

Investigating protection layers

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Nikhef/Bonn LepCol meeting
October 19, 2020

Conductivity of 11 samples of different SixNy protection layers

- Received from IZM (Yevgen) via Uni Bonn on March 2020
- Layer thickness 2 μm
 - For GridPix we normally use 4 μm
- **Conductivity** calculated from IV measurements
- Process parameters varied
 - **SiH₄/N₂** concentration
 - Plasma **power**
 - Plasma **HF/LF**
- **N10** is the **reference**, same process as used for 2018 production

GelPak	SiH ₄ /N ₂	SiH ₄	N ₂	Plasma power	Frequency	Deposition time
Nikhef	(%)	(sccm)	(sccm)	(W)	(HF / LF)	(s)
N10	2	50	2450	250	HF	263
N11	1	25	2475	250	HF	797
N12	4	100	2400	250	HF	578
N13	2	50	2450	450	HF	539
N14	1	25	2475	450	HF	662
N15	4	100	2400	450	HF	190
N16	2	50	2450	900	HF	369
N17	1	25	2475	900	HF	699
N18	4	100	2400	900	HF	269
N19	2	50	2450	250	LF	601
N21	4	100	2400	250	LF	428

Experimental setup

- HP 4140B pA meter
- Mercury probe MDC MP-811
- Soap probe
- Nikhef MiniHV unit, tripping at 5000 nA
- HV control and current RO by PC with LabVIEW program
- Measurements due to **corona regulations** done at my **home**



Mercury probe

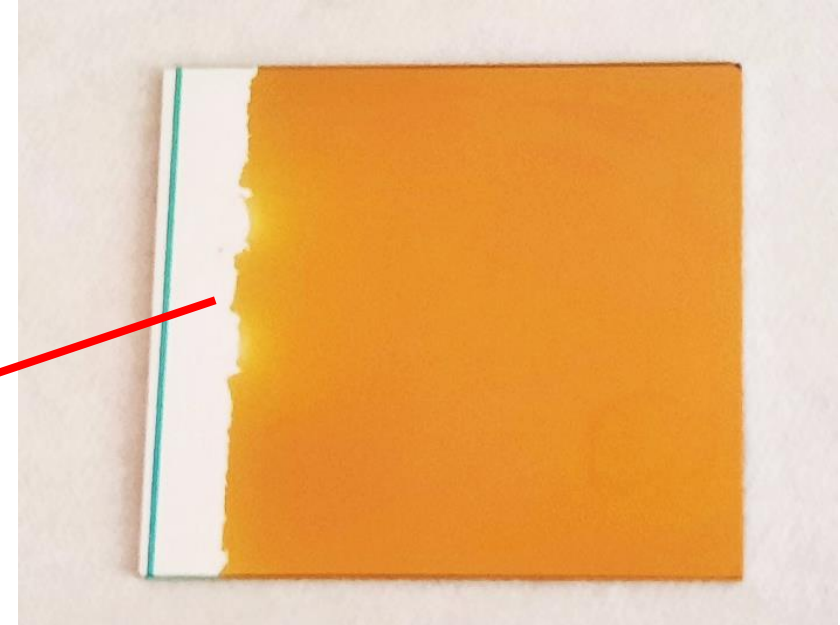


The samples

- Silicon substrate
- Metal layer
- Si_xN_y layer of $2\ \mu\text{m}$
- Back side insulated

- The samples were covered by a wax like layer often preventing good electrical contact
- Cleaned using alcohol

Return
contact



~ 3 cm

The probes

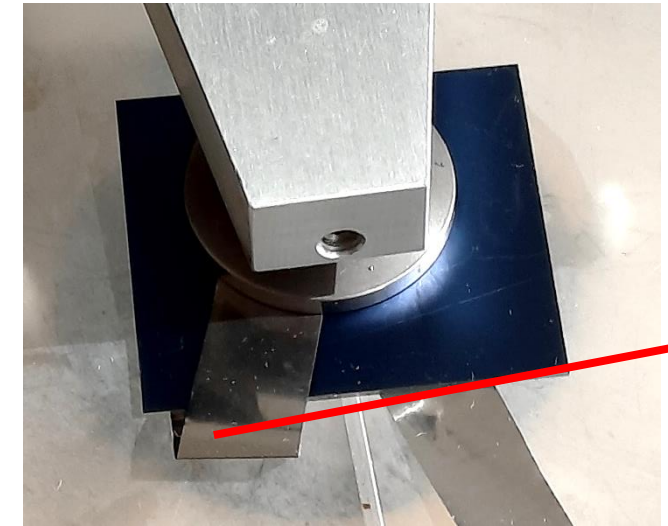
- Hg probe
 - Mercury suck by vacuum to make contact under the sample
 - Contact surface 18.9 mm^2
 - $10 \mu\text{m}$ SS contact foil needed
 - Sample is insulated at bottom side

- Soap water probe
 - 8 mm SS disk as HV electrode
 - $80 \mu\text{m}$ wire to miniHV
 - A drop of soap water under it
 - => contact surface 50.3 mm^2

Mercury contact
(Top view, seen through a glass plate)

