

Investigating protection layers

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Nikhef/Bonn LepCol meeting
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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 Nikhef	SiH ₄ /N ₂ (%)	SiH ₄ (sccm)	N ₂ (sccm)	Plasma power (W)	Frequency (HF / LF)	Deposition time (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**



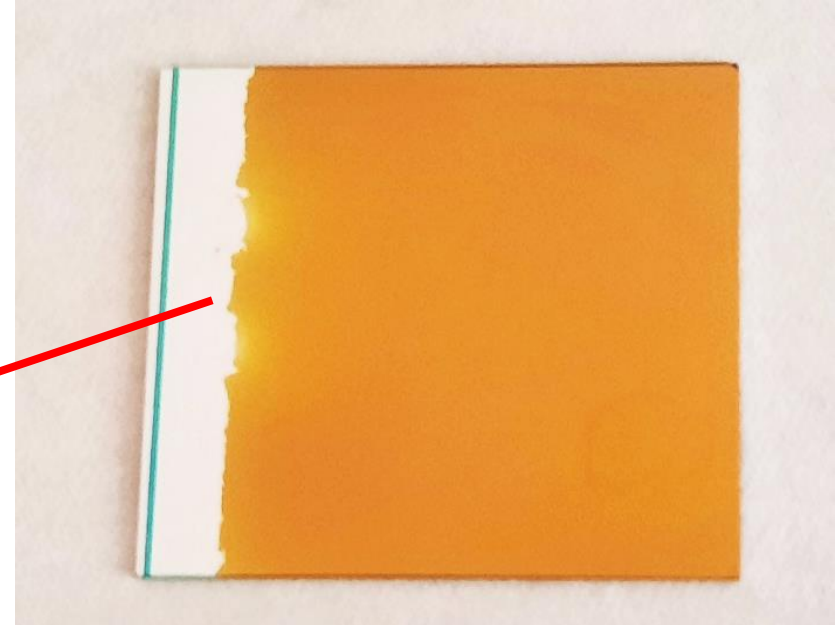
Mercury probe



The samples

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

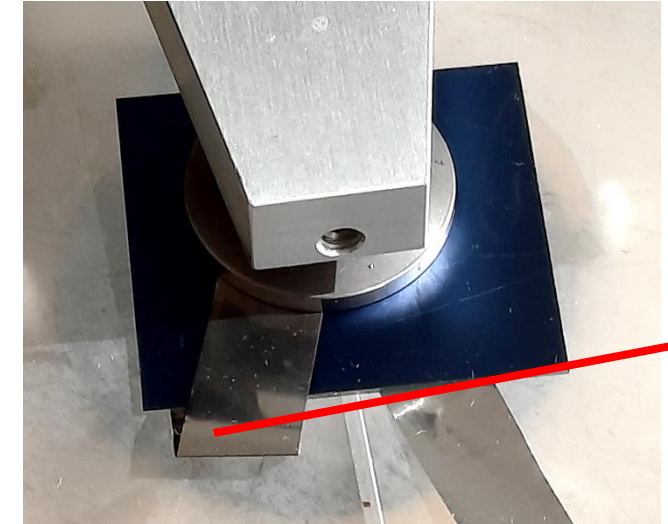
- Mercury sucked by vacuum to make contact under the sample
- Contact surface 18.9 mm^2
- $10 \text{ }\mu\text{m}$ SS contact foil needed
 - Sample is insulated at bottom side

Mercury contact

(Top view, seen through a glass plate)



