates for CSC

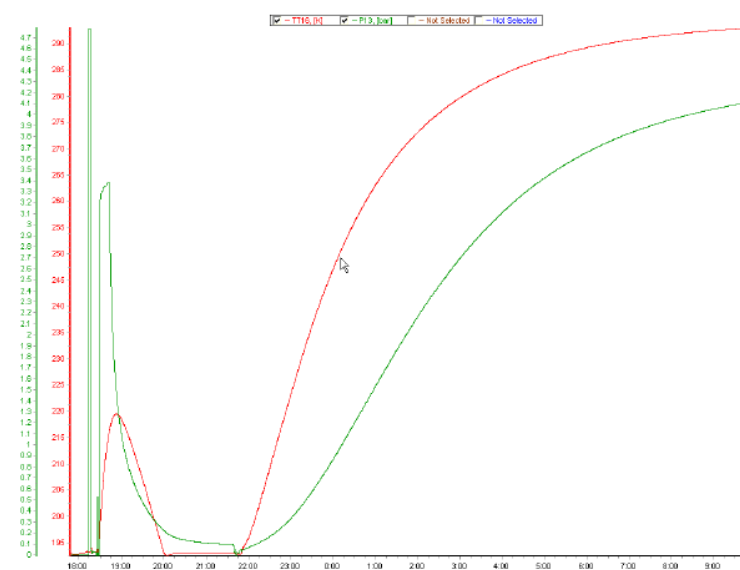


Рис. 4: Графики температуры и давления в системе в процессе очистки. Красный цвет температура, зеленый давление

### Properties

#### Chemical formula

**CF<sub>3</sub>I**

#### Molar mass

195.91 g/mol

Colorless odorless gas

2.5485 g/cm<sup>3</sup> at -78.5 °C

2.3608 g/cm<sup>3</sup> at -32.5 °C

#### Density

#### Melting point

-110 °C (-166 °F; 163 K)

#### Boiling point

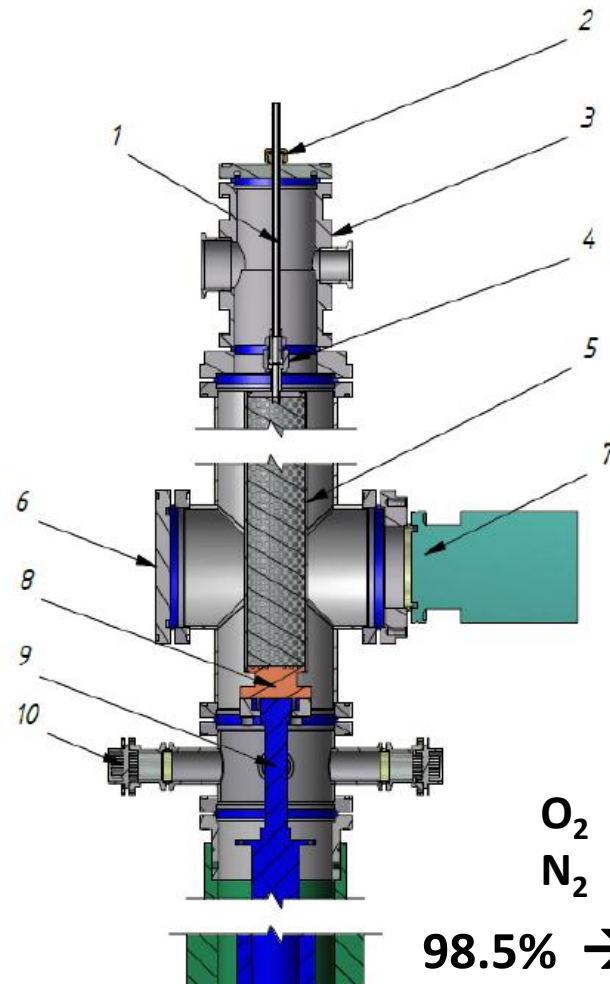
-22.5 °C (-8.5 °F; 250.7 K)

#### Solubility in water

Slightly

#### Vapor pressure

541 kPa



O<sub>2</sub> ~ 0.16%

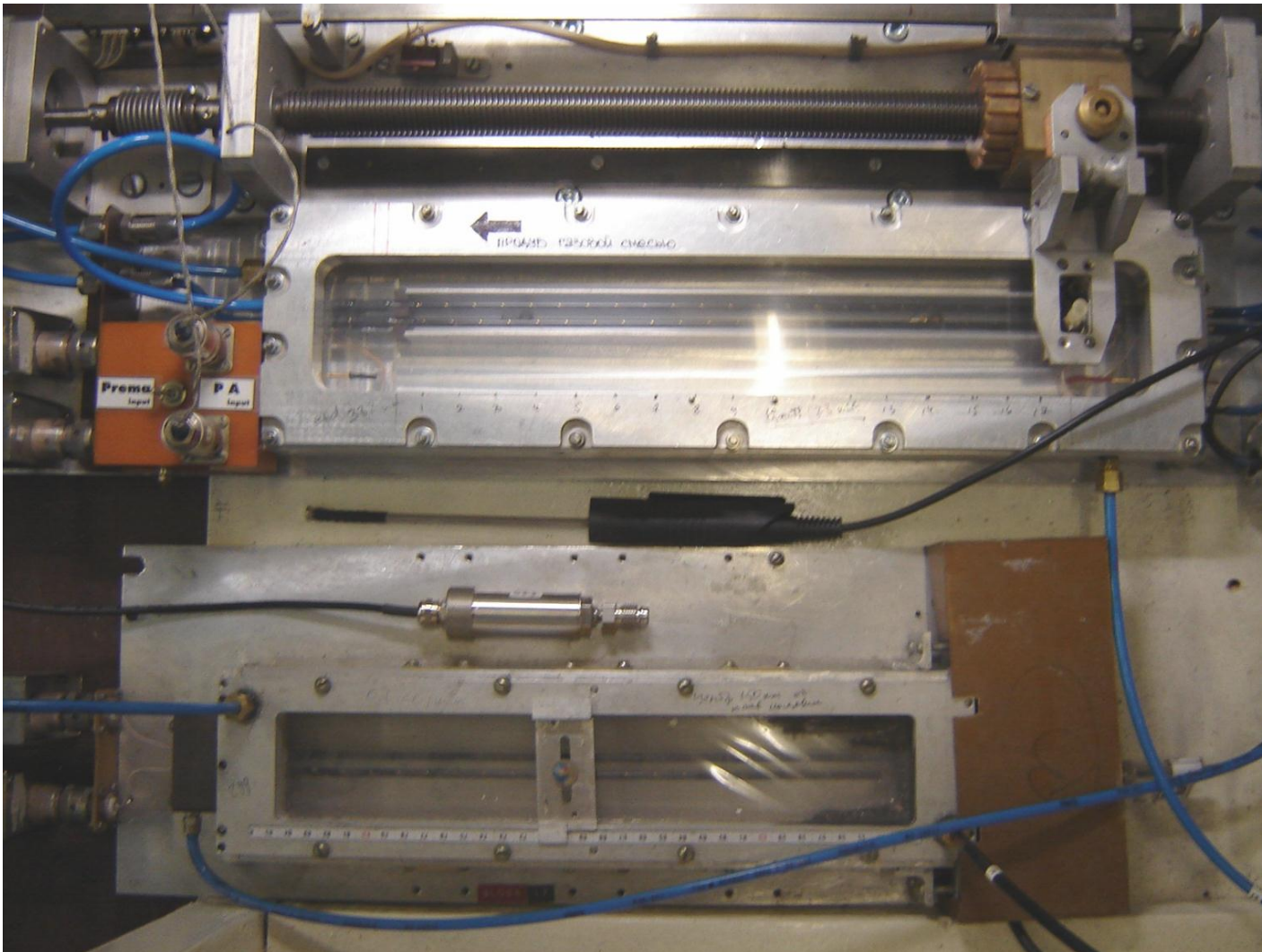
N<sub>2</sub> ~ 0.74%

98.5% → 99.99%

Рис. 2: Схема установки. Внешняя коммутация газовых линий и ряд подробностей опущены. 1) газовая трубка; 2) скользящее уплотнение 3) верхний вакуумный адаптер; 4) криогенное разъемное соединение 5) снабженная внутренней медной сеткой конденсационная ячейка 6) основной вакуумный объем; 7) турбомолекулярный насос; 8) тепловой мост, снабженный нагревателем; 9) головка криогенератора 10) сигнальный разъем  
Г.Е. Гаврилов, семинар ОФВЭ



# Gas mixtures straw-test set up



- Two identical blocks with 2 straws:
1. Test block (top) is irradiated by  $\text{Sr}^{90}$  with rate 15 MHz per 3.5 cm spot
  2. Gas gain monitoring with  $^{55}\text{Fe}$   $\gamma$ -source,  $E_{\gamma}=6$  keV
  3. Control block (bottom) is not irradiated and used for compensation of the air pressure and temperature influence

$$\frac{\Delta G}{G} = k \left( \frac{\Delta P}{P} - \frac{\Delta T}{T} \right)$$

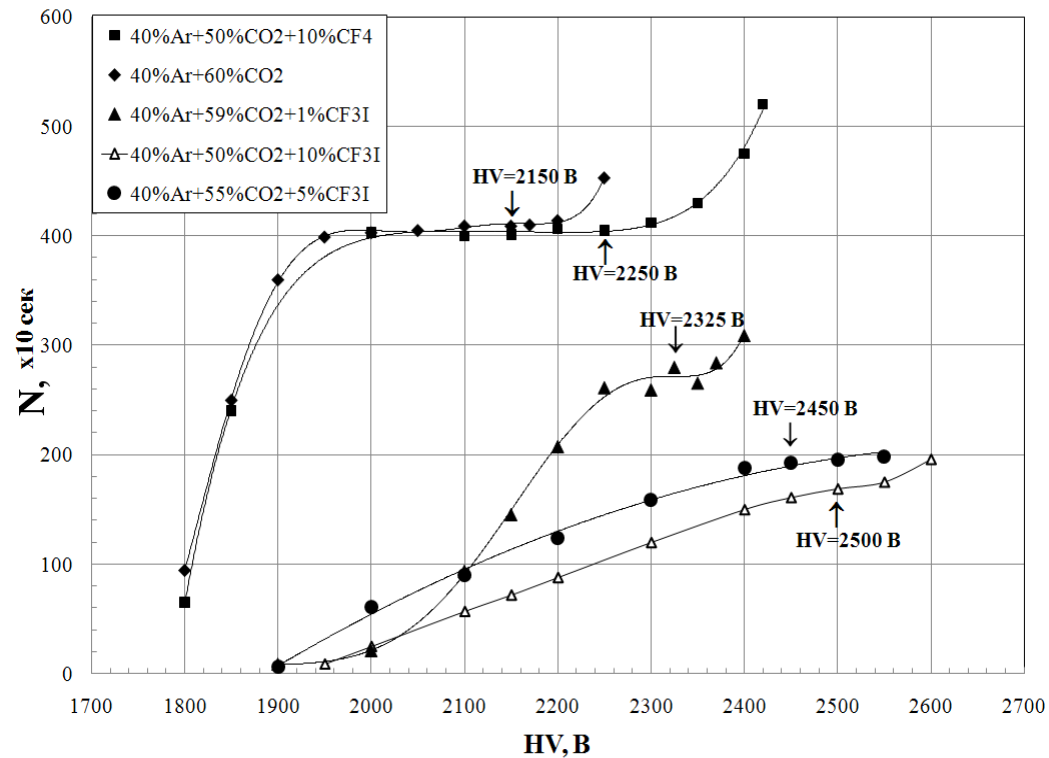
- 50  $\mu\text{m}$  W anode wire, 5% weight of Au coating
- 4 mm straw diameter, 38 mm length
- 72  $\mu\text{m}$  thickness of the kapton made cathode wall

Both aging tests and gas mixtures study are available



# Gas mixtures straw-test

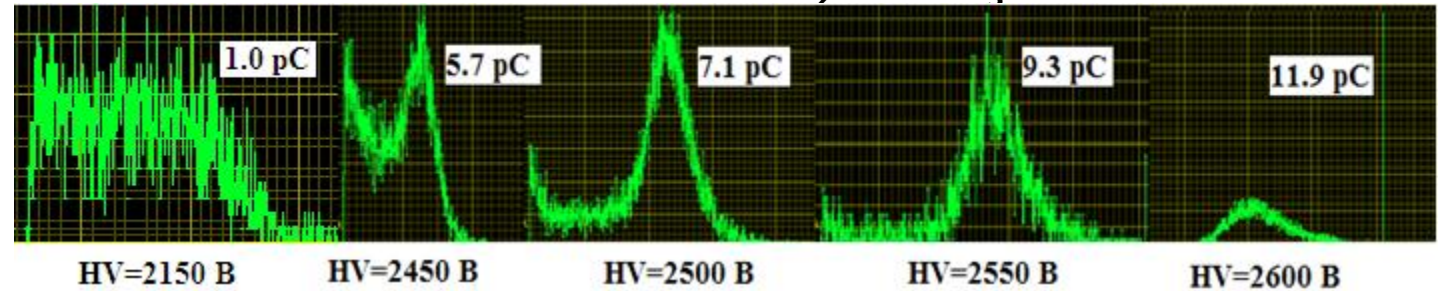
## Счетные характеристики от $^{55}\text{Fe}$



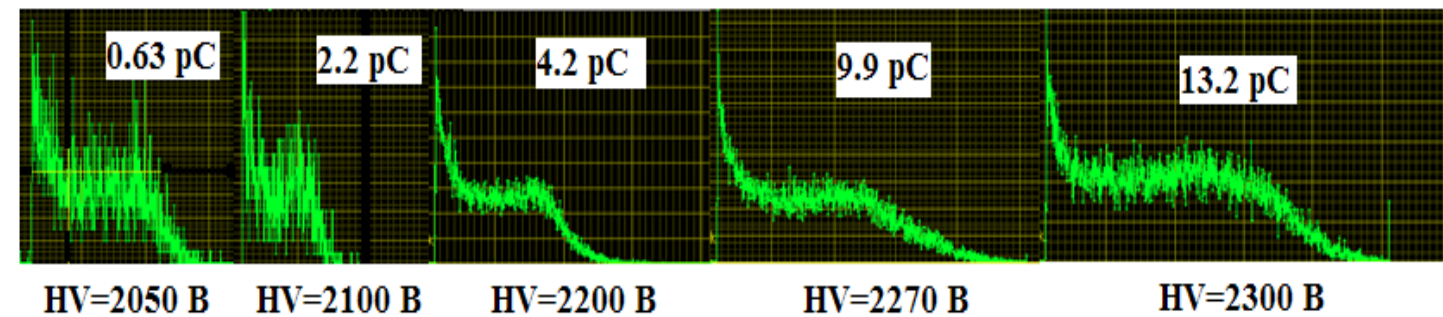
Count rates plateau appears at  
Limited Streamer mode  $Q \gg 2\text{pC}$