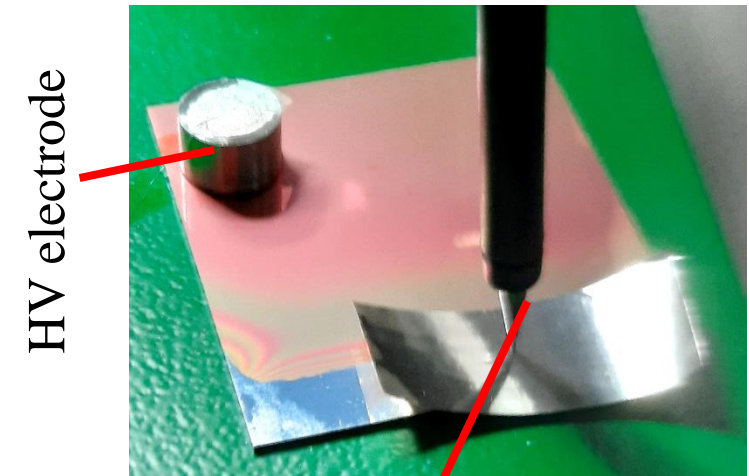


Sample under Hg probe



Contact strip

Sample with soap probe

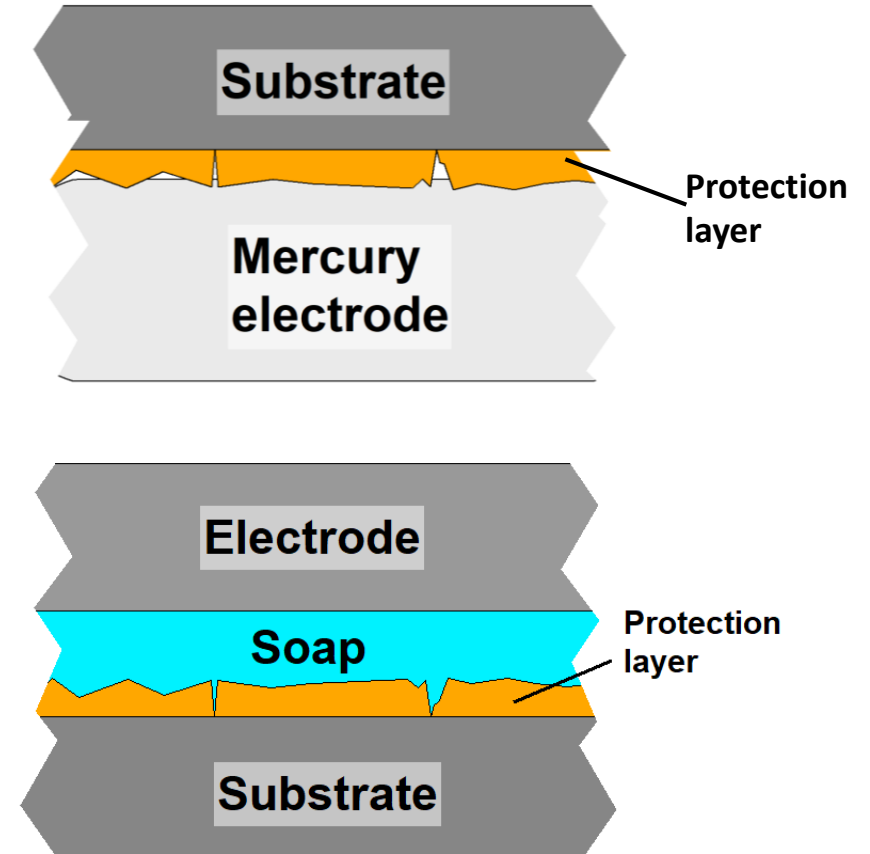


HV electrode

Return contact to
pA meter

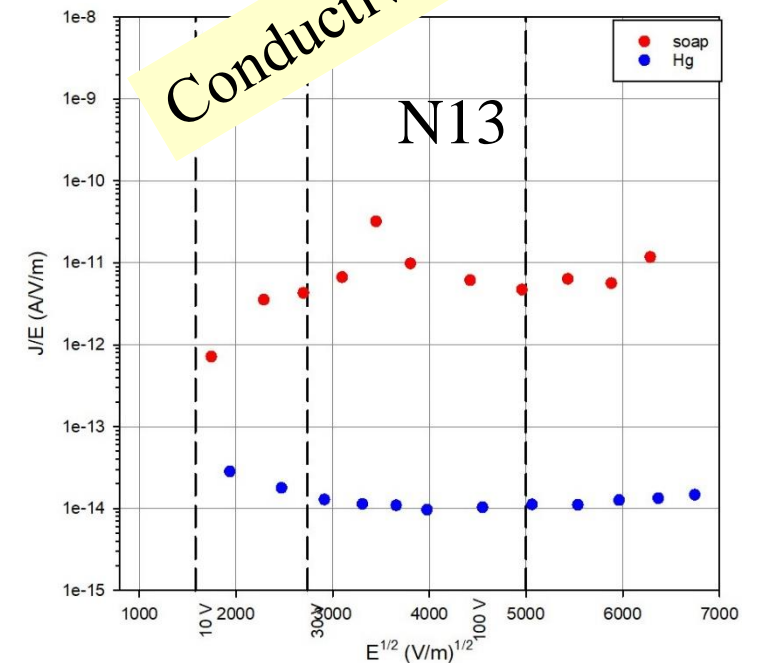
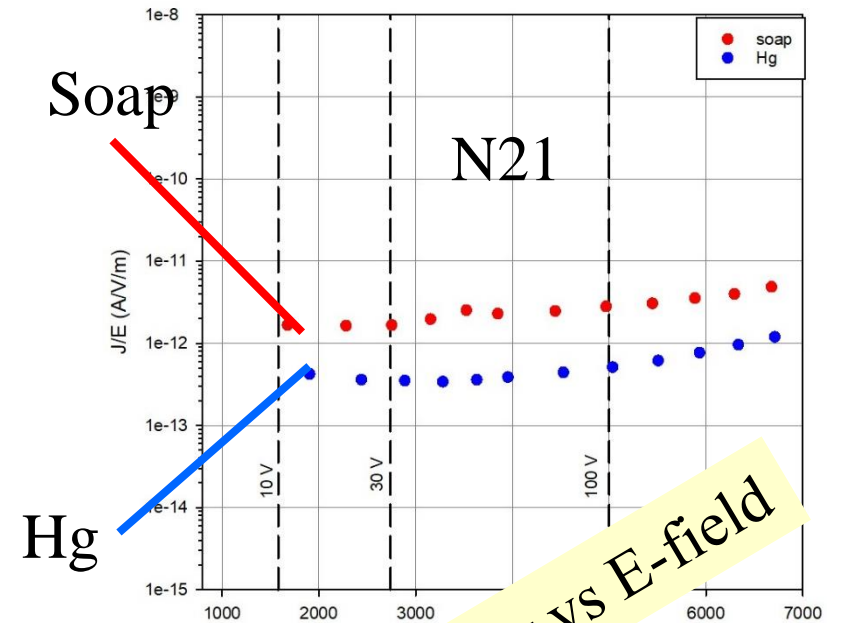
Mercury vs soap, which is best?

- Mercury has **high surface tension**
 - It may not fill pinholes
 - Does not always follow surface roughness well
- Soap water has **very low surface tension**
 - Creeps in every pinhole
 - (with the soap measurements I noticed some light sensitivity)
- What simulates best detector operation in gas?
 - Gas also fills every hole and unevenness



Which probe?