Sample under Hg probe

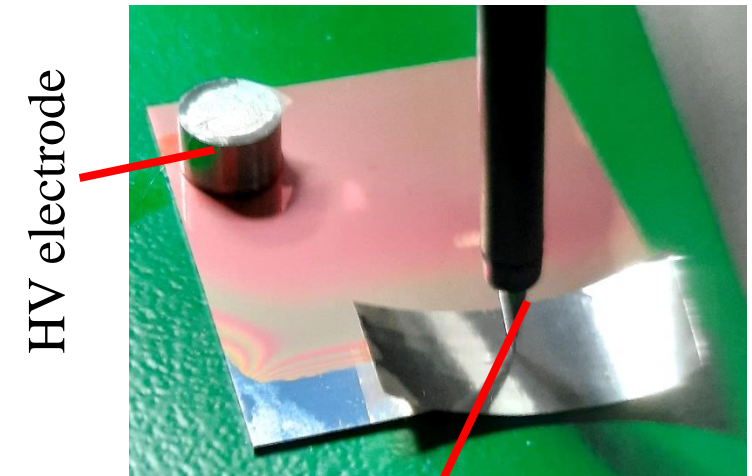


Contact strip

■ Soap water probe

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

Sample with soap probe

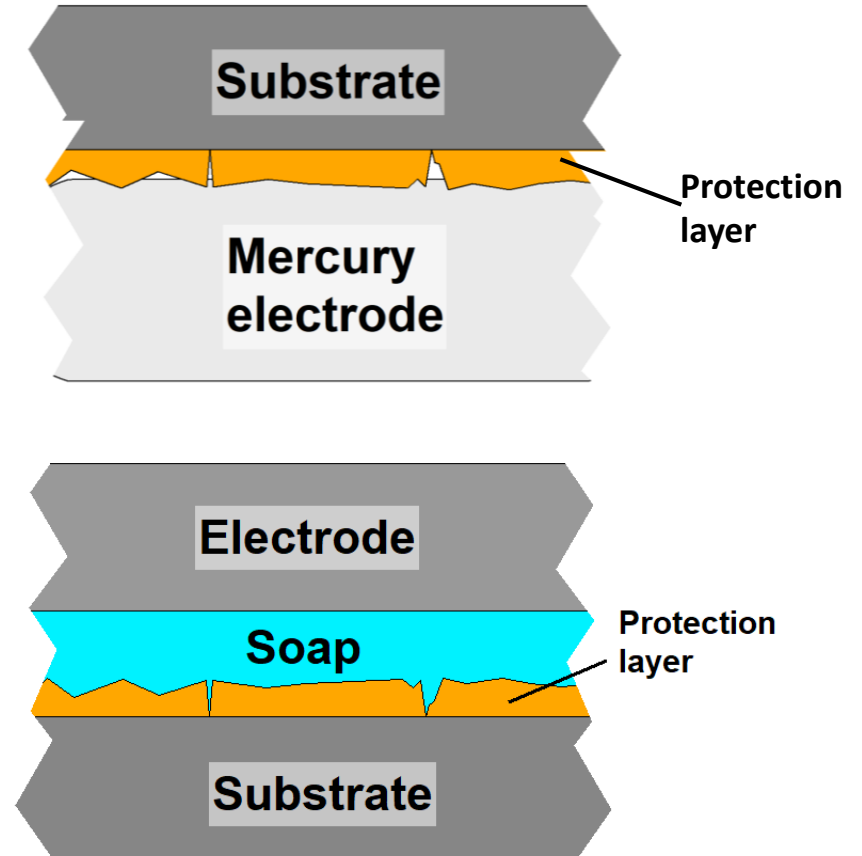


HV electrode

Return contact to
pA meter

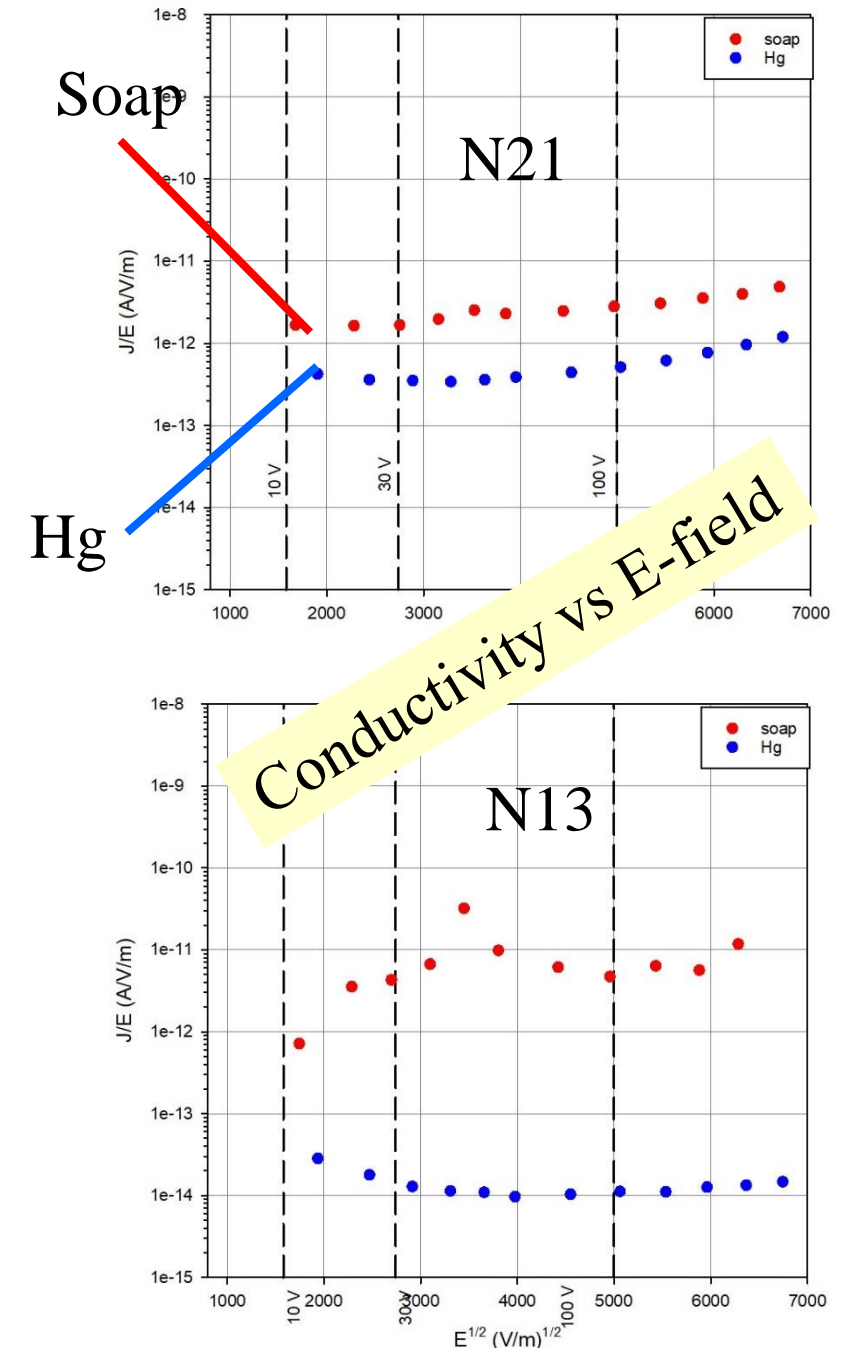
Mercury vs soap, which is best?

- **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 gives 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*