## Амплитудные спектры

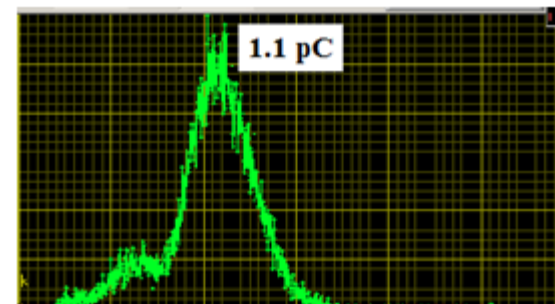
40%Ar+50%CO<sub>2</sub>+10%CF<sub>3</sub>I

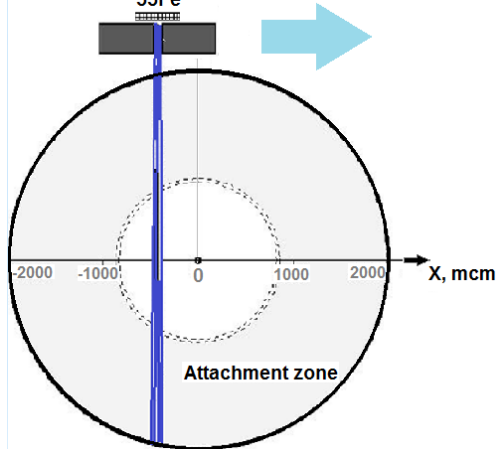


40%Ar+59%CO<sub>2</sub>+1%CF<sub>3</sub>I

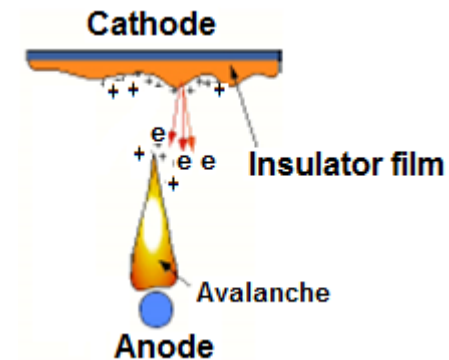
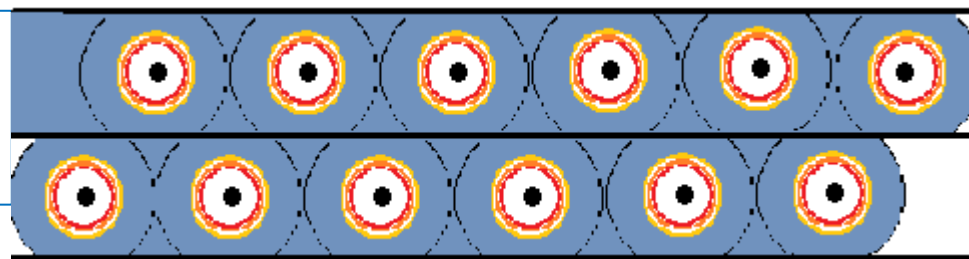


40%Ar+50%CO<sub>2</sub>+10%CF<sub>4</sub>

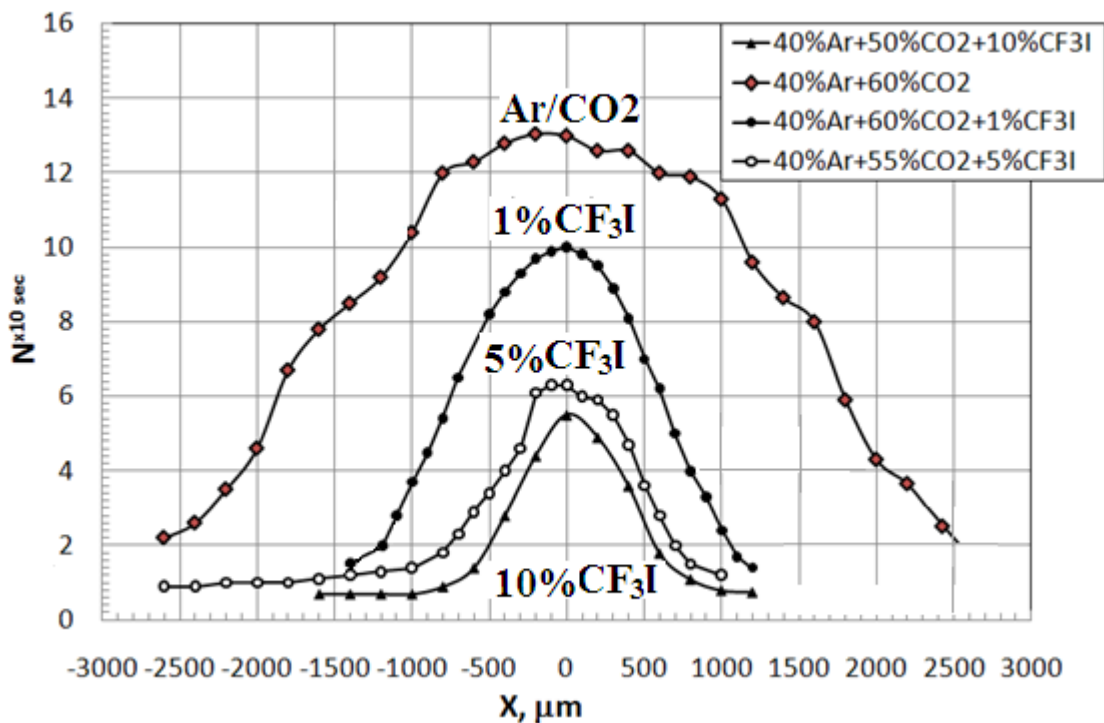




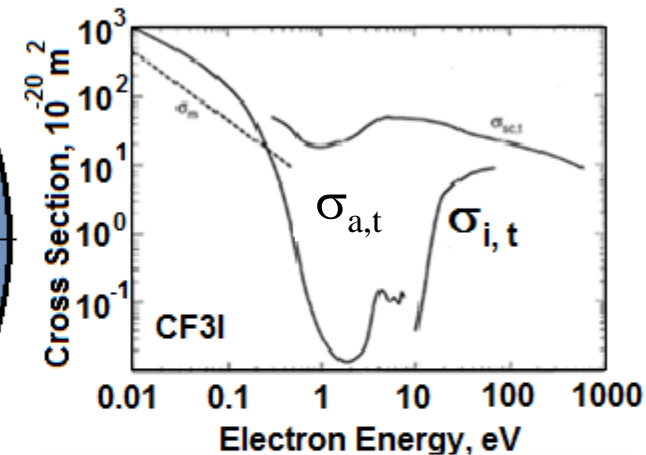
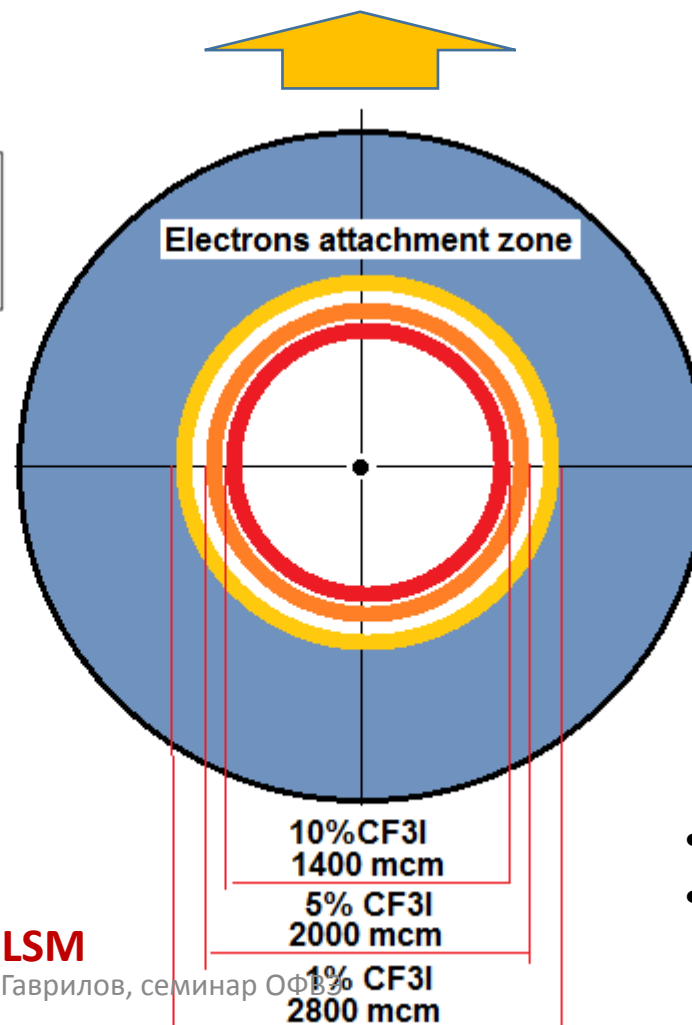
with high angle selectivity, operating in Limited Streamer Mode



$^{55}\text{Fe}$  count rates across straw



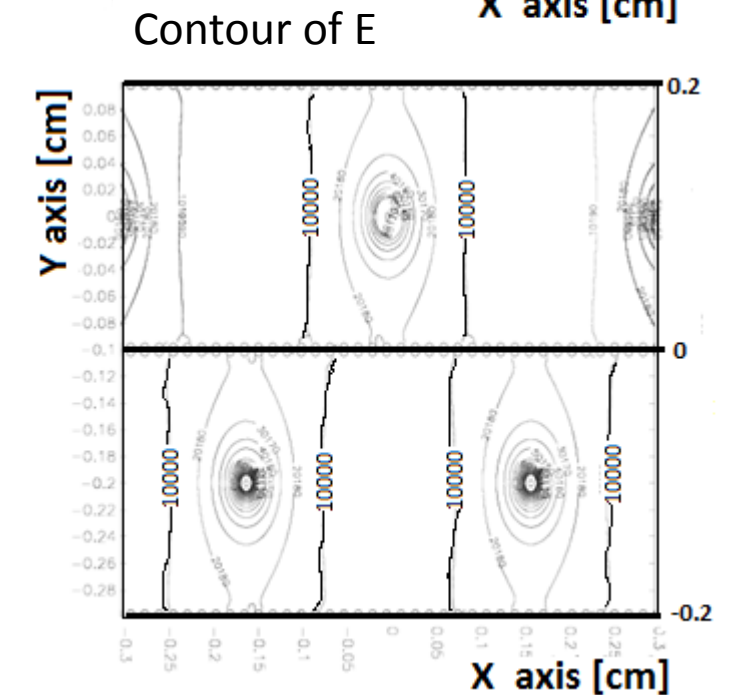
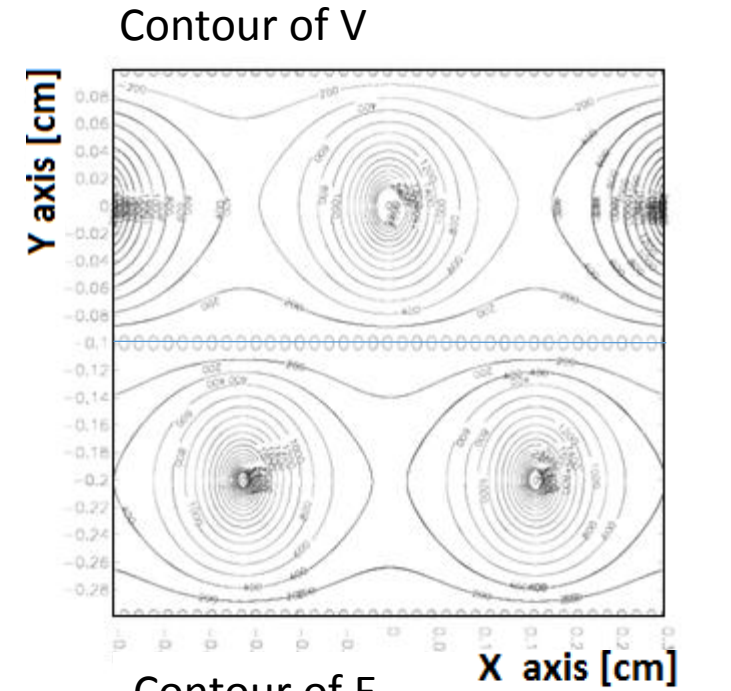
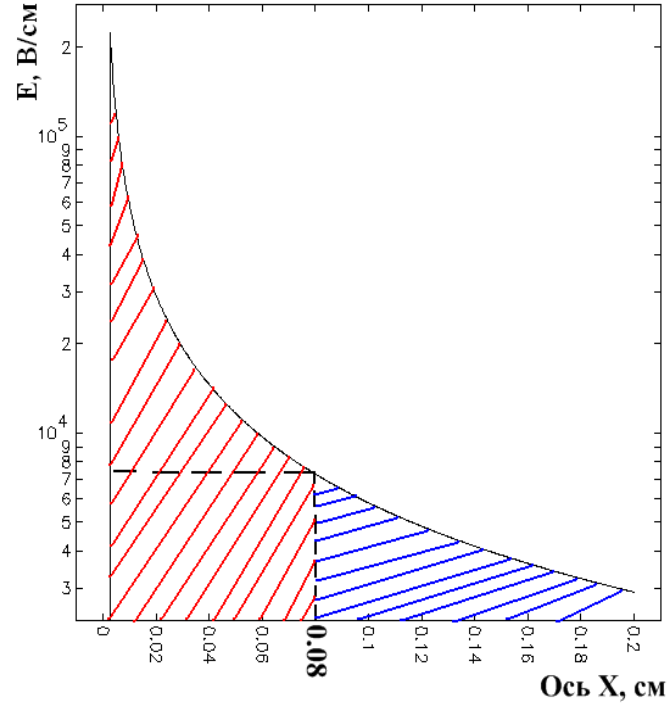
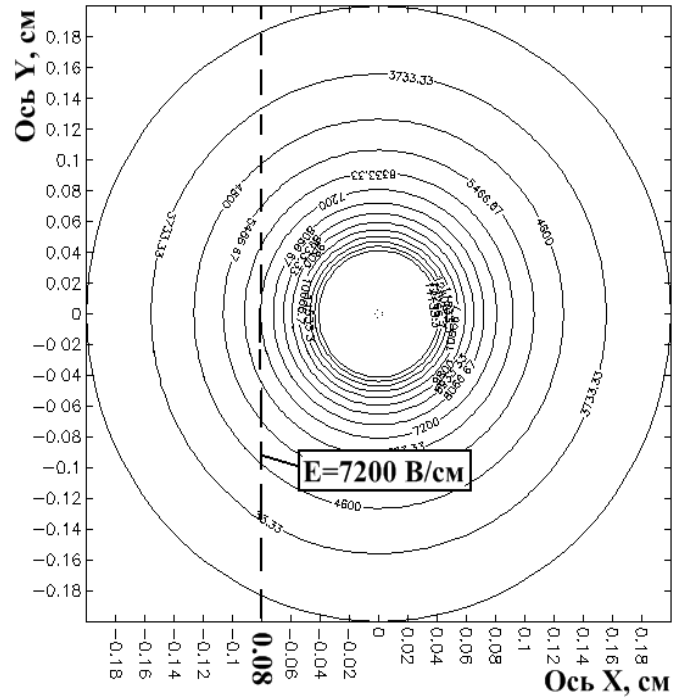
- Malter aging safe detector
- High signal amplitude due to LSM
- High angle resolution