- The **mercury** probe normally give stable, reproducible results
- The **soap** probe always gives **higher** conductivity
 - Sometimes well reproducible
 - But often unstable currents, discharge/breakdown like phenomena
- My guess: **soap** is sensitive for irregularities, pin holes, roughness of the layer
 - A layer with stable HV behavior in soap might give reliable HV protection
 - *So soap may be used as a quality check for the layer*

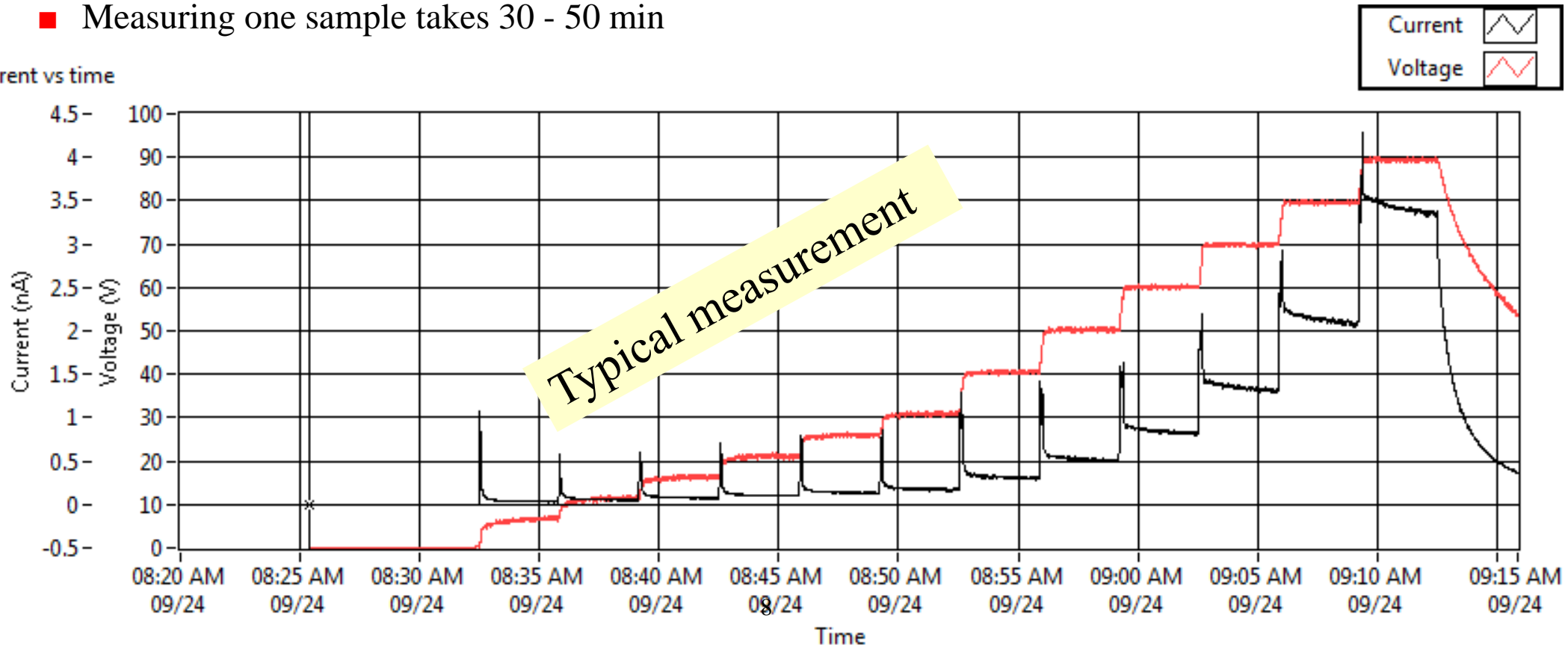


Measuring method

- LabVIEW program
 - Applying negative bias voltages of 0, 5, 10, 15, 20, 25, 30, 40, 50, 60, 70, 80 and 90 V
 - The higher voltages were tested to check the discharge protection
 - The average of 50 – 100 current measurements in the last 25 – 33% of the measuring period was registered
 - Currents were sometimes a few pA => long time needed to stabilize
 - Single negative bias voltage point takes 2 – 4 min
 - Measuring one sample takes 30 - 50 min

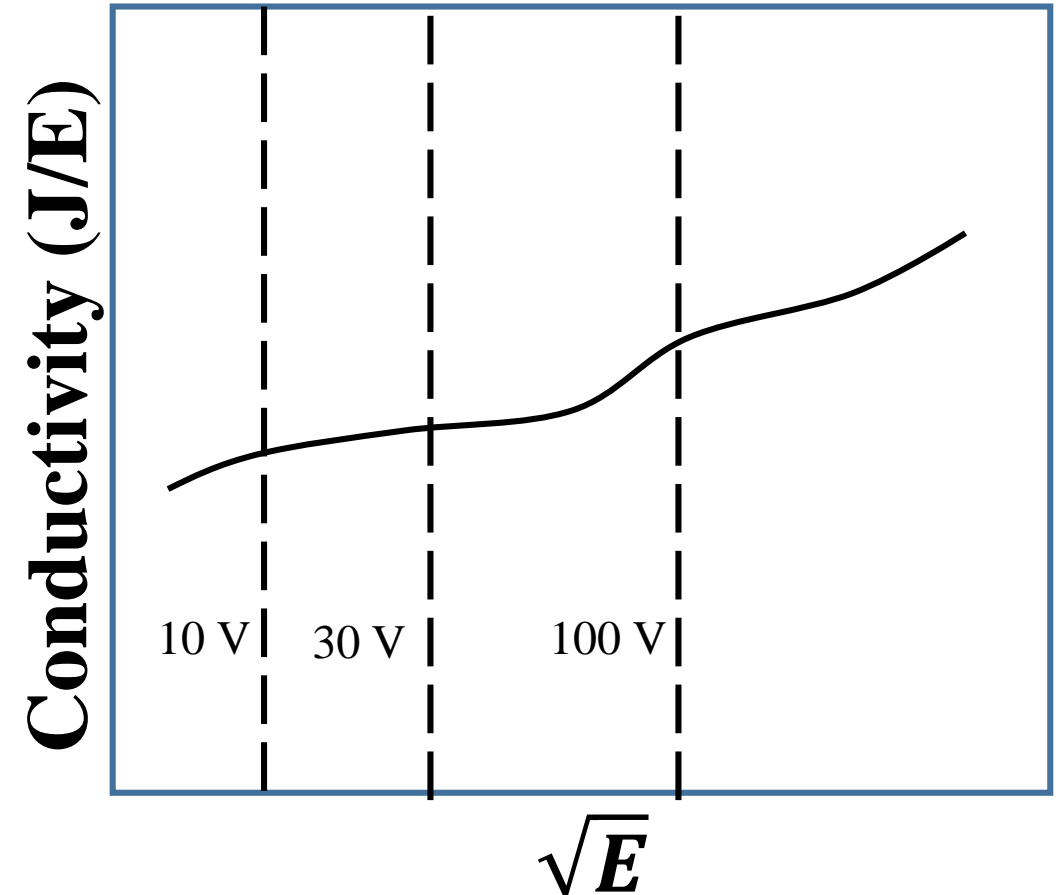
5 samples were also tested at **positive bias**
No significant difference was observed