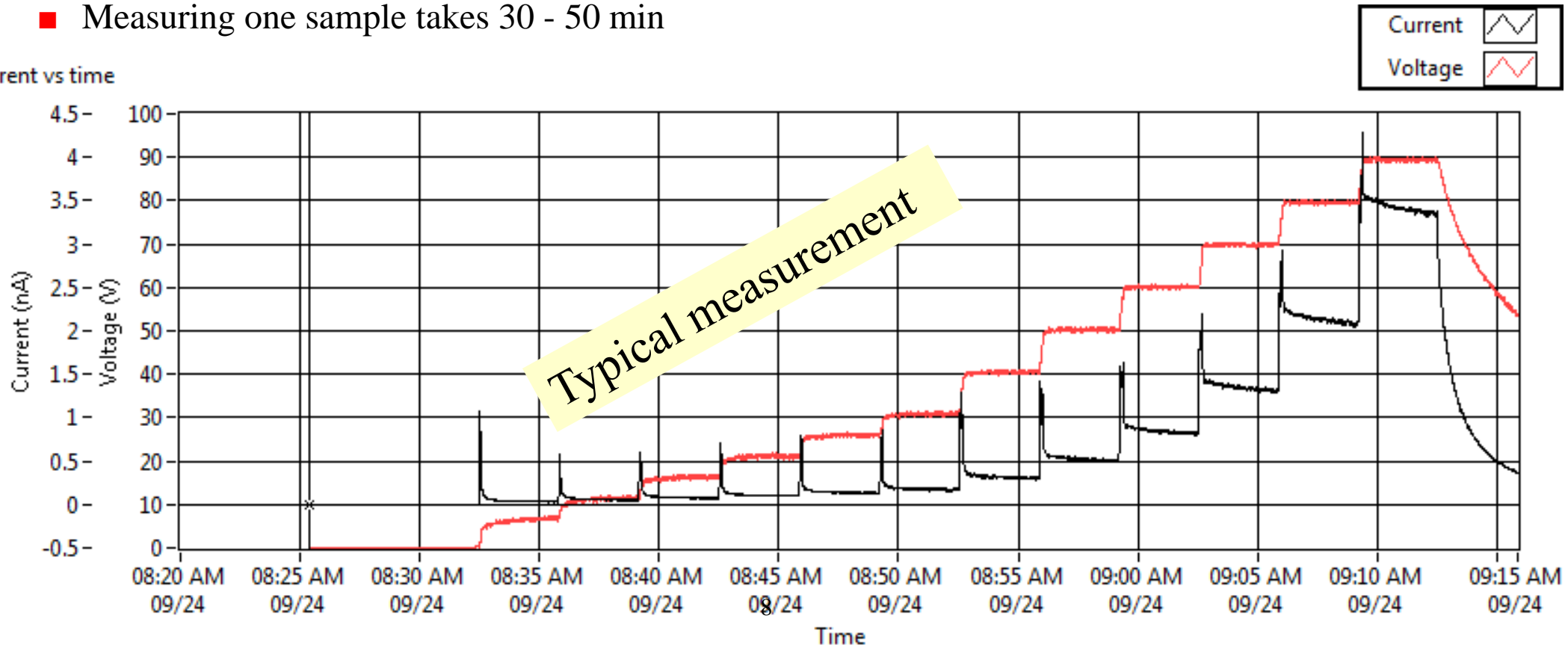
■ LabVIEW program

- 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

Measuring method

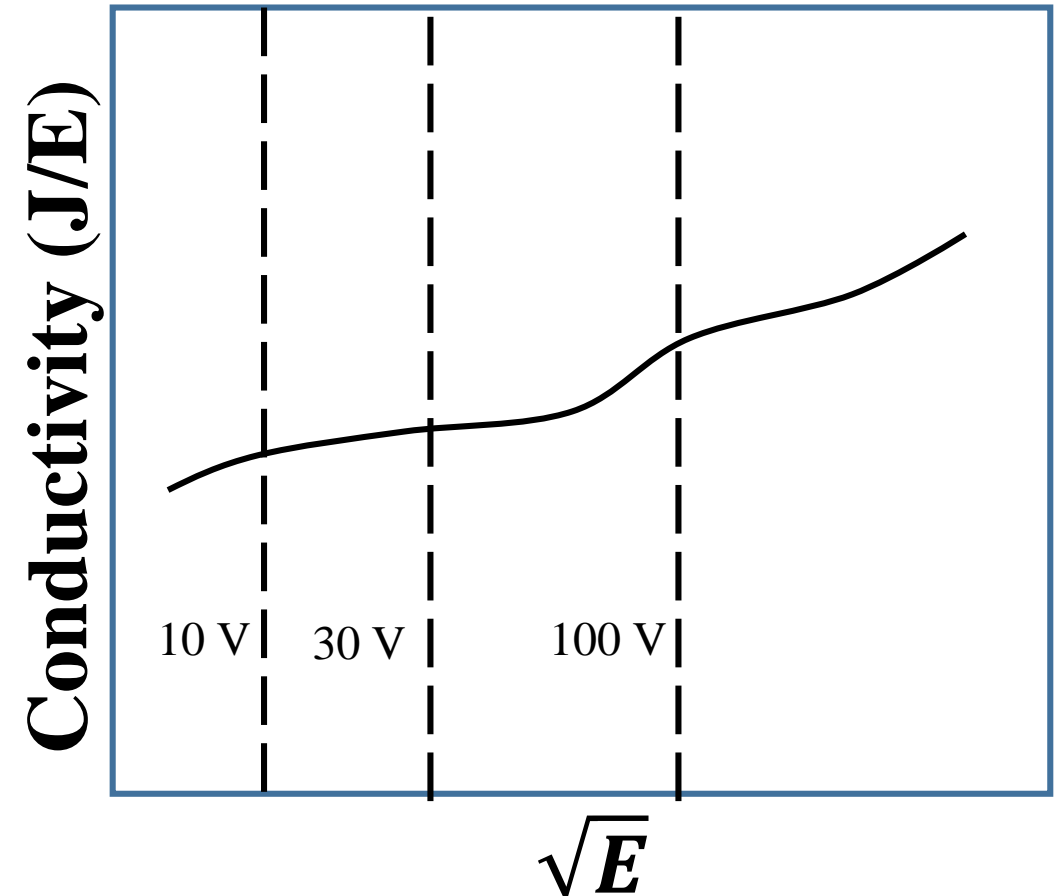
5 samples were also tested at **positive bias**
No significant difference was observed