- Low efficiency
- Small efficiency plateau



# Probable detector structure



# Working gas mixtures with CF3I

## PRO - CONS

**10 times high pulses amplitude good for operation at high background**

**Not good for CSC because of low efficiency**

**Safe from Malter aging effect, because insensitive to cathode electrons emmision**

**Short efficiency plateau ~ 100 V**

**Limited amplification zone in vicinity of the anode wire implies good space resolution**

## Outlook

- **CF3I concentration < 0.1% with adding methane (to increase plateau) have to be studied**
- **Searching for new gas components have to be continued**

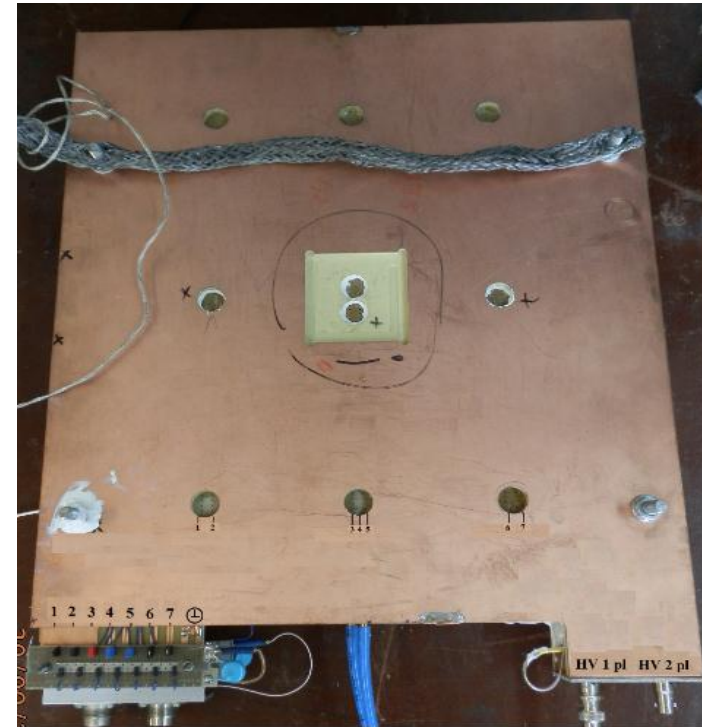


## II. CF3I gas mixtures study with CSC prototype

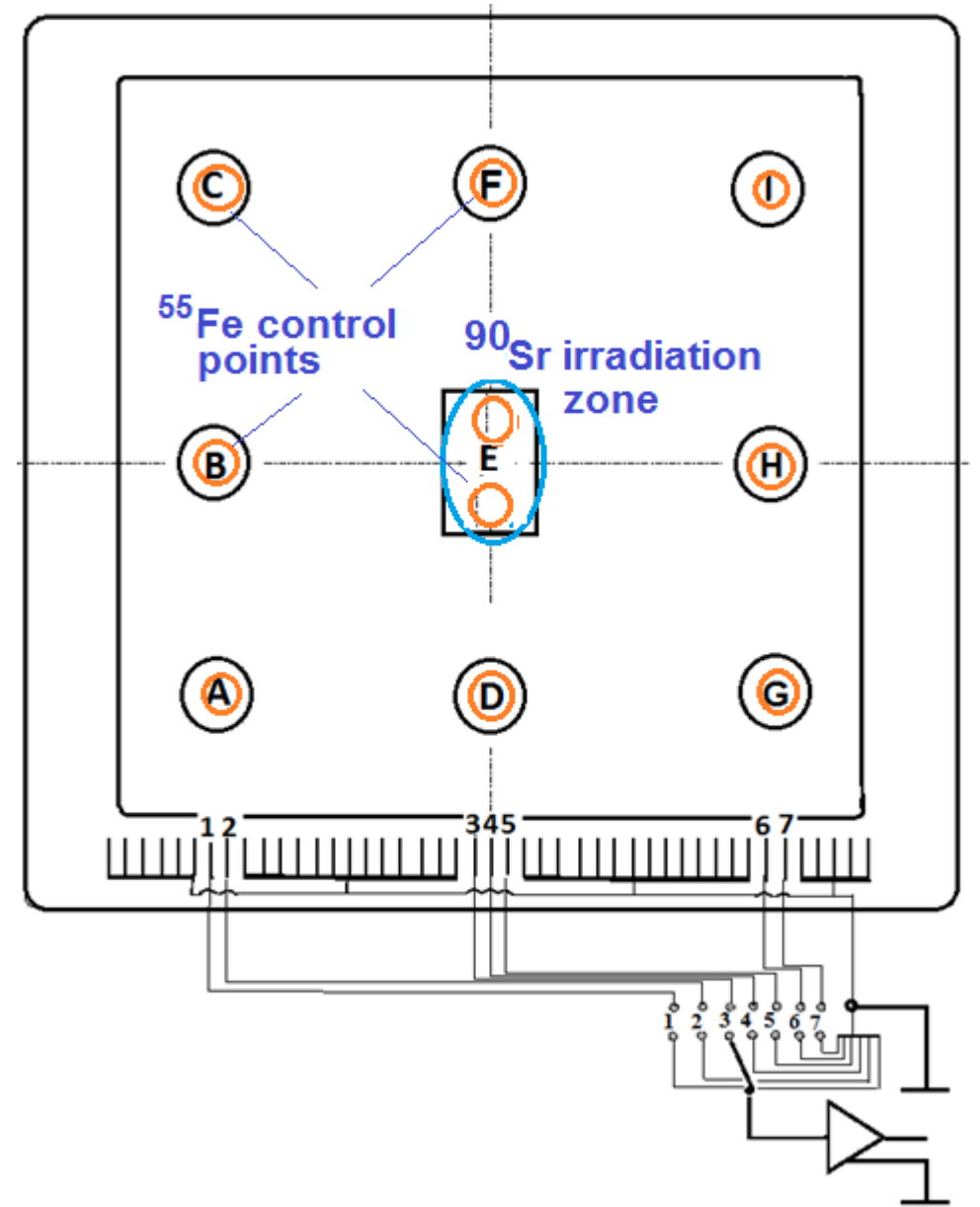
- 2 planes with 7 controlled anode wires in each one
- 50  $\mu\text{m}$  gold-coated anode wire
- 300 x 300 mm<sup>2</sup> sensitive area
- S = 3 mm
- L = 4.5 mm
- Identical geometry, construction materials to CSC

**BUT**

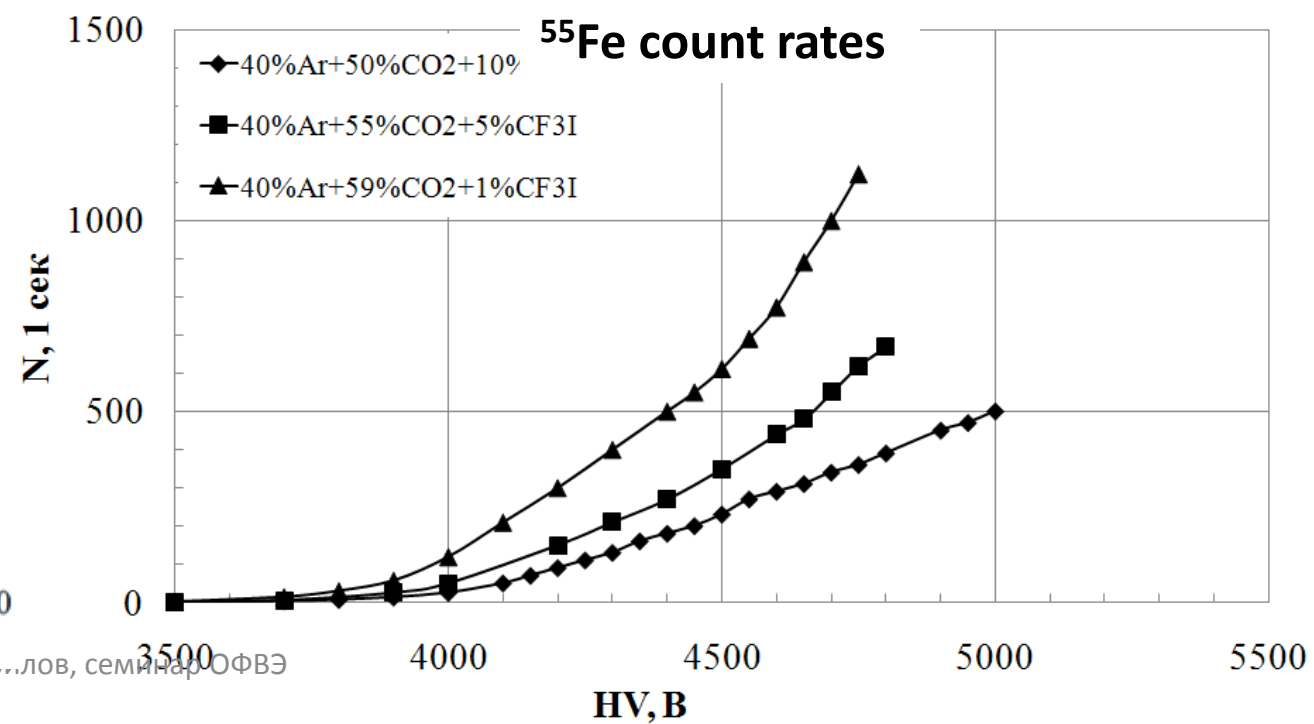
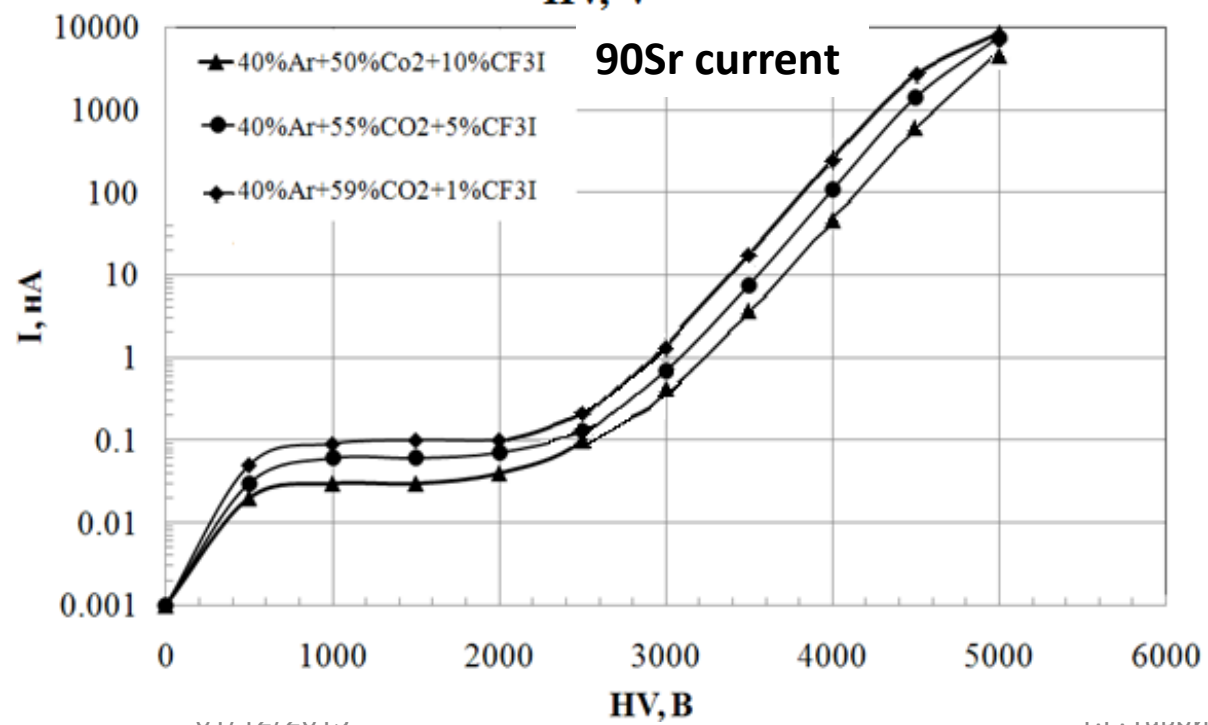
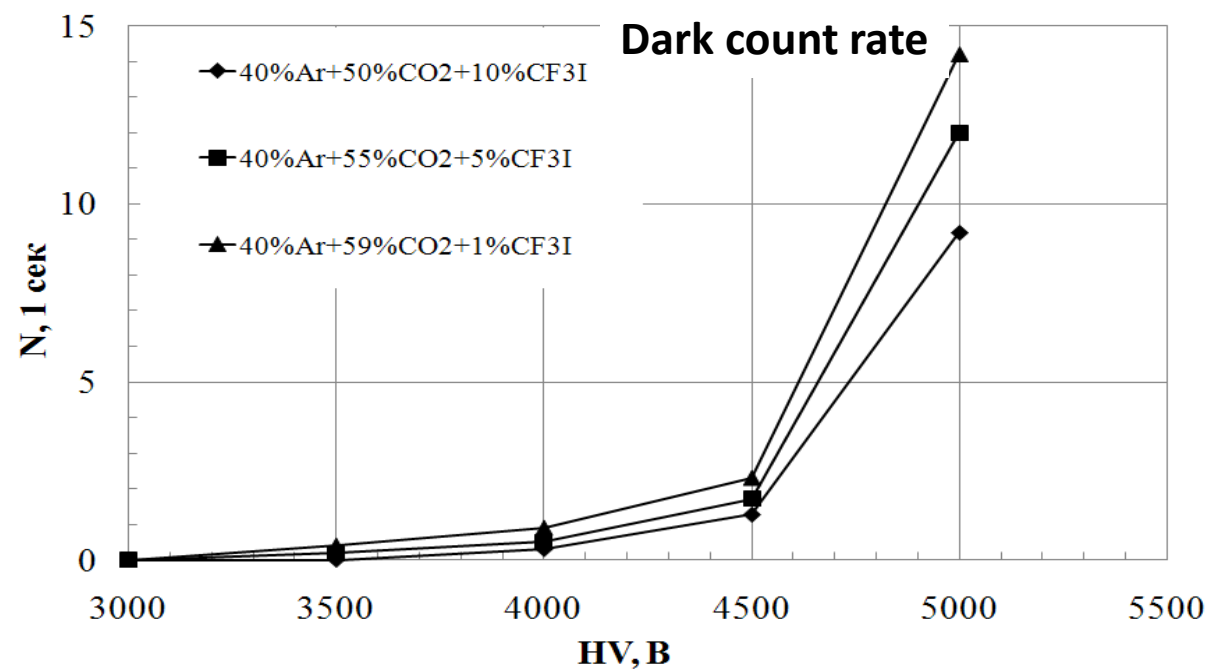
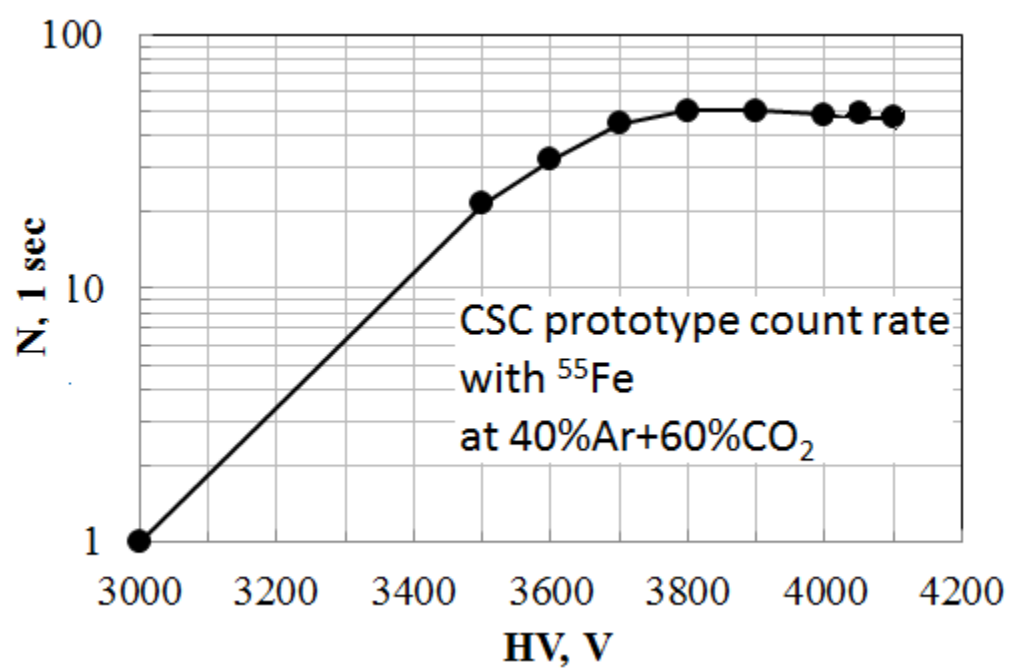
- \* No strips, readout from anode wires only
- \* - HV applied to the cathode