Current vs time



Way of plotting

- **Conductivity vs square root of the field** across the layer
- Common practice when studying electrical thin layer properties
- Three reference lines plotted as potentials across 4 μm protection layer
 - 10 V \Rightarrow 75% gain for T2K gas
 - 30 V \Rightarrow 40% gain for T2K gas
 - 100 V \Rightarrow 5% gain for T2K gas
- We aim for the highest conductivity
 - Giving the highest rate capability



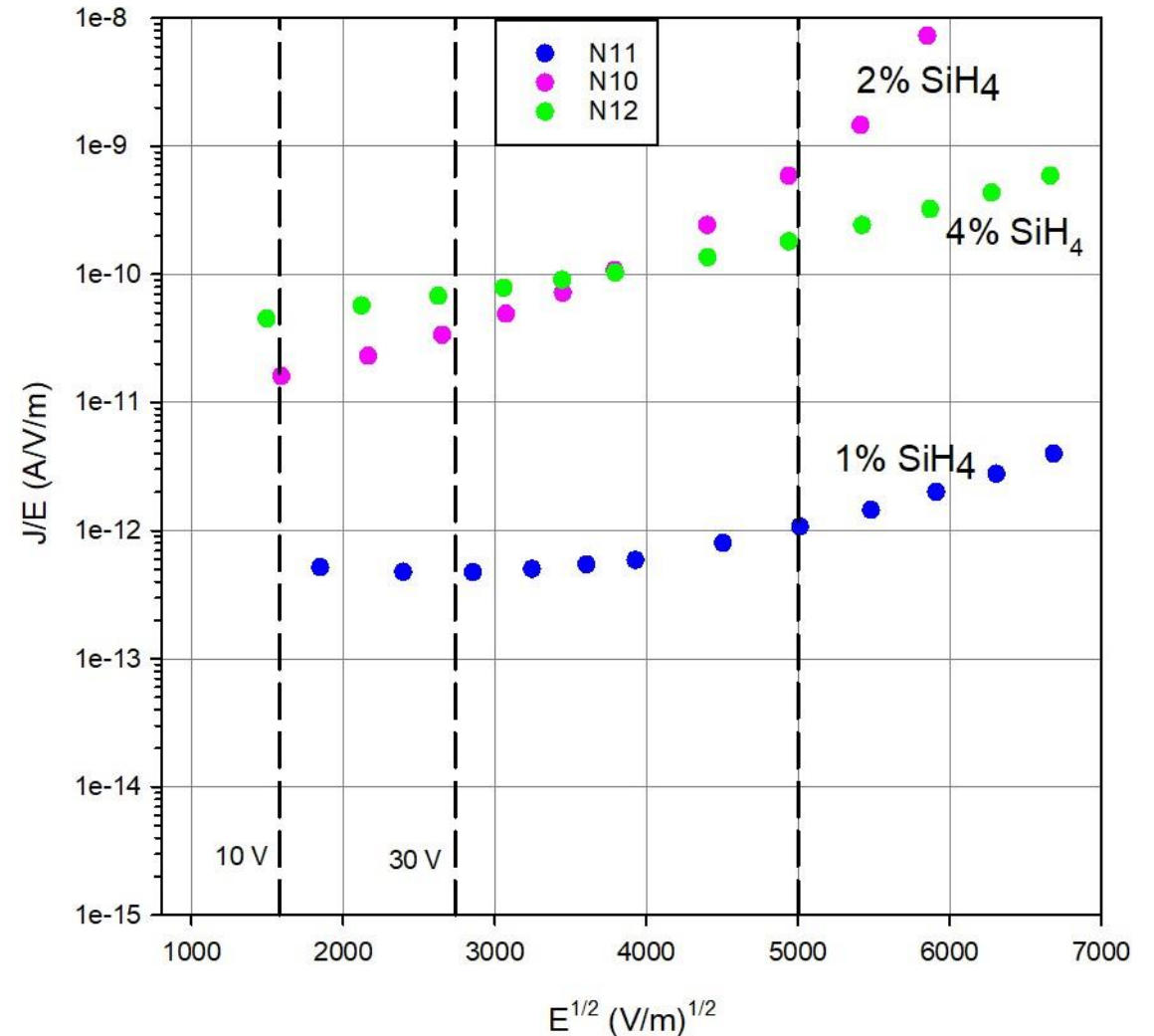
Sample N10 – N12

Plasma power **250 W**

- Conductivity of N10 (2% silane, **reference**) grows largely at higher fields
- Conductivity of N11 (1% silane) is much lower, too low to be useful
- Conductivity of N12 (4% silane) shows much less variation than N10
 - Higher conductivity at low fields

Conductivity (J/E) vs square root electric field ($E^{1/2}$)

Sample N10, N11, N12
Layer thickness 2 μm SixNy
Layer production Feb 2020 IZM
Tests by Hg probe
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Comparing reference sample N10 with earlier measurements

Conductivity (J/E) vs square root electric field ($E^{1/2}$)

Sample N10 and TPX3 dummy (bare si substrate)