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



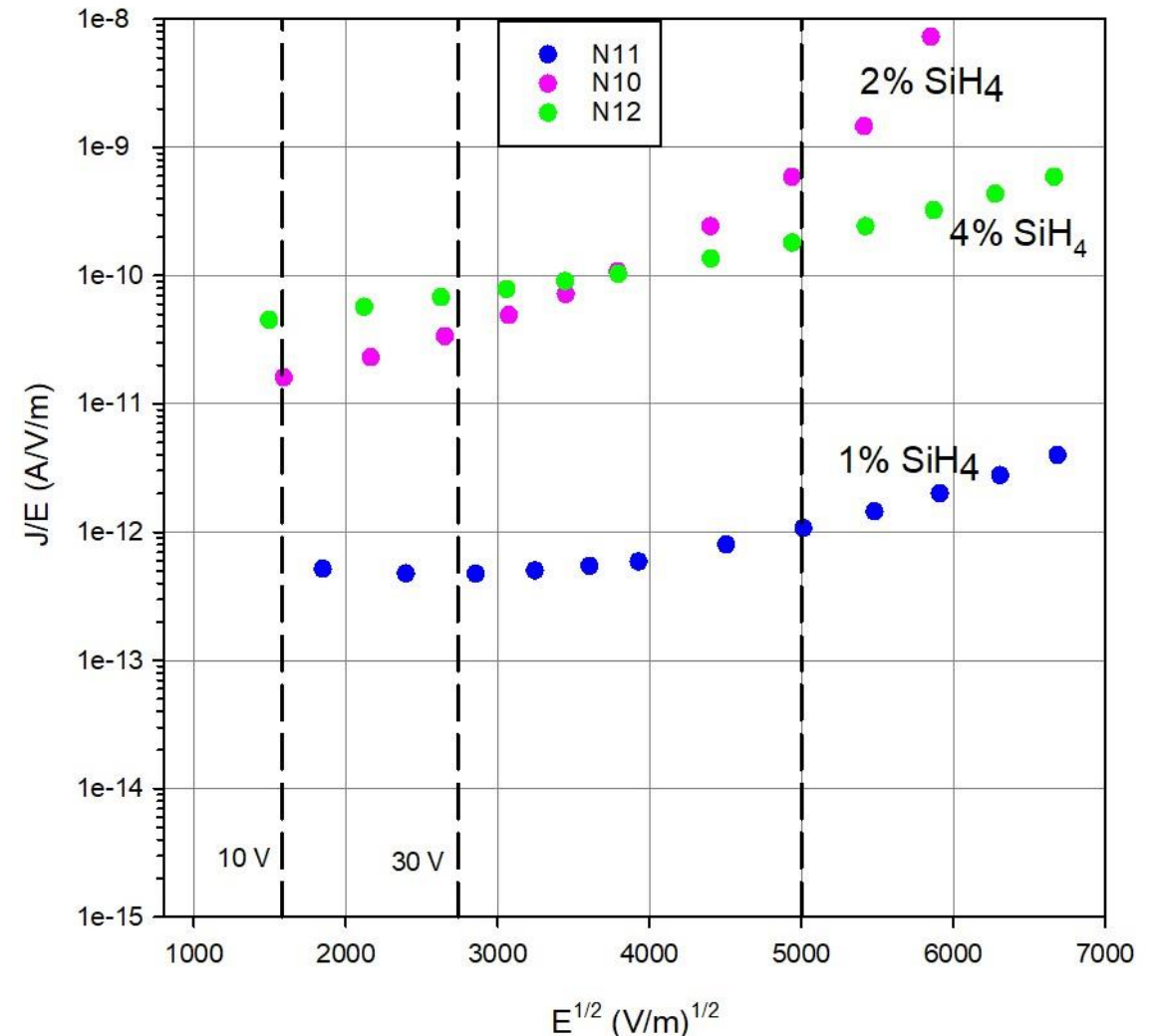
Sample N10 – N12

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

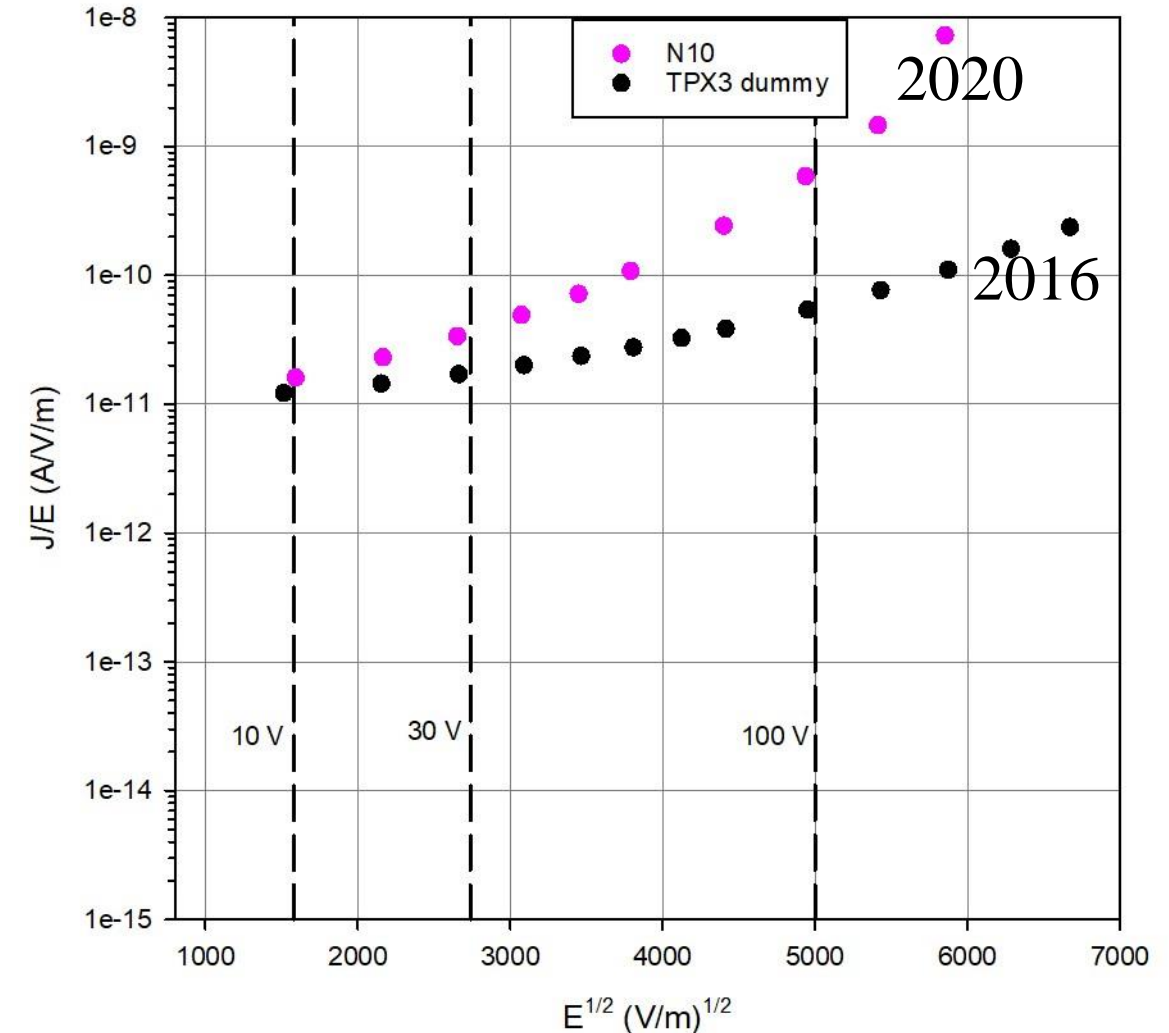
Layer production Feb 2020 IZM and 2016/2017