# CSC test prototype at ATS in PNPI

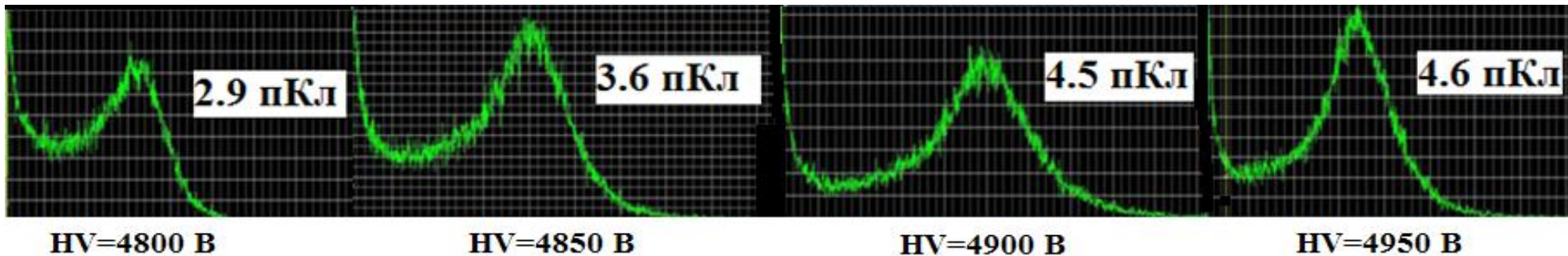




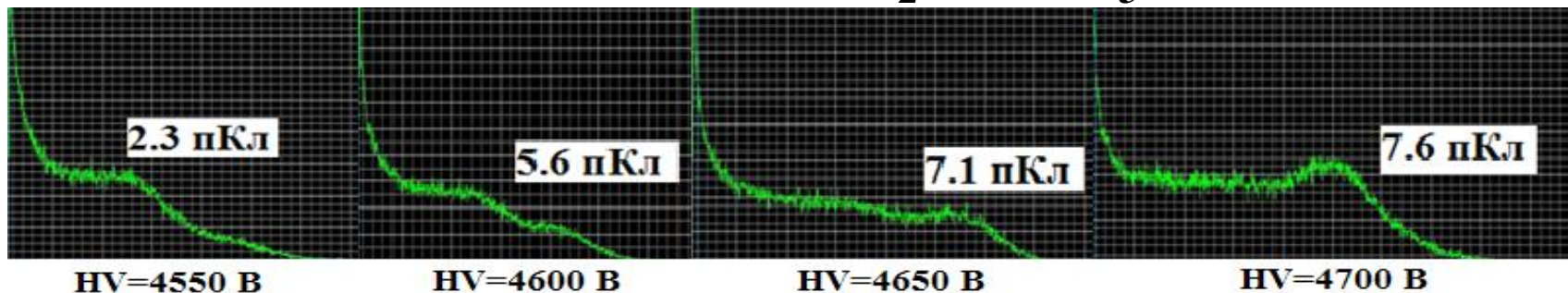


# Амплитудные спектры от источника рентгеновских фотонов $^{55}\text{Fe}$

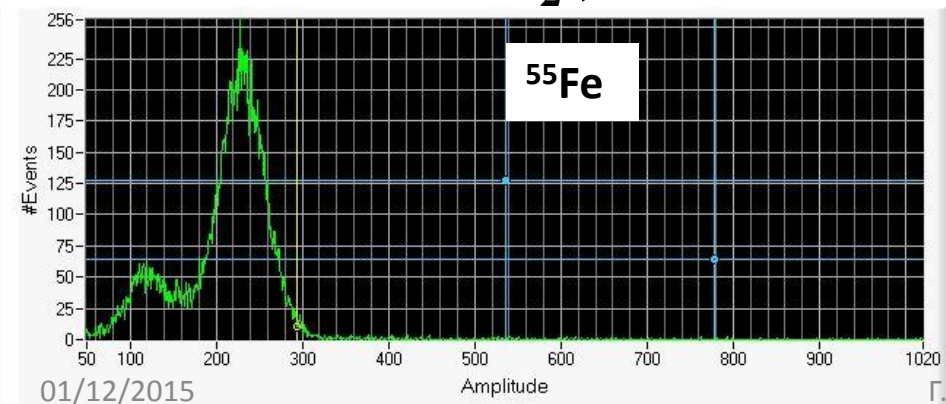
40%Ar+50%CO<sub>2</sub>+10%CF<sub>3</sub>I



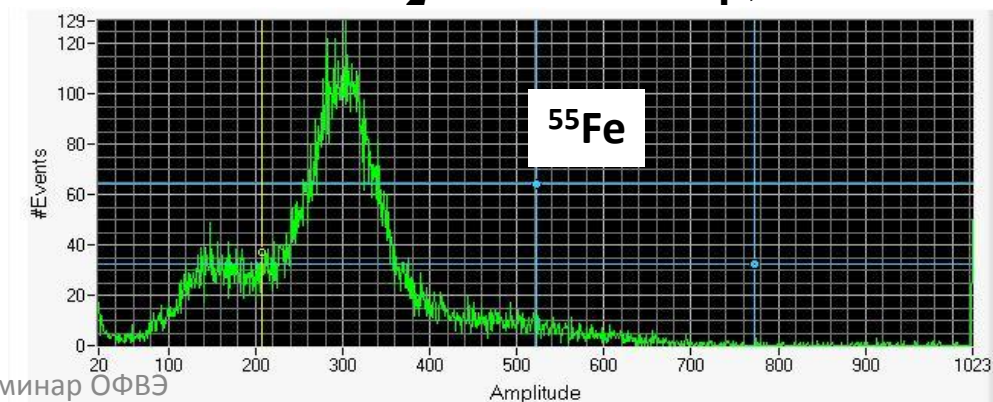
40%Ar+59%CO<sub>2</sub>+1%CF<sub>3</sub>I



40%Ar+60%CO<sub>2</sub>, HV=3650 В



40%Ar+50%CO<sub>2</sub> +10% CF<sub>4</sub>, HV=4100 В



# Возможный кандидат $C(CH_3)_4$ - n - пентан

**GWP  $C(CH_3)_4$  < 15 за 100 лет**

Пентаны — насыщенные ациклические углеводороды класса алканов. Имеют пять атомов углерода в молекуле. Изопентан обладает наркотическим действием. Класс опасности четвёртый

Формула:  $C_5H_{12}$

Температура кипения:  $36,1^\circ C$

Название ИЮПАК: Pentane

Плотность:  $626,00 \text{ кг/м}^3$

Молярная масса:  $72,15 \text{ г/моль}$

Температура плавления:  $-129,8^\circ C$

Классификация: Алканы



# Simulation Studies of Characteristics and Performance of small-strip, Thin Gap Chambers for the ATLAS New Small Wheel Muon Detector Upgrade

## Muon Detector Upgrade

J. Chapman<sup>a</sup>, T. Dai<sup>a</sup>, E. Diehl<sup>a</sup>, H. Feng<sup>a</sup>, L. Guan<sup>b,a,\*</sup>, G. Mikenberg<sup>c</sup>, V. Smakhtin<sup>c</sup>, J. Yu<sup>a</sup>, B. Zhou<sup>a</sup>, J. Zhu<sup>a</sup>, Z. Zhao<sup>b</sup>

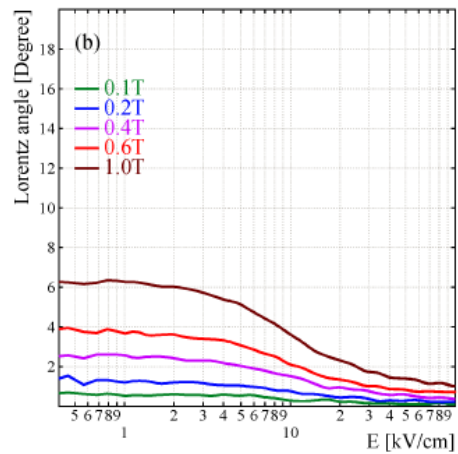
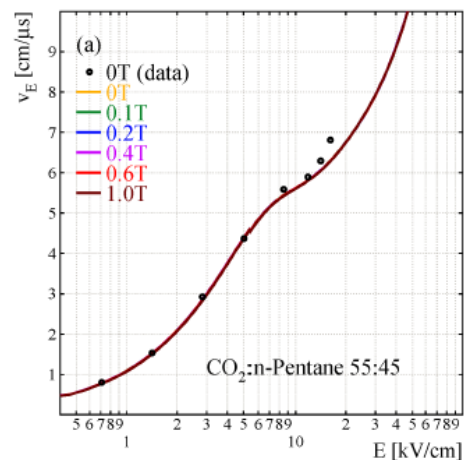
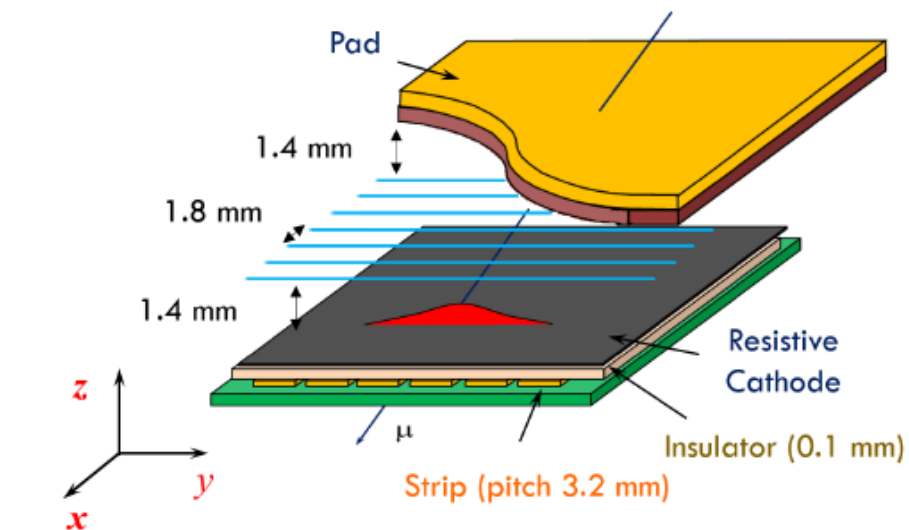


Figure 8: The electron drift velocity (a) and Lorentz angle (b) as a function of the electric field in CO<sub>2</sub>:n-pentane (55:45) for several values of an orthogonal magnetic field.