Layer thickness 2 and 4 μm SixNy

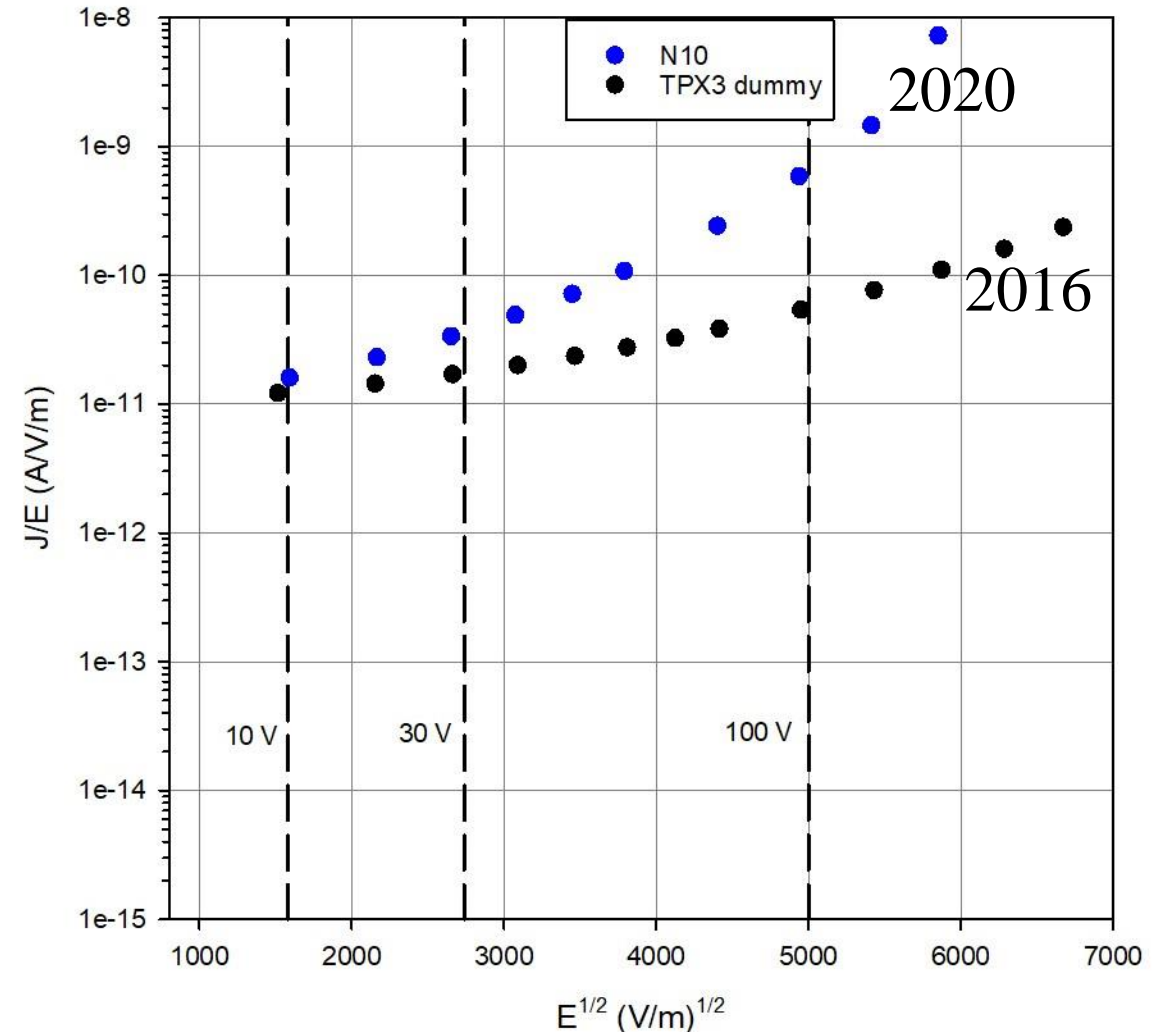
Layer production Feb 2020 IZM and 2016/2017

TPX3 dummy file <TPX3_dummy_4um_SiN_W3.txt> date 7-2-2017

Hg file <N10_repeated.txt> date 23-9-2020

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- Production of test samples in 2016
 - Using a bare silicon substrate covered with the protection layer (TPX3 dummy)
- At low layer potentials N10 and TPX3 dummy converge but for higher potentials the conductivity of N10 grows more rapidly
 - Less good spark protection??



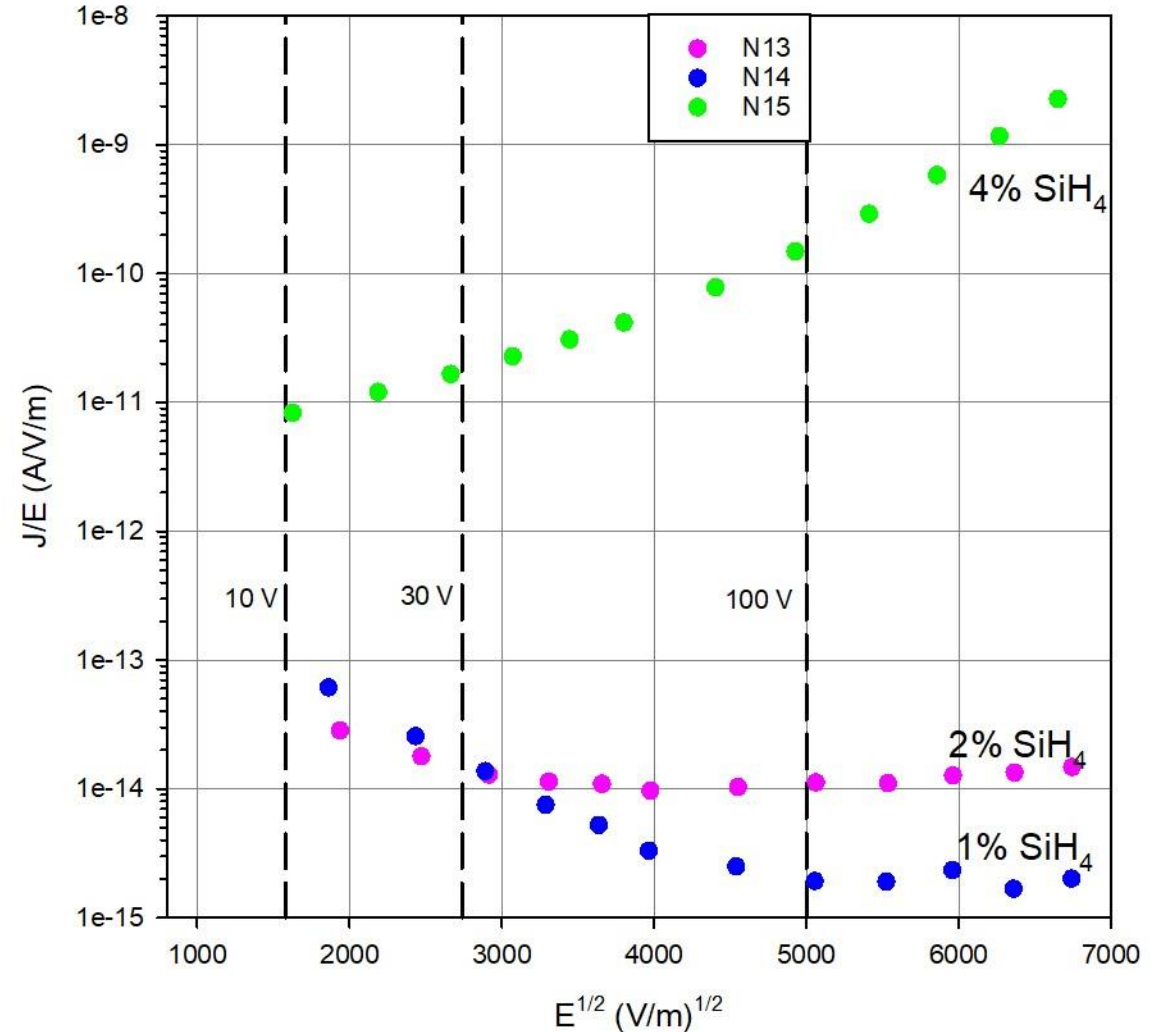
Sample N13 – N15

Plasma power **450 W**

- N15 (4% silane) is best, but still not as good as N12 or N10
- N13 and N14 have very low conductivity
 - Hard to measure (pA currents)