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??



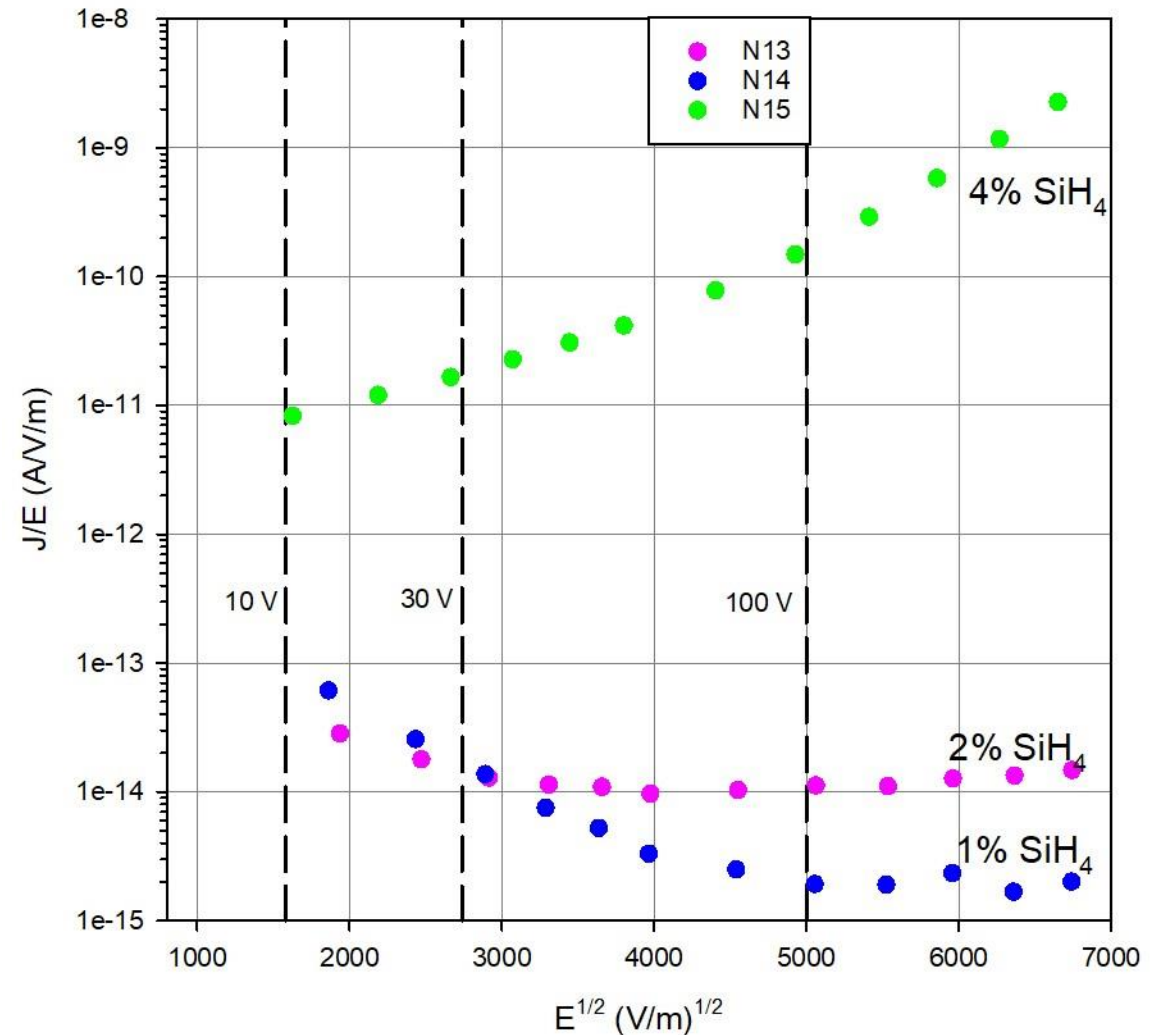
Sample N13 – N15

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 ($E^{1/2}$)