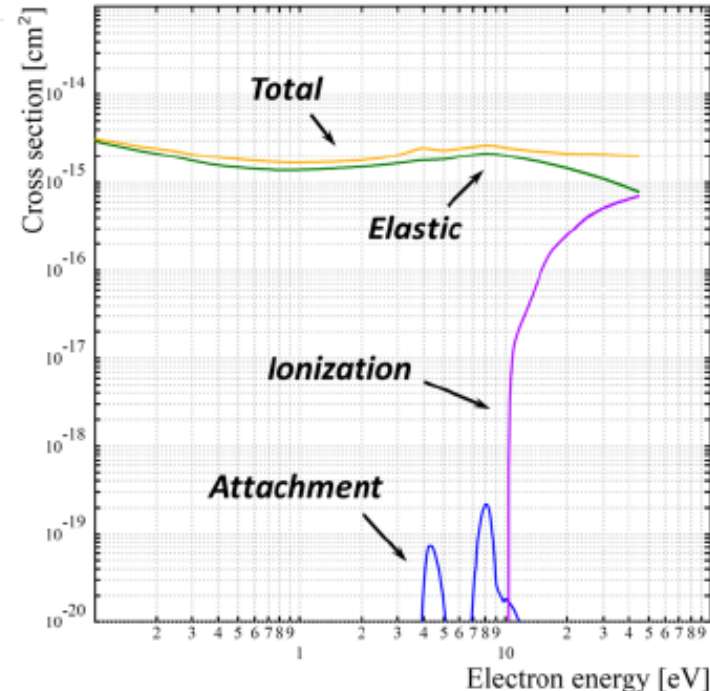
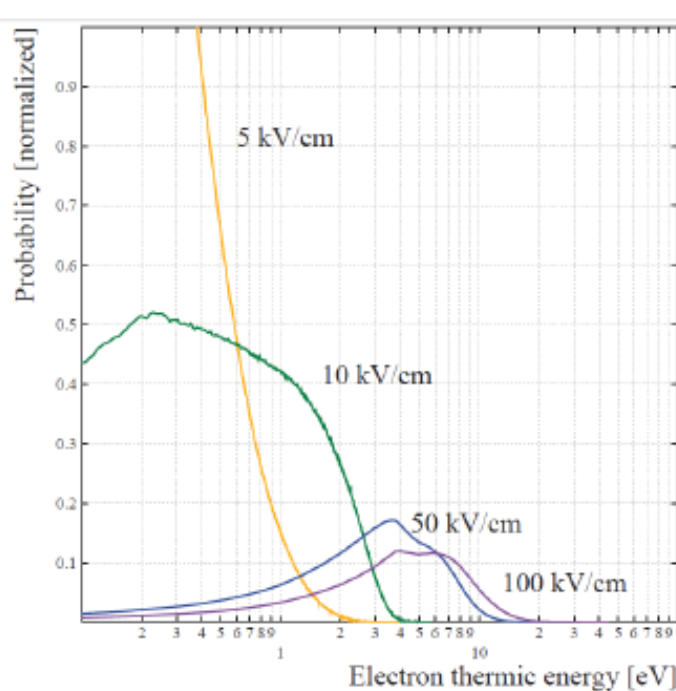
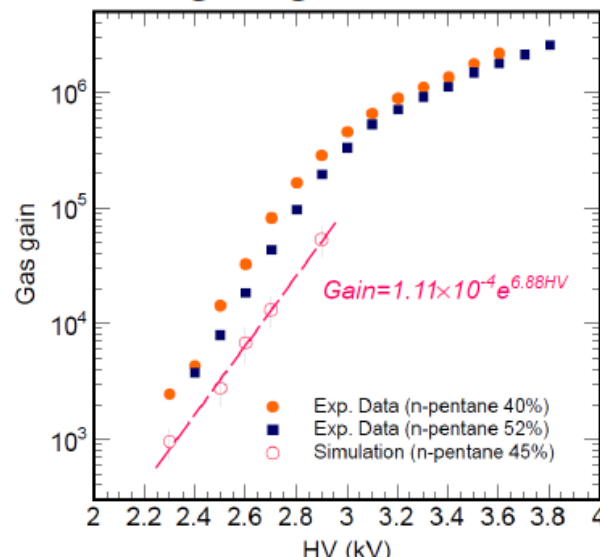
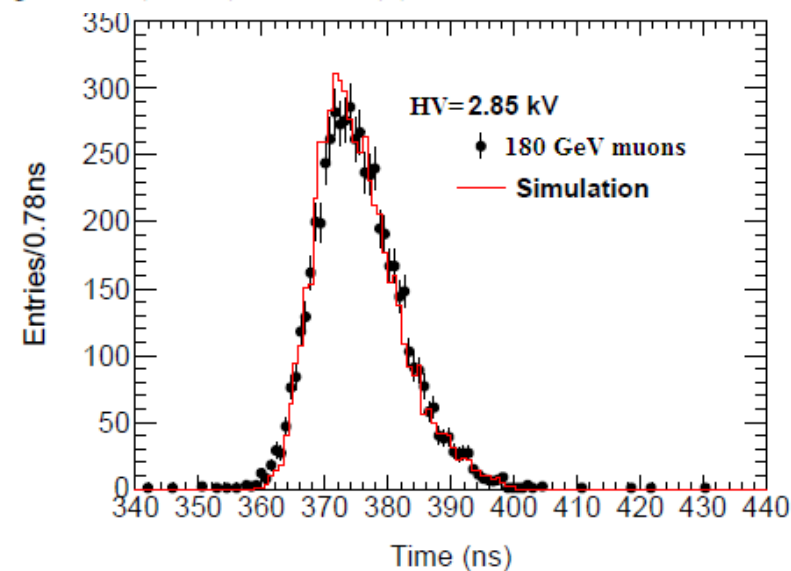


Figure 5: Electron thermal energy distributions in an electric field (a) and various cross-sections for electrons interacting with gas molecules in a CO<sub>2</sub>:n-pentane (55:45) mixture (b).



Gas gain as a function of the high voltage.

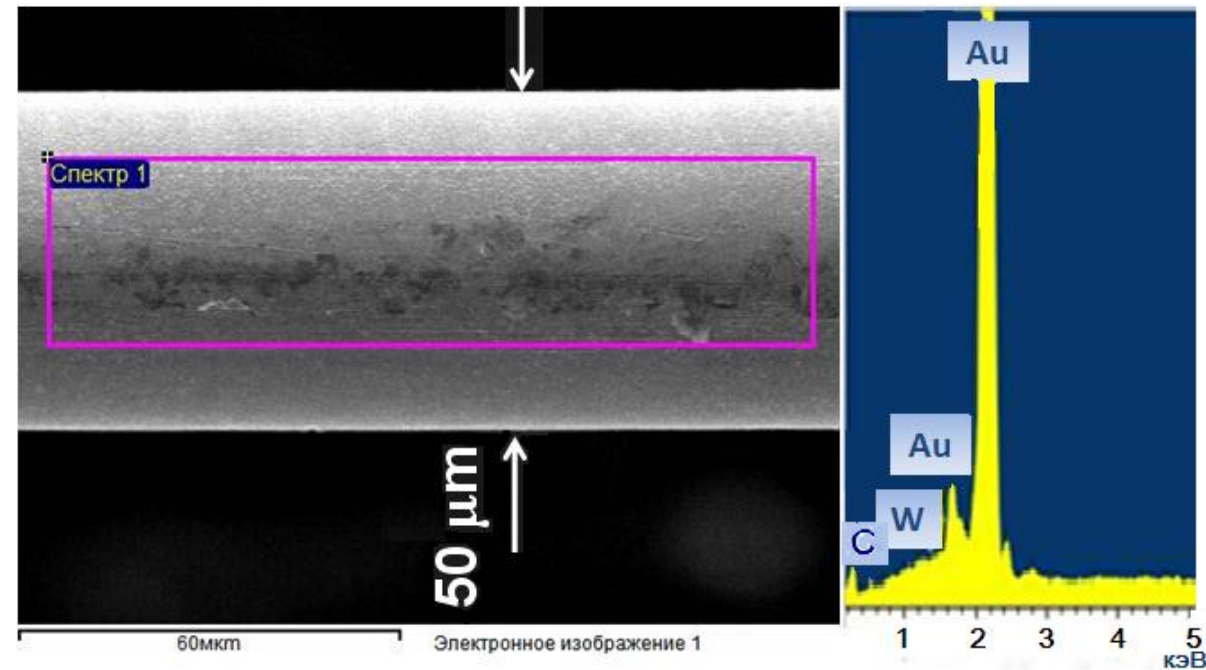
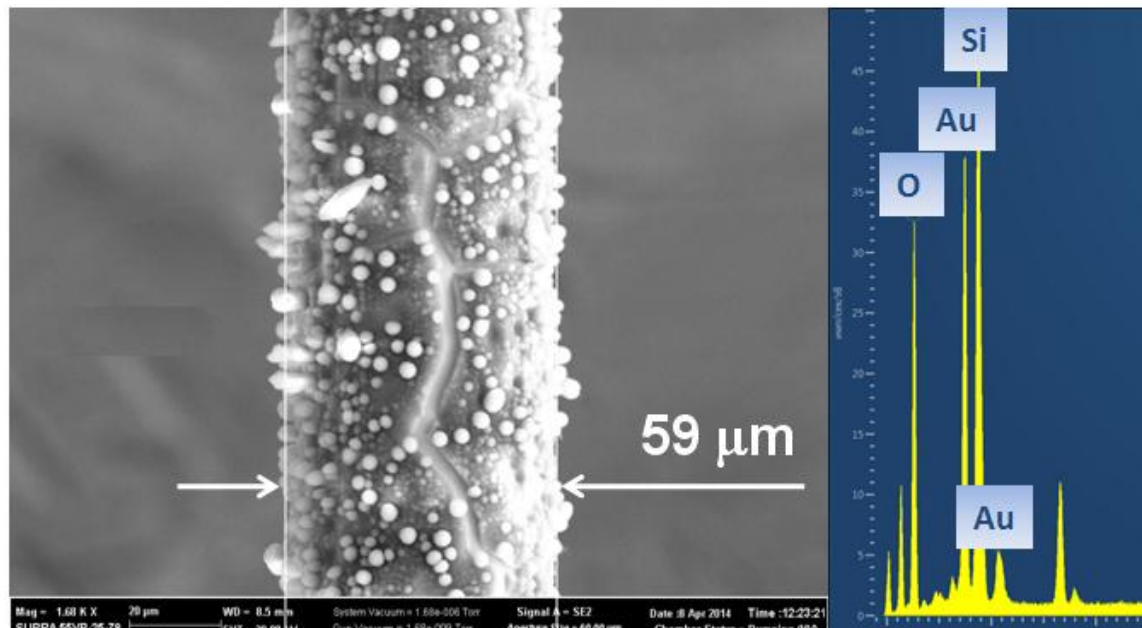




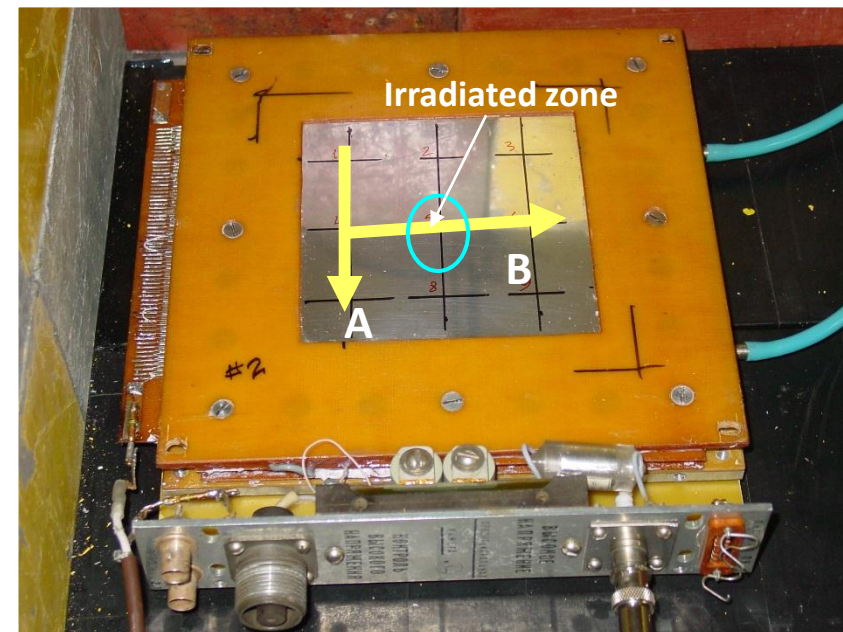
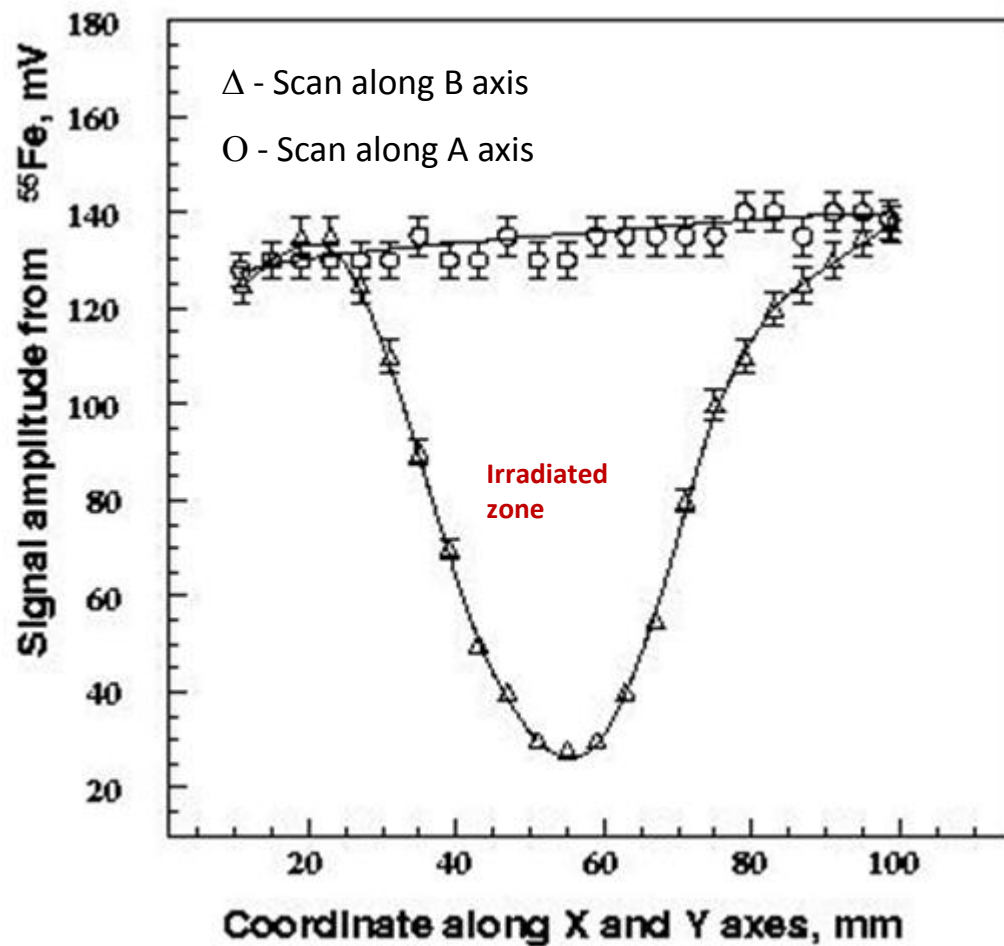




### III. Si aging recovering method



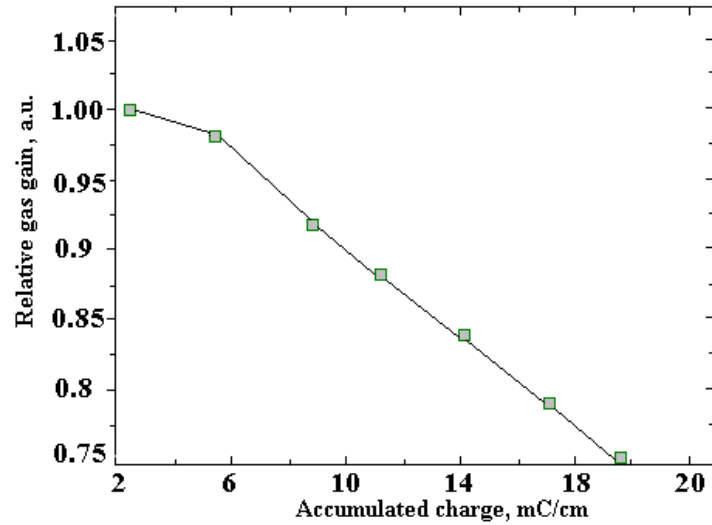
# Magnet Chambers (HERMES, DESY ) ageing studies