Conductivity (J/E) vs square root electric field ($E^{1/2}$)

Sample N13, N14, N15
Layer thickness 2 μm SixNy
Layer production Feb 2020 IZM
Hg measurements
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Sample N16 – N18

Plasma power 900 W

- All very low conductivity, almost **no effect on silane concentration**
- Observed differences on conductivity between various silane concentration not significant

Conductivity (J/E) vs square root electric field ($E^{1/2}$)

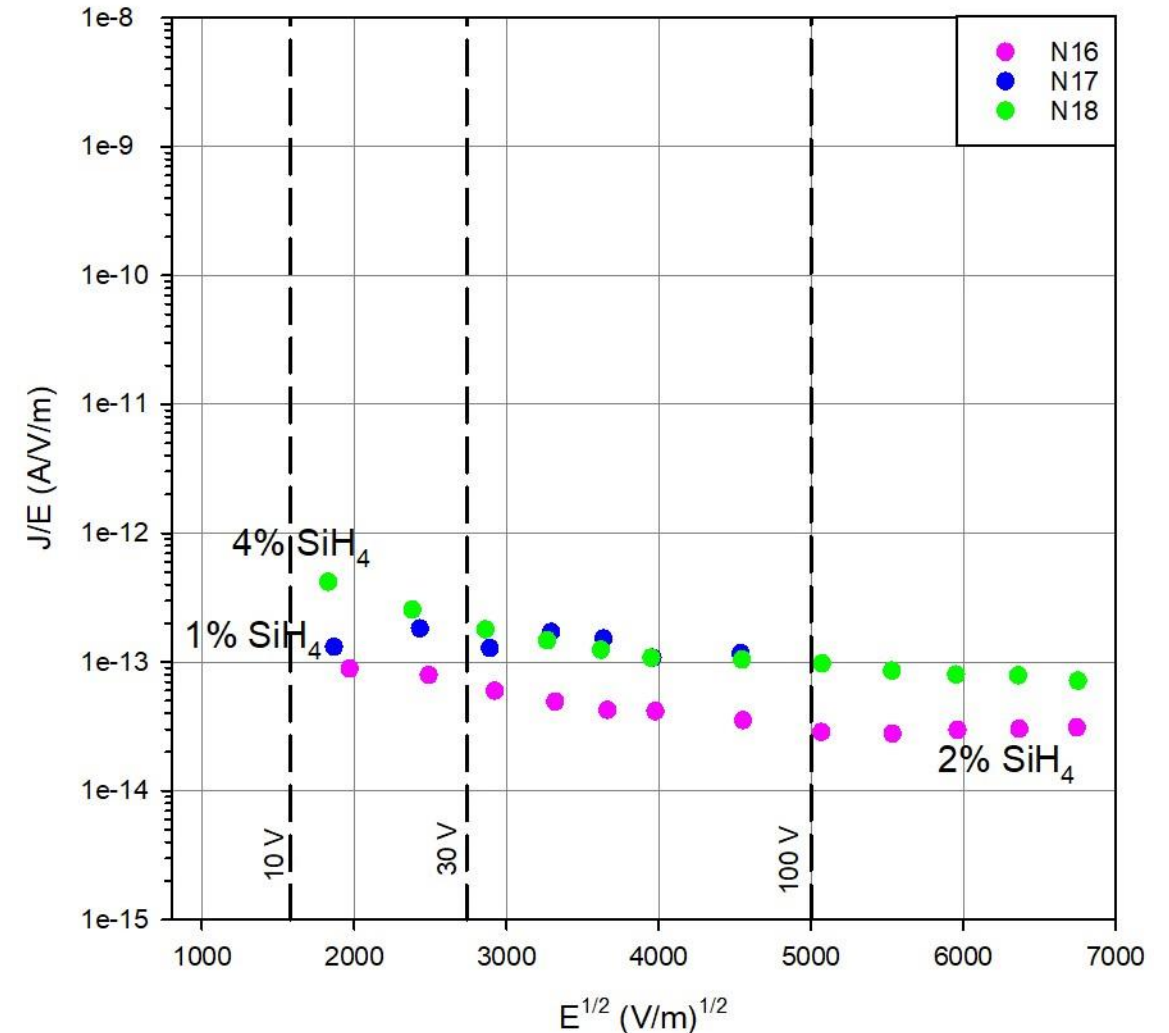