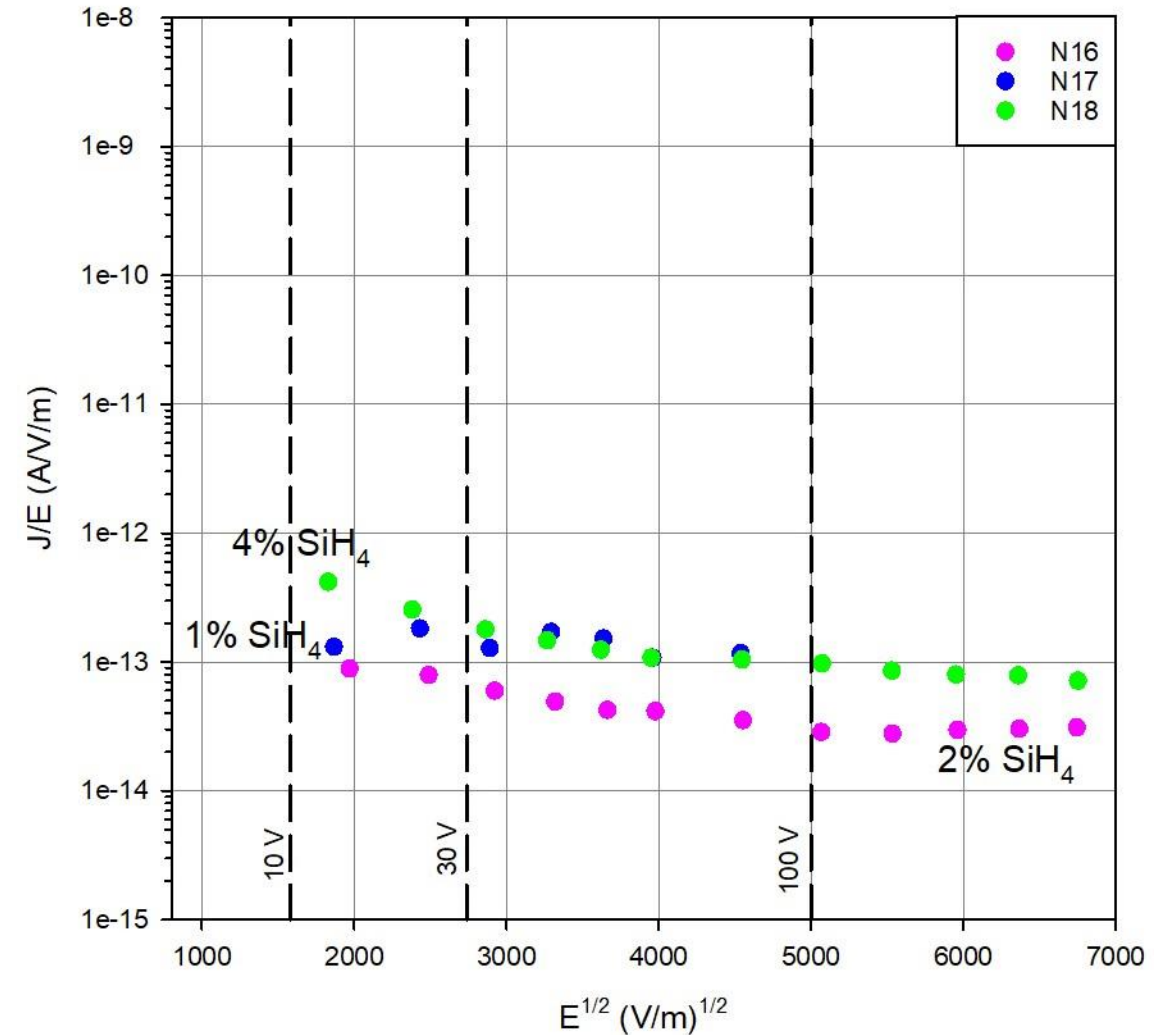
Sample N16, N17, N18

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 ($E^{1/2}$)