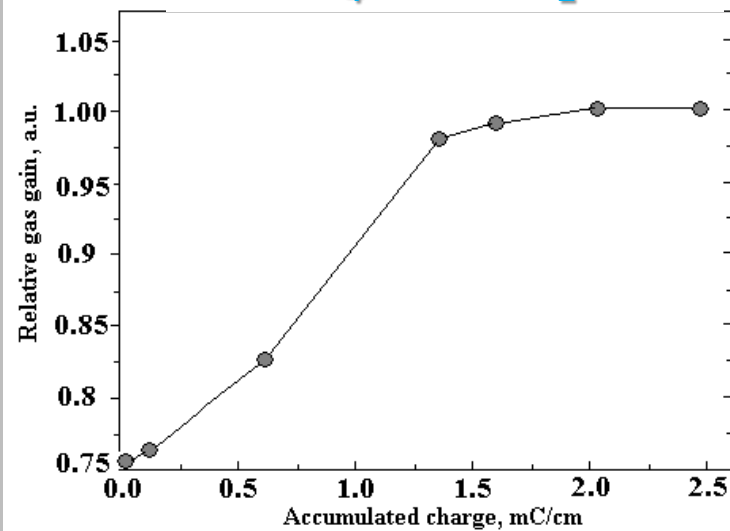
- Gas gain in the irradiated zone drops 4 times at  $Q_{\text{anode}} = 32 \text{ mC/cm}$
- Out of the irradiated zone the gas gain is constant
- Anode wire 30 mcm

# Cleaning of Si, SiO<sub>2</sub> deposits from the anode wire with 80%CF<sub>4</sub>+20%CO<sub>2</sub>

65%Ar+30%CO<sub>2</sub>+5%CF<sub>4</sub>



80%CF<sub>4</sub>+20%CO<sub>2</sub>



☀ Training with 80%CF<sub>4</sub>+20%CO<sub>2</sub> gas mixture at inversed HV = -3400 ÷ -3600 V

☀ Aged point irradiated with a <sup>55</sup>Fe X-ray source (6 keV)

~2μm Si, SiO<sub>2</sub> cleaned per 4 h from the wire surface with accumulated charge **Q ~ 2 mC/cm !**

<sup>55</sup>Fe amplitude spectra

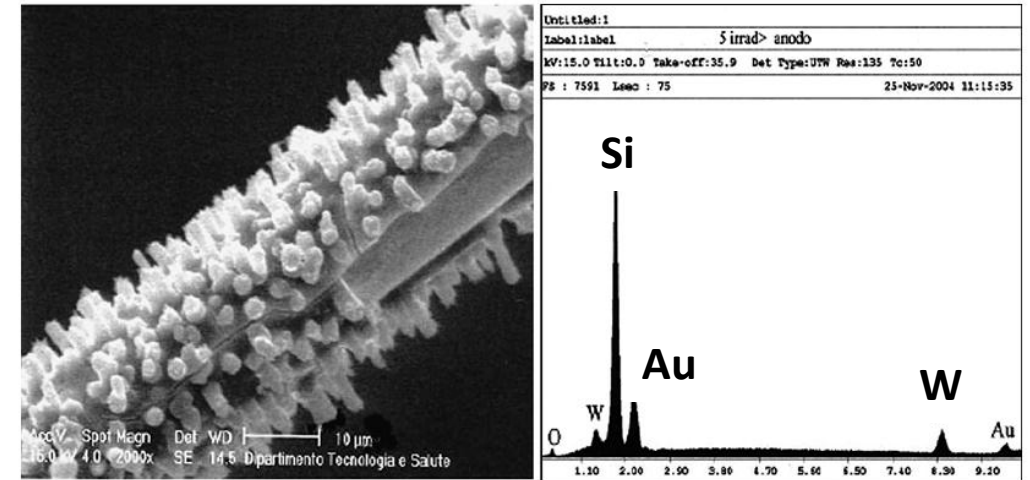
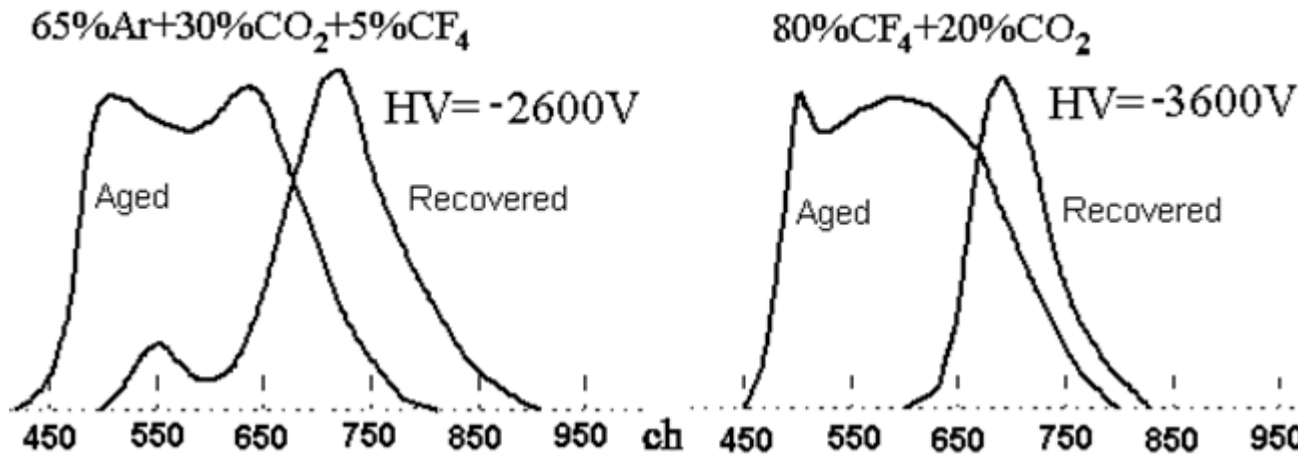


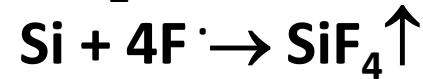
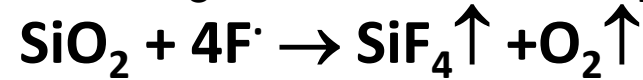
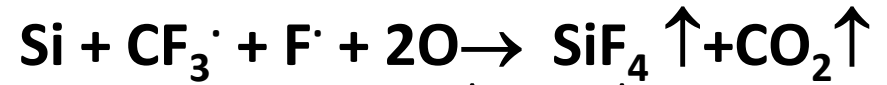
Fig. 11. SEM micrograph of the anode wire irradiated in the fifth test point (see Fig. 6) after accumulation of  $Q_{\text{anode}}^{\text{point 5}} = 32 \text{ mC/cm}$  and XEM spectra of the deposits on the surface.



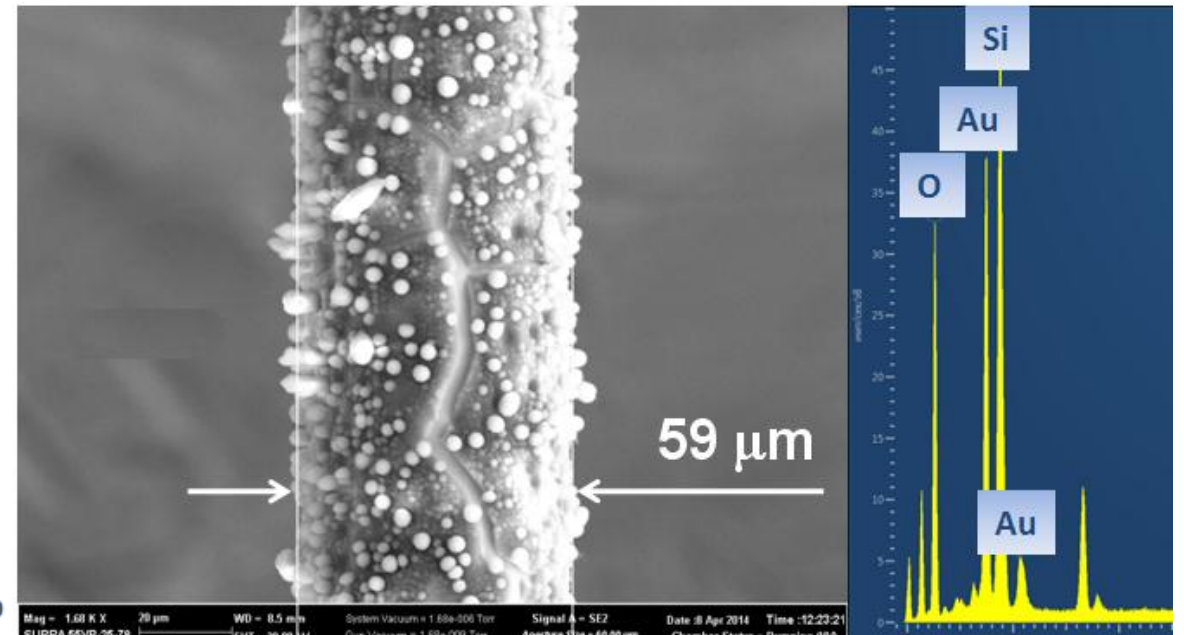
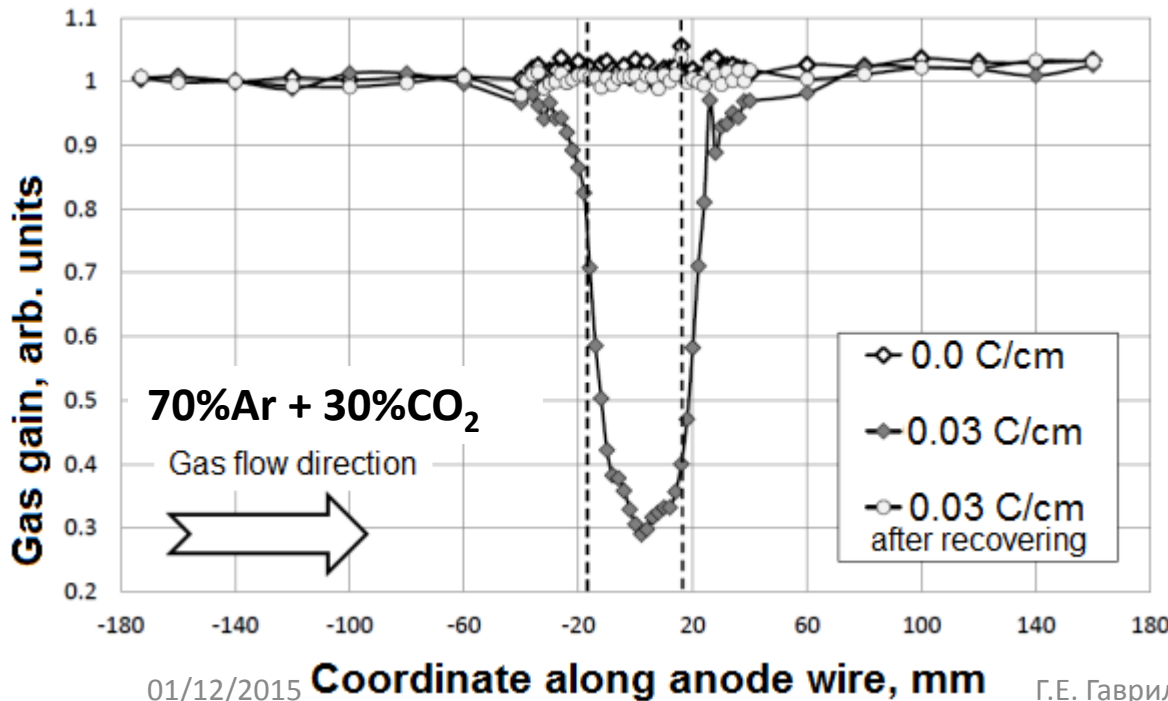
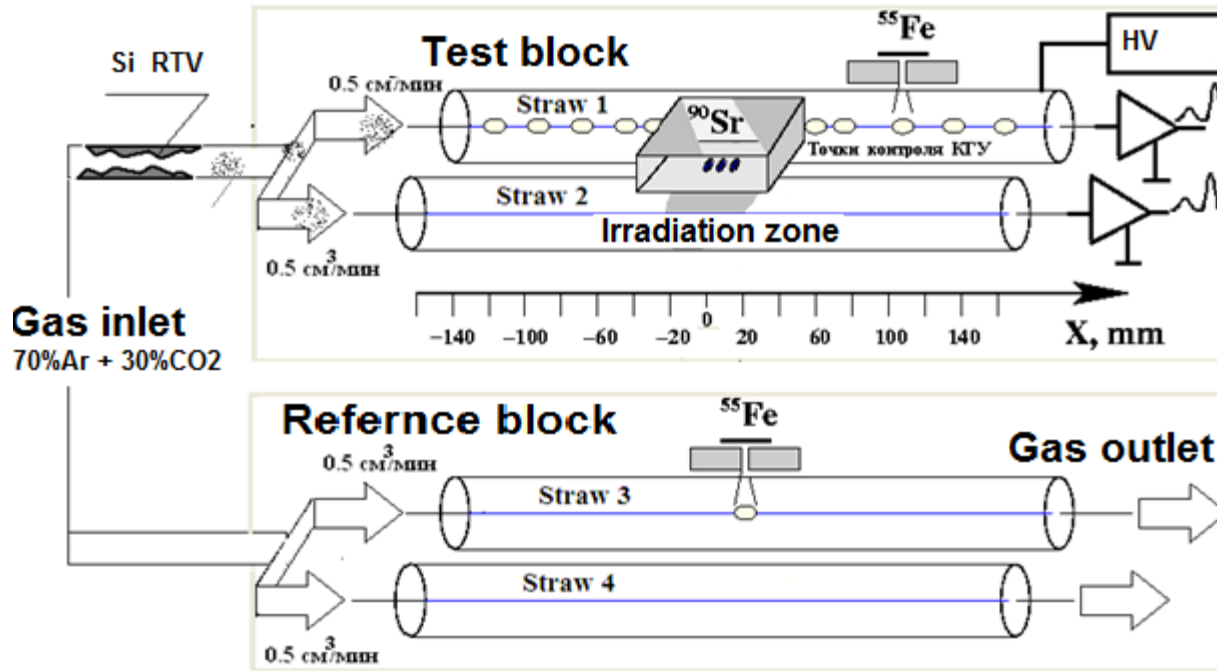
## Free radicals generation