Sample N16, N17, N18

Layer thickness 2 μm SixNy

Layer production Feb 2020 IZM

Hg measurements

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Sample N19 and N21

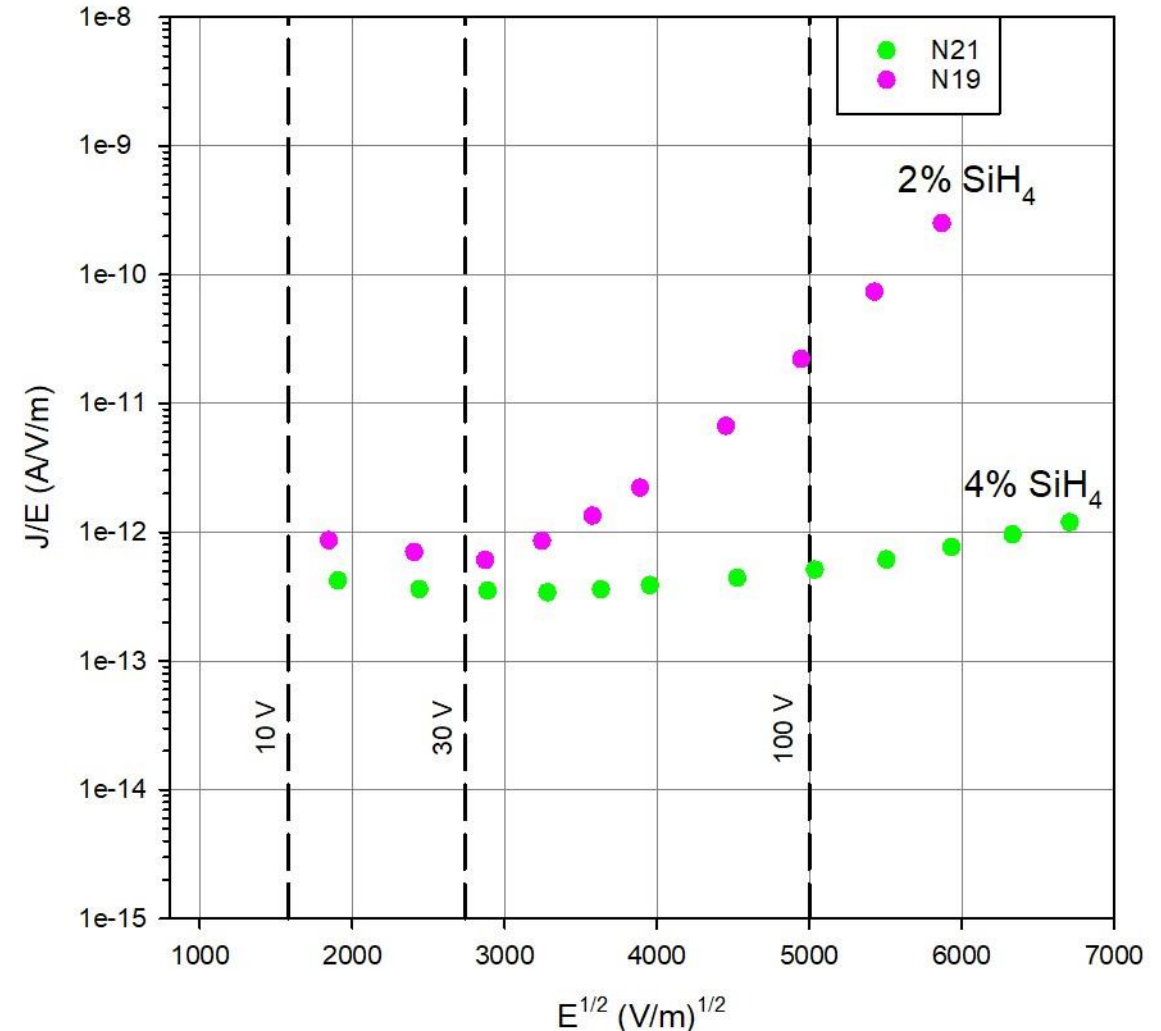
Plasma power **250 W**

low frequency plasma

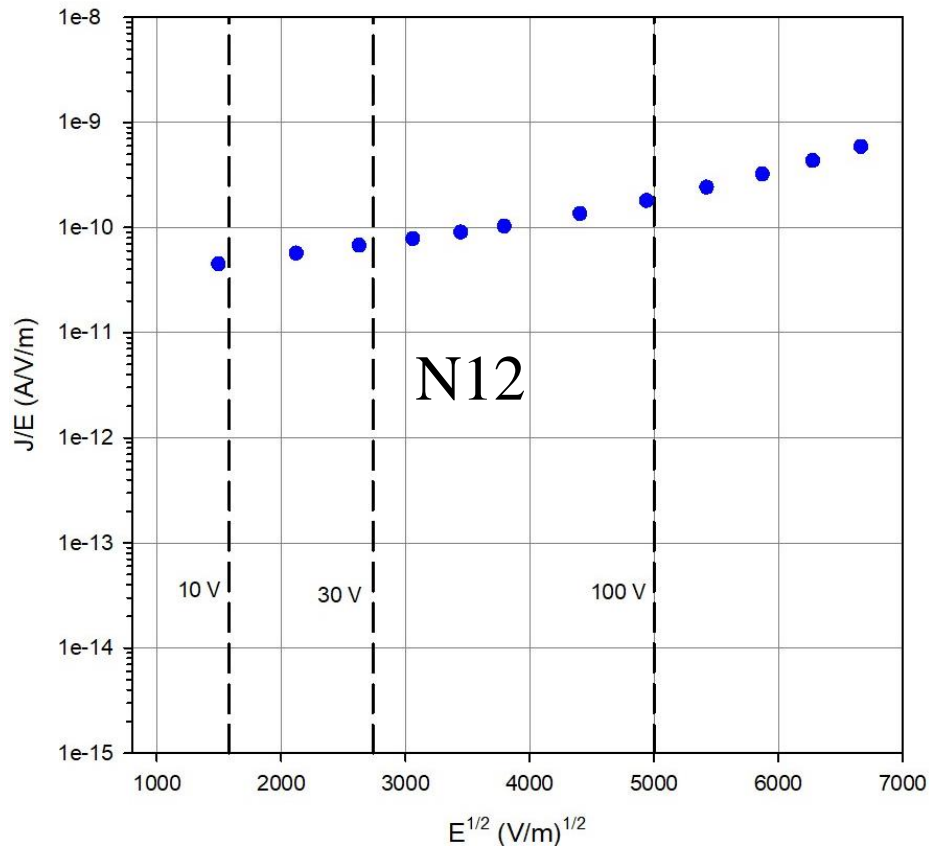
- Surprisingly the 4% silane sample has a **lower conductivity** than the 2% one
- All conductivities are lower than the reference sample N10

Conductivity (J/E) vs square root electric field ($E^{1/2}$)