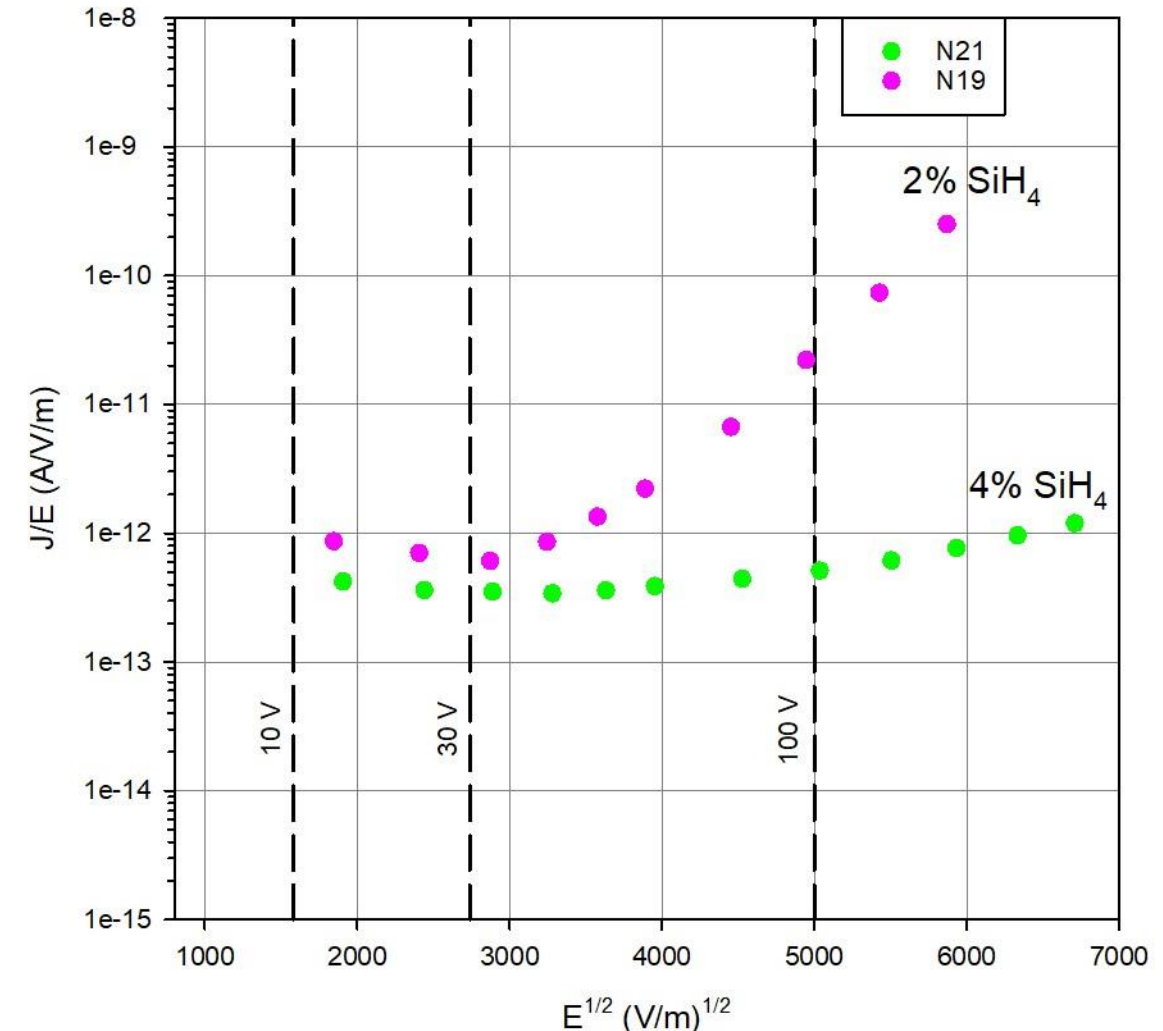
Sample N19 and N21

Layer thickness 2 μm SixNy

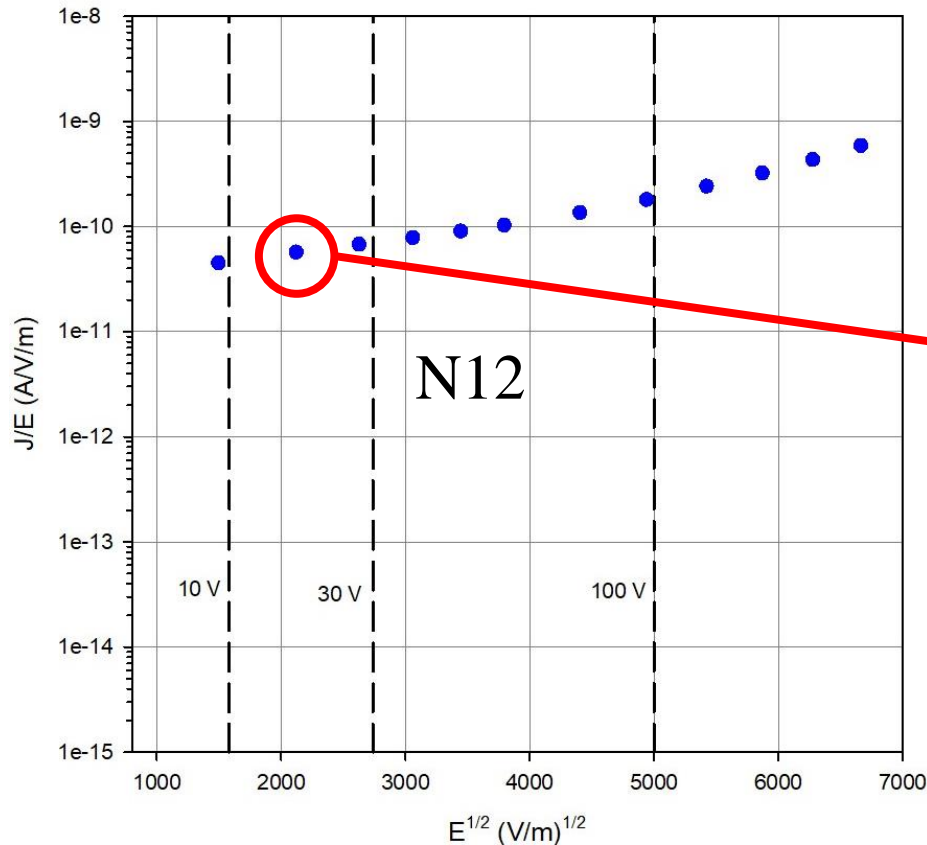
Layer production Feb 2020 IZM

Hg measurements

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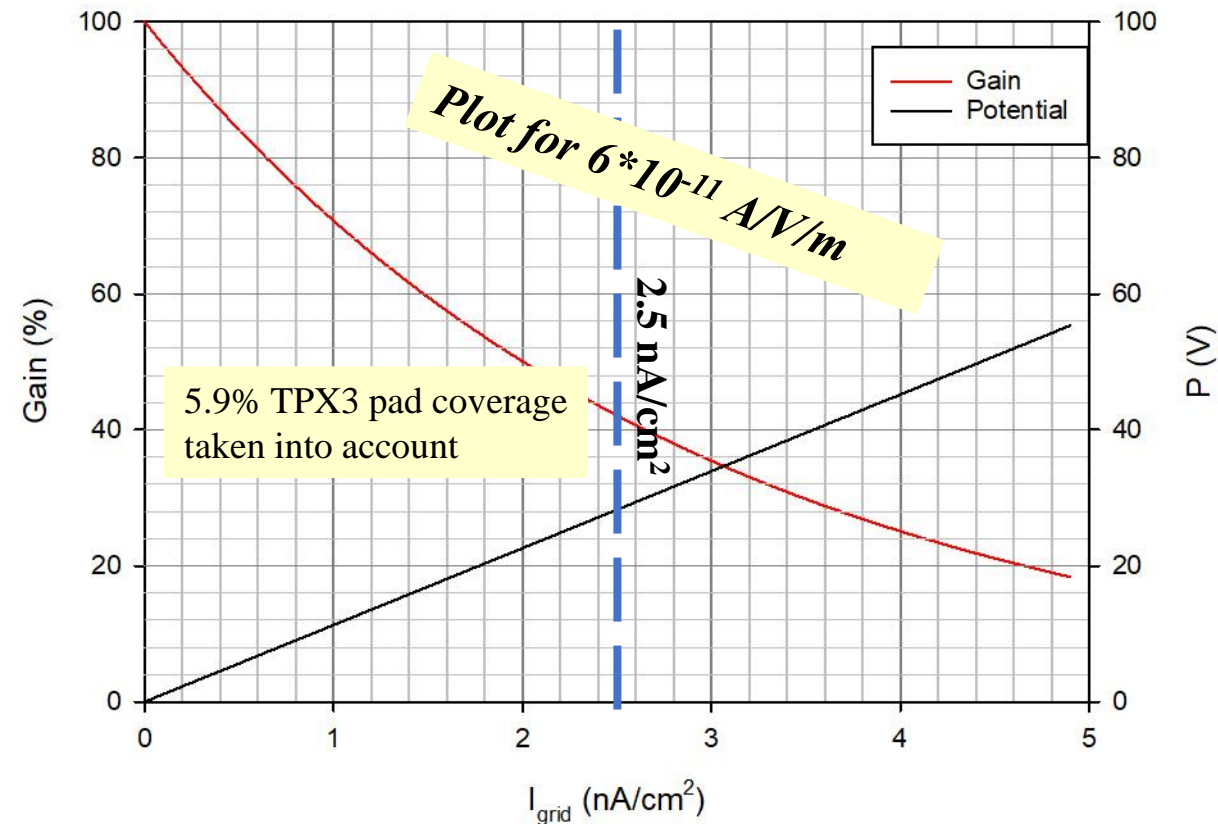
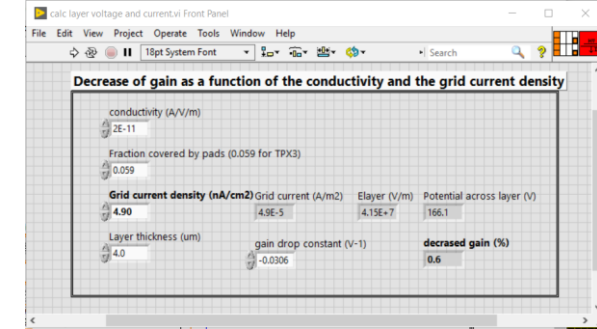


- The highest conductivity from these samples is given by **N12**
 - 4% silane, 250 W, HF
 - Conductivity @ 10 V: $5 \cdot 10^{-11}$ A/V/m
 - Conductivity @ 30 V: $8 \cdot 10^{-11}$ A/V/m
 - \Rightarrow at **2.5 nA/cm²** we have a gain drop of 50% for T2K gas
 - **Reference N10**: 50% gain drop at **0.7 nA/cm²**