## Etching of silicon deposits



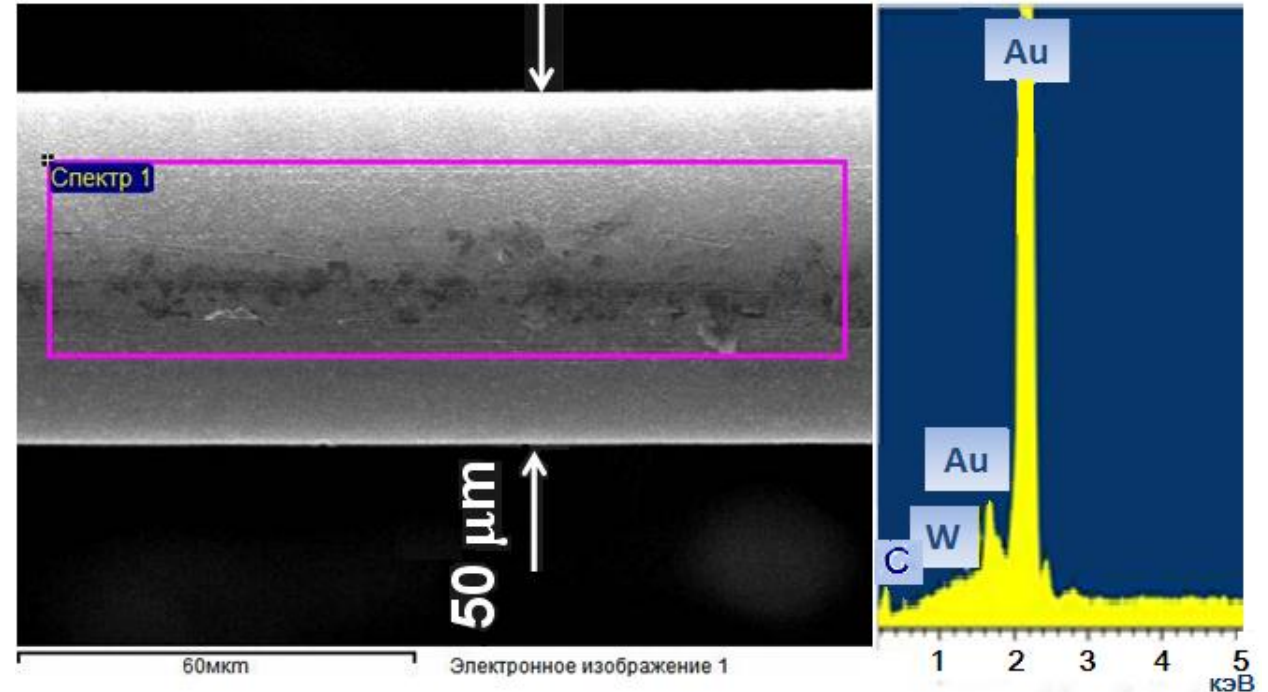
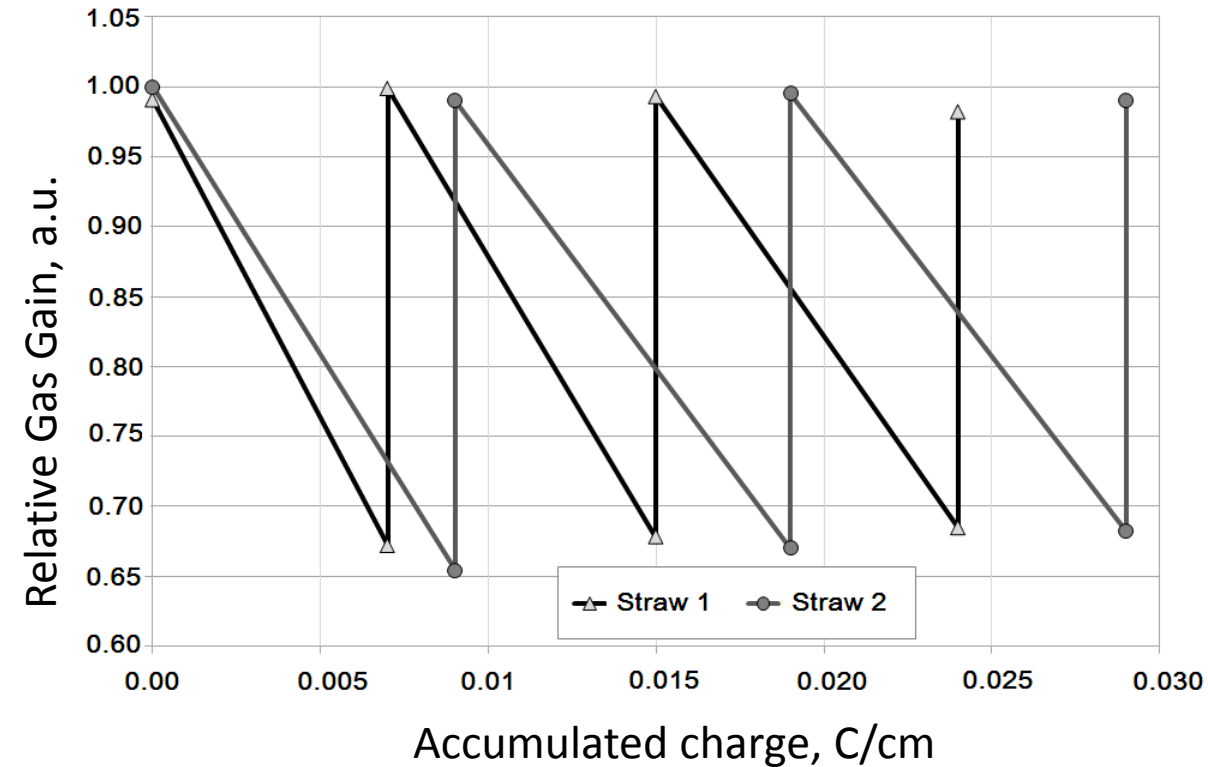
$$\frac{d\lambda}{\lambda} = 0.2 \frac{\Delta D}{D} \quad \frac{dG}{G} = \left[ \ln G + \frac{\lambda \cdot \ln 2}{2\pi\epsilon_0 \cdot \Delta V} \right] \frac{d\lambda}{\lambda} = 59\%$$



# Multiple cleaning of the anode wire with plasma chemistry glow discharge

Straw aging performed with a **60%Ar + 30%CO<sub>2</sub> + 10%CF<sub>4</sub>** working gas mixture and limited by  $dG/G \sim 33\%$

Straw recovering with a **80%CF<sub>4</sub> + 20%CO<sub>2</sub>** gas mixture



**Multiple cleaning with plasma etching of the silicon compounds on the wire surface leads to complete restoration of the detector performance and few times increases the lifetime.**



# Methods for recovery of aged CSC

## Chamber aging and eco-friendly gas mixtures

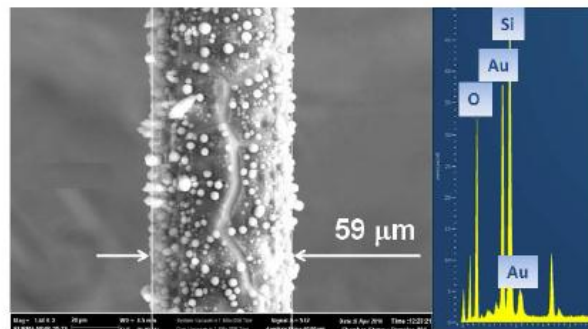
Armando Lanaro (UW)

CSC Coll. Meeting CMS week

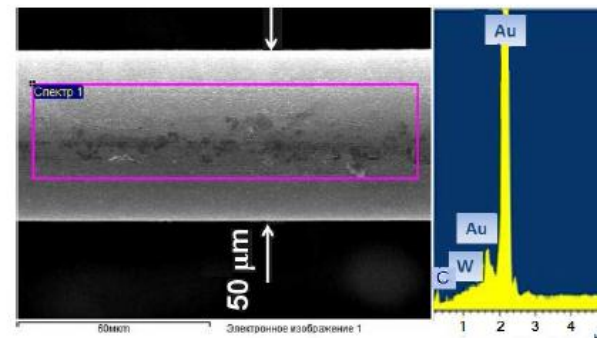
Oct 20, 2015

Many thanks to all colleagues providing material and inputs

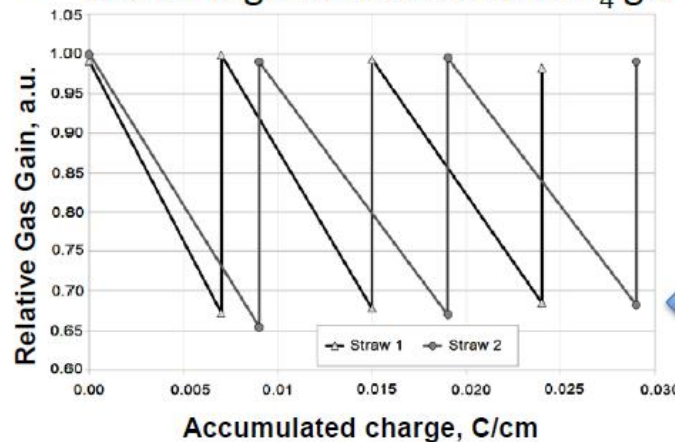
- PNPI collaborators are actively studying this subject (see last week presentation <https://indico.cern.ch/event/454874/> by G. Gavrillov )



**Cleaning of Si, SiO<sub>2</sub> deposits from the anode wire with 80%CF<sub>4</sub>+20%CO<sub>2</sub>**



- Use of high concentration CF<sub>4</sub> gas mixtures (glow discharge) produces etching of Si



- Aged point irradiated with a <sup>55</sup>Fe X-ray source (6 keV)
  - Perform recovery of aged CSC in equipped lab (B904)

Multiple cleaning leads to complete restoration of detector performance



# Strategy for future

- Preliminary targeting aging tests in PNPI. At ATS with intensive  $^{90}\text{Sr}$  local irradiation will be performed studies of alternative gas mixtures.



- Then at GIF will be tested compact CSC prototypes with recirculation gas system flushed by the tested at ATS gas mixture.



# Заключение

1. Все составы исследованных газовых смесей демонстрируют выход на плато счетной характеристики в режиме ограниченного стримера. Это продемонстрировано на пропорциональном счётчике straw при работе с прототипом CSC.
2. Большая амплитуда сигнала в области плато – в 4-10 раз большая, чем в обычной пропорциональной моде – позволяет рассмотреть возможность создания нового типа детектора, работающего в режиме ограниченного стримера.
3. Наличие зоны сильного прилипания электронов вблизи катодной плоскости делает детектор, работающий с рабочей газовой смесью содержащей  $CF_3I$ , безопасным для эффектов вторичной эмиссии электронов с катода, которые возникают в результате старения при длительном облучении.
4. Но. Применение подобных смесей для камер CSC невозможно из-за их низкой эффективности регистрации и необходимости подавать значительно более высокое напряжение.
5. Необходимо изучить возможности разработки новых рабочих газовых смесей с использованием других компонент

# IV Planning

1. Aging test of straw (50  $\mu\text{m}$  anode wire) at ATS with the 5% gas mixture.
2. Aging test of the CSC prototype (50  $\mu\text{m}$  anode wire) at ATS with 5% gas mixture.

## & Outlook

- Reducing of  $\text{CF}_4$  content in gas mixture up to 1-2%



This makes shorter the efficiency plateau

To compensate an X component have to be added. It can be  $\text{CH}_4$ ,  $\text{C}_2\text{H}_4$  and e t c.

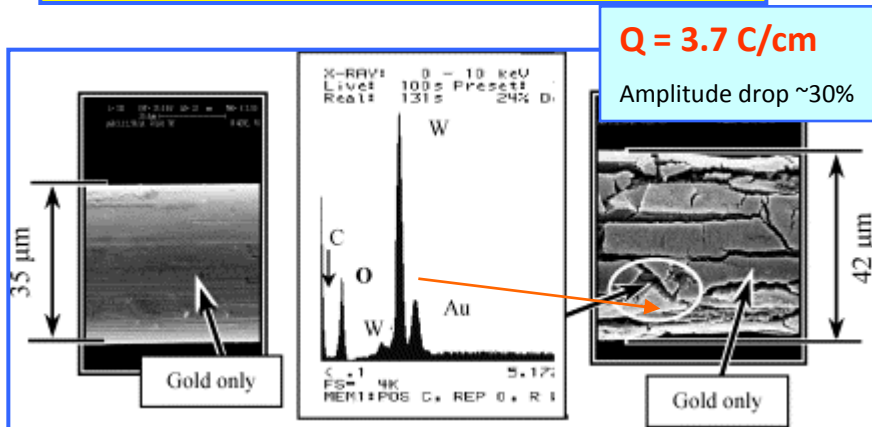
- Reducing the  $\text{CF}_3\text{I}$  content in the gas mixture up to 0.1 %
- Looking for new gas components

# How to cure ageing?

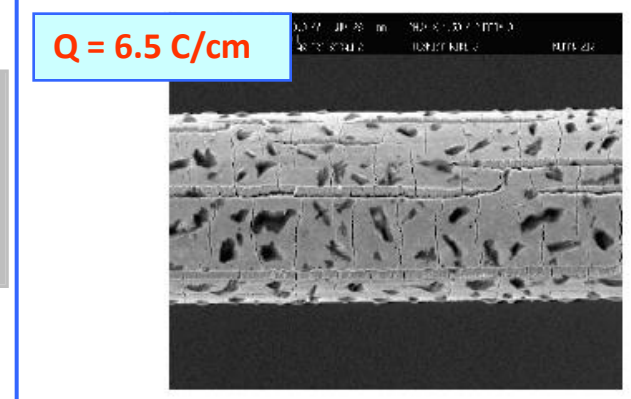
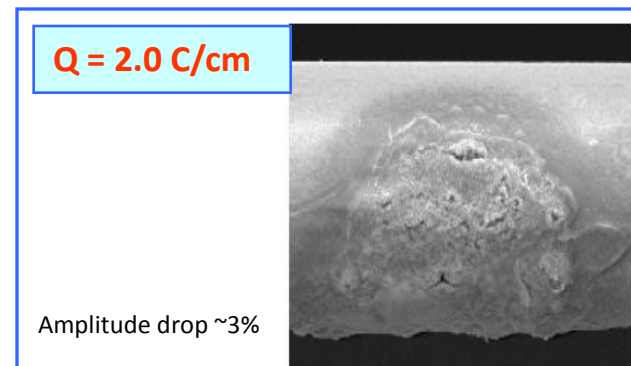
- R. Openshaw, R.S. Henderson et al IEEE Trans. Nucl. Sci NS-34, 528 (1987) - **training with 80%CF<sub>4</sub>+20%iC<sub>4</sub>H<sub>10</sub> glow discharge**
- T. Marshall NIMA 515 (2003), 50 – **“Zap” cleaning by quick current heating (C=1.5μF HV=3500V l = 190 cm)**
- M. Kollefrath, V. Paschhoff, M. Spiegel et al NIMA419 (1998), 451 – **reversed HV and glow discharge in Ar +O<sub>2</sub> (the current I=0.7÷23 μA/cm cleaned carbon)**
- A.M. Boyarski NIMA515(2003), 190 – **2h of training with He + iC<sub>4</sub>H<sub>10</sub>+ O<sub>2</sub> under <sup>55</sup>Fe irradiation cured the Malter effect (29nA/cm)**
- S. Belostotski, S. Frullani, G. Gavrilov... NIMA 591 (2008) 353–366 **4 hours training of HERMES Magnet chamber prototype with 80%CF<sub>4</sub>+20%CO<sub>2</sub> with <sup>55</sup>Fe irradiation and inversed HV cured anode (~2 mcm Si cleaned from the anode with 20nA/cm glow discharge current)**