Sample N19 and N21
Layer thickness 2 μm SixNy
Layer production Feb 2020 IZM
Hg measurements
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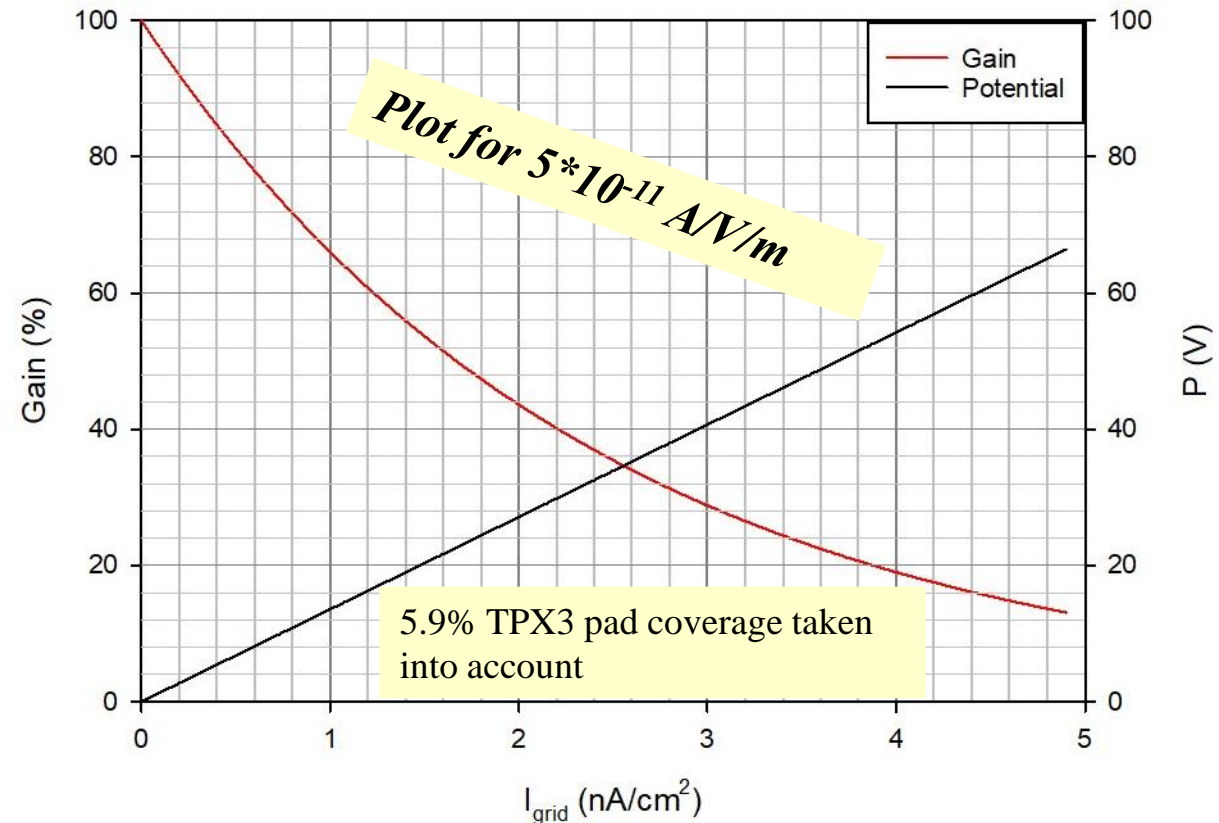
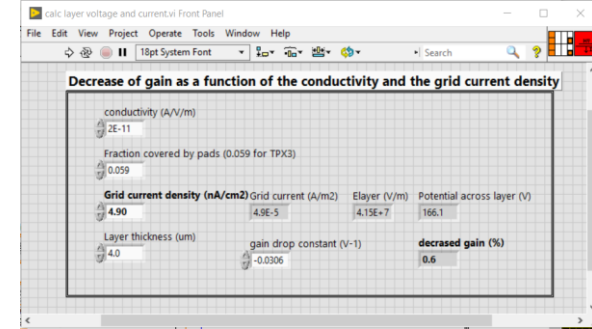


- The highest conductivity from these samples is given by **N12**
 - 4% silane, 250 W, HF
 - Conductivity @ 10 V: $5 \cdot 10^{-11}$ A/V/m
 - Conductivity @ 30 V: $8 \cdot 10^{-11}$ A/V/m
 - => at **2.5 nA/cm²** we have a gain drop of 50% for T2K gas
 - **Reference sample: 50% gain drop at 0.7 nA/cm²**



Gain as a function of conductivity and grid current

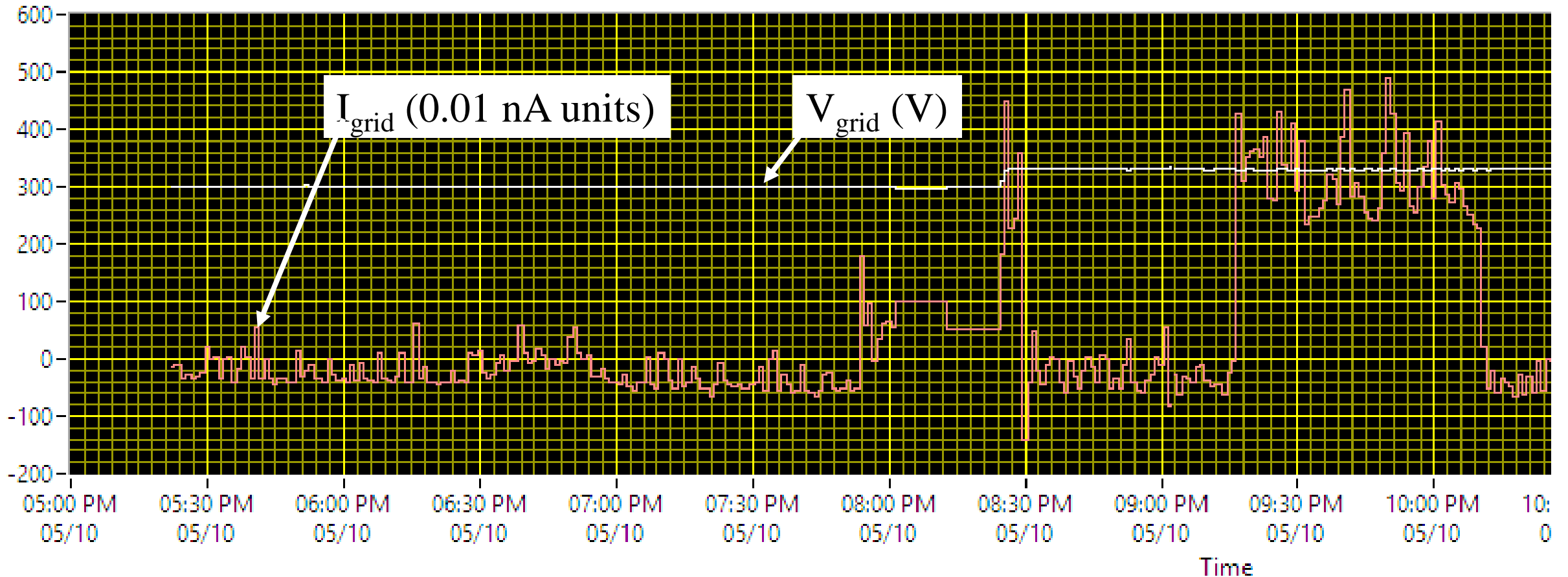
LabVIEW routine



Grid currents during 2018 testbeam in Bonn

- 1 nA @ 300 V_{grid} => **0.4 nA/cm²**
- 2.7 nA @ 330 V_{grid} => **1.1 nA/cm²**

Covered surface by beam ~ 2.5 cm²



Additional remarks and conclusions

- I found some indication of a **temperature dependence**
 - Conductivity increasing at higher temperatures
 - During operation the chips have a temperature increased by 10 – 20 °C, this may significantly enhance the rate capability
 - In Bonn the chip temperature was possibly ~ 40 °C
- *At my home I have no opportunity to study this effect*
- Most samples show **worse performance** compared to the reference N10
- **Only N12** has higher conductivity but the improvement compared to N10 is limited
 - A significant improvement can be achieved by enlarging the TPX3 pad size by postprocessing
- Before using the recipe of N12 on a new batch of chips, at first extensive tests have to be done to check the reproducibility of the process and the spark protection (maybe by the soap probe as well)