Gain as a function of conductivity and grid current

LabVIEW routine

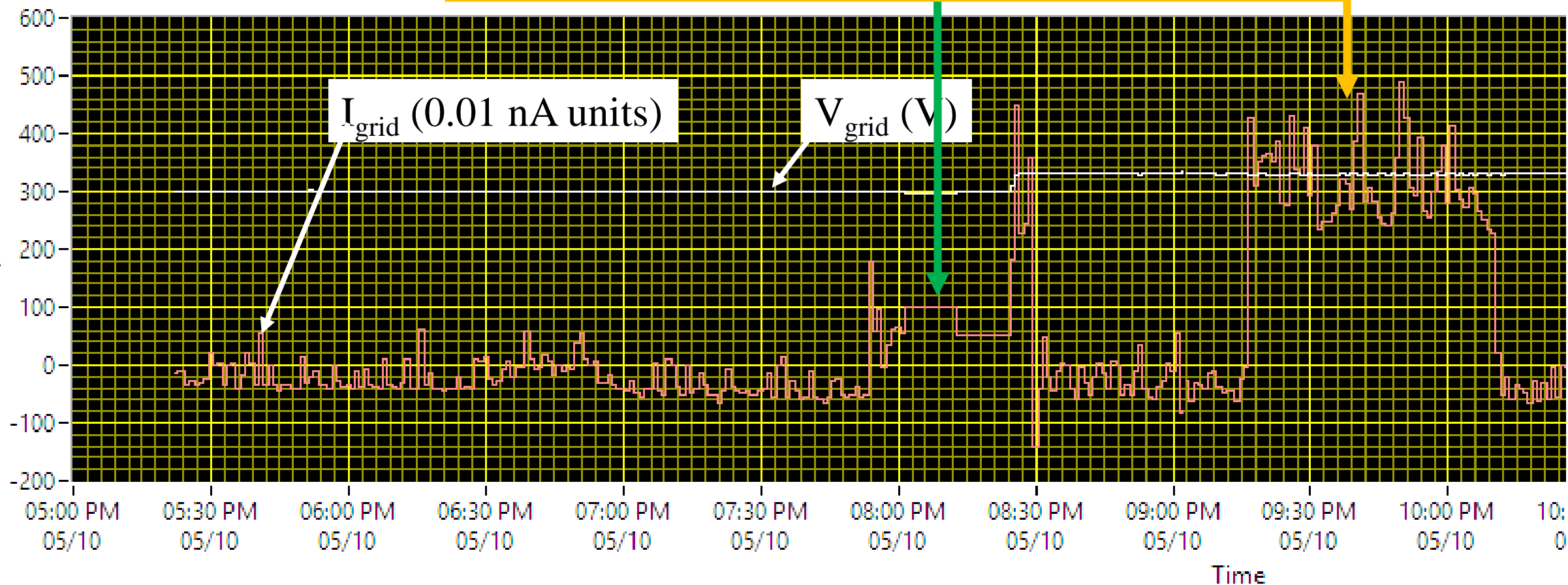


Grid currents during 2018 testbeam in Bonn

■ 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

- While testing 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 => reduced rate effects
- *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 (3 – 4) can be achieved by enlarging the TPX3 pad size by postprocessing
 - The pads of TPX3 cover only **5%** of the chip surface
- 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 (the **soap probe test**)