# Ageing terms and faces

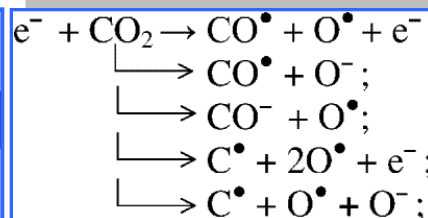
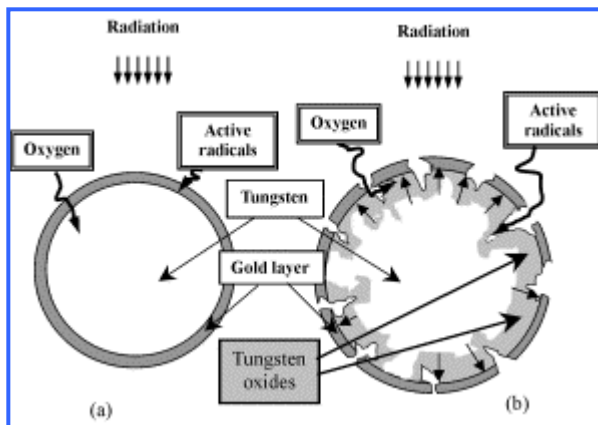
## Anode wire swelling effect



T.Ferguson et al. NIMA 478(2002) 254-258



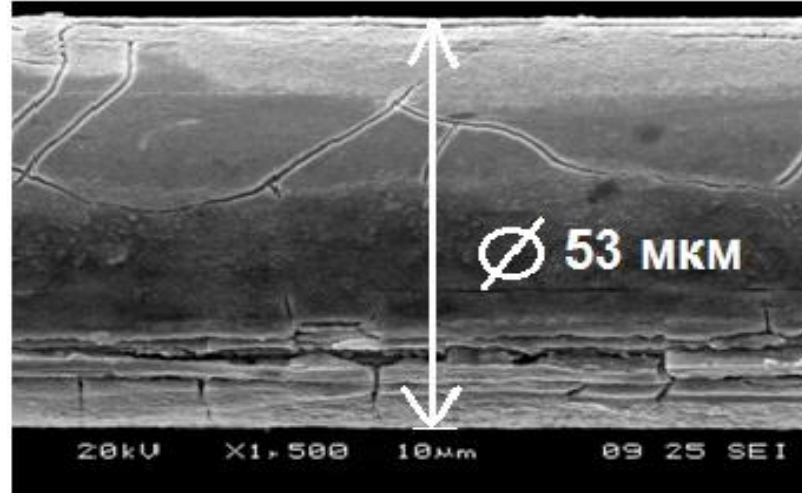
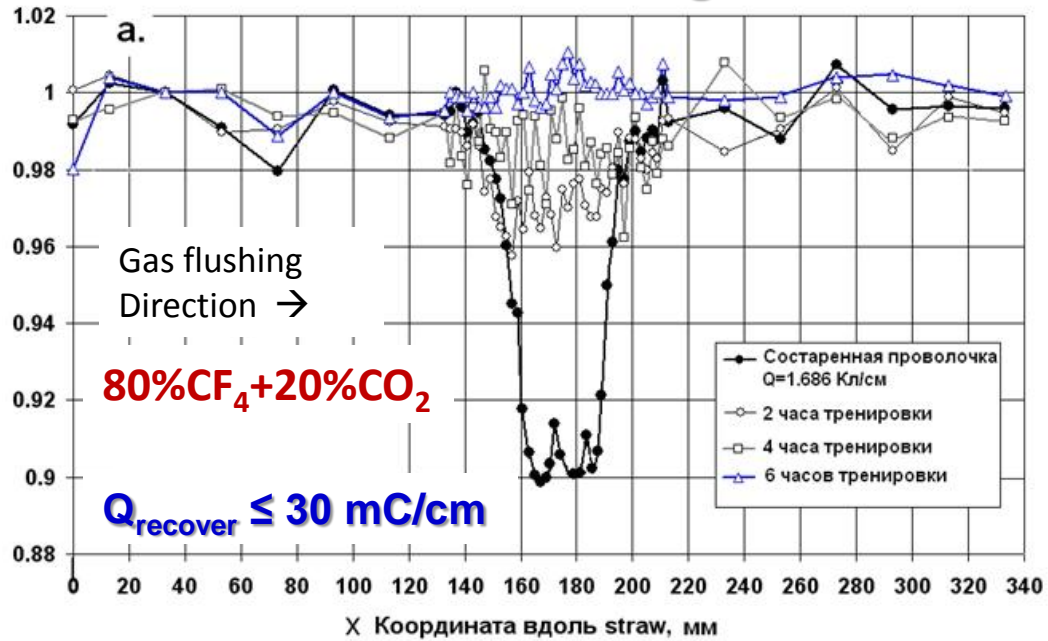
T.Akesson et al. NIMA 515(2003) 166-179



T. Ferguson, G. Gavrilov et al, Swelling phenomena in anode wires aging under a high accumulated dose, NIMA 515 (2003) 266–277

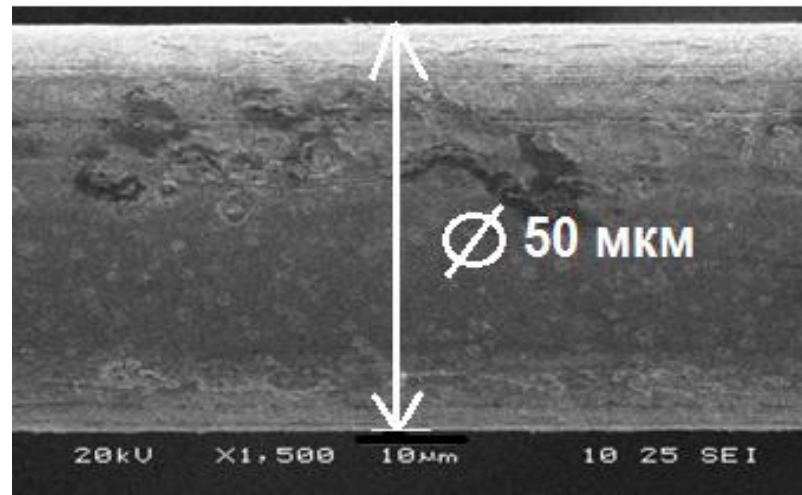
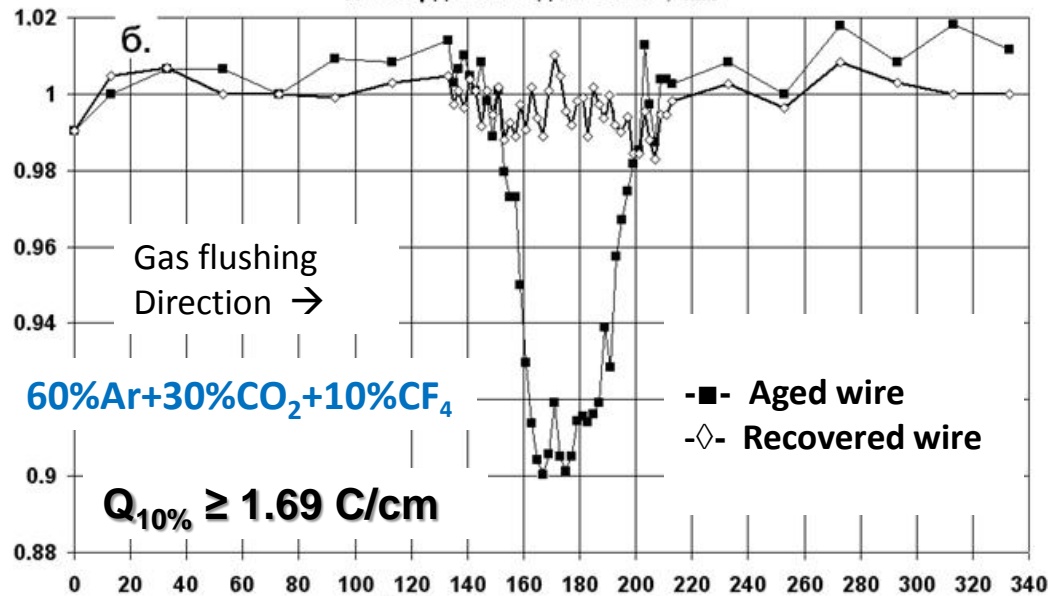


# Curing of the anode wire in the straw from swelling



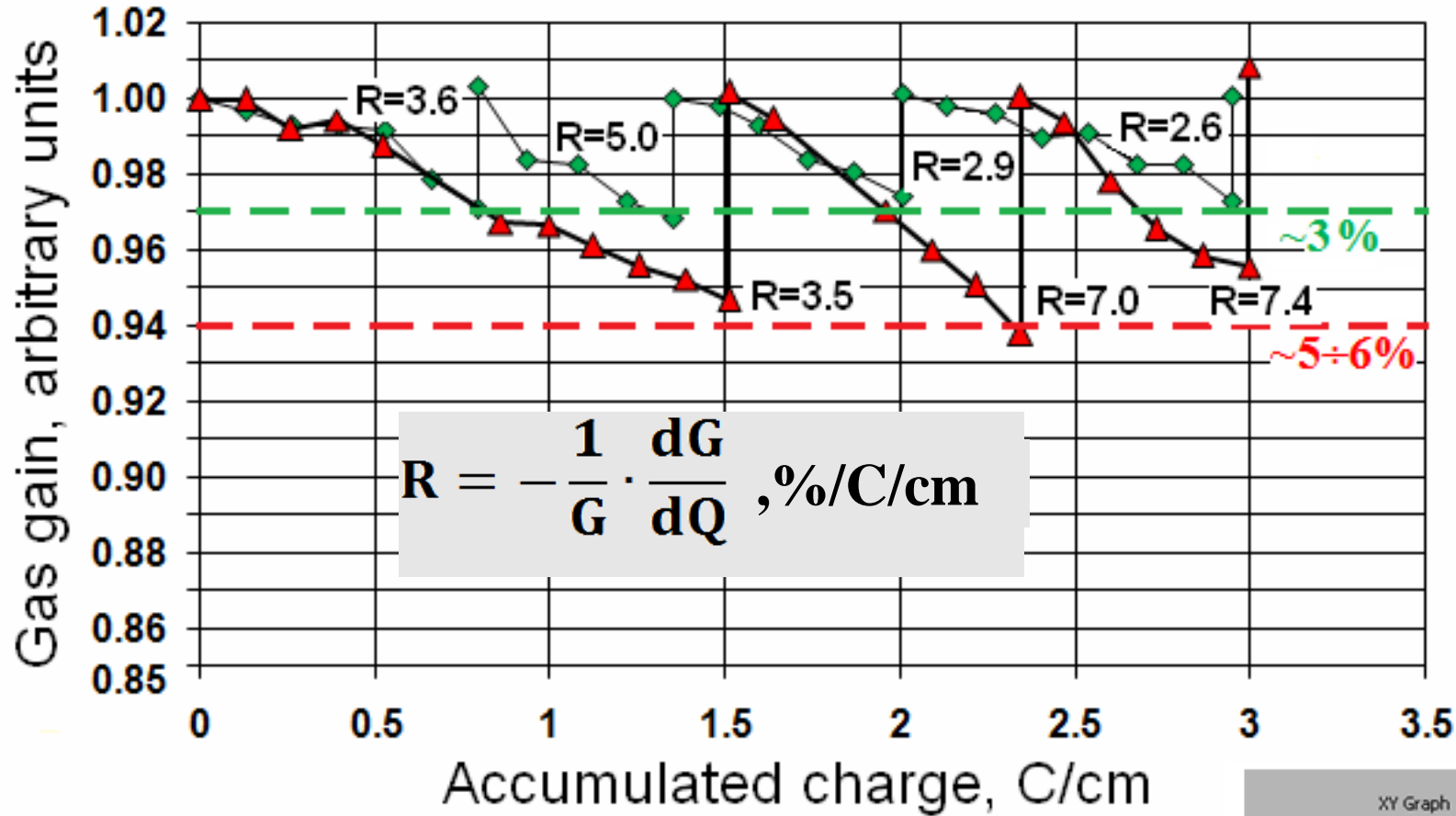
$$\frac{dG}{G} = 10\% \Rightarrow \frac{dG}{G} \leq 1\% \quad \downarrow$$

Relative gas gain, arbitrary units

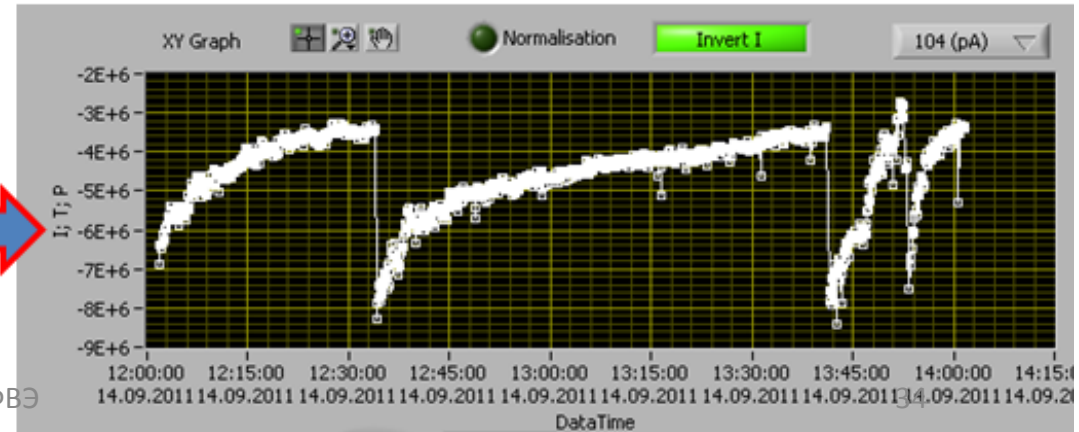


Backup

# Multiple recovery test results



**Currents changing during recovering**



Backup

G.E. Gavrilov, D.A.Aksenov, R.Conti et al., Using an 80%CF4+20%CO2 gas mixture to recover aged anode wires in proportional chambers, NIMA 694 (2012) 167–172

# PAST EXPERIENCE

DETECTOR	AGEING EFFECT	SOLUTION
<b>ZEUS Central Tr.</b> 83%Ar+12%CO <sub>2</sub> +5%C <sub>2</sub> H <sub>6</sub> +0.5%C <sub>2</sub> H <sub>5</sub> OH	<b>Malter effect (0.1 C/cm)</b>	<b>Addition of water 0.1%</b>
<b>H1 Central Jet Ch.</b> 50%Ar+50%C <sub>2</sub> H <sub>6</sub> +0.1%H <sub>2</sub> O	<b>Malter effect</b> <b>Anode ageing (0.01 C/cm in CJC2)</b>	<b>5 years with 0.1% water, then changed on 0.8% ethanol</b>
<b>D0 Drift Tube Ch</b> 90%Ar+4%CO <sub>2</sub> +6%CF <sub>4</sub>	<b>Anode ageing</b>	<b>Remove outgasing material and clean wires</b>
<b>CDF Central Tr.</b> 50%Ar+50%C <sub>2</sub> H <sub>6</sub> +1%C <sub>2</sub> H <sub>5</sub> OH	<b>Anode ageing (20 mC/cm)</b>	<b>Clean of gas system and gas filtering</b>

**Existing methods of ageing preventing are passive and only delay the process development**



