European School of Instrumentation
in Particle & Astroparticle Physics

European Scientific Institute

DETECTOR TECHNOLOGIES

Lecture 4: An history of R&D : Diamond Detectors.

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Diamond detectors

	CVD Diamond	**Si**
Z	6	14
Energy Gap	5,5 eV	1,21 to 1,1eV
Resistivity	10^{13} – 10^{16} Ωcm	10^{5} – 10^{6} Ωcm
Breakdown	10^{7} V/cm	3.10^{5} V/cm
Mobility (electrons)	2000 $cm^2/V/s$	1350 $cm^2/V/s$
Mobility (holes)	1600 $cm^2/V/s$	480 $cm^2/V/s$
Displacement Energy (e^-)	43 eV/atom	13 à 20 eV/atom
Pairs Creation	13 eV	3.6 eV
Charge Collection Distance	250 μm	100 m ?
Mean signal (MIP)	3600 e^- / μm	8900 e^- / μm
Dielectric Constant	5.5	10 à 12
Thermal Conductivity (W/m·K)	1600 - 2000	150

← Low noise (gap difficult to cross)

← Fast signal

← **SMALLER SIGNAL**

← Low capacitance, low noise

← No need for extra cooling

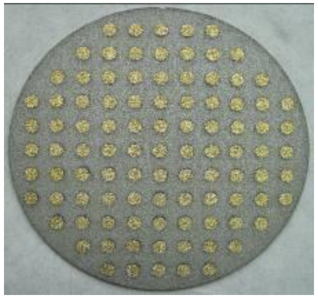
Semiconductors : diamonds

Diamond is **better** than Silicon

- Does not need any doping
  - In ay case, extremely difficult to implant something…
- Better radiation hardness
- Better thermal conductivity
- Better speed (1psec vs 1 nsec)
- Light insensitive
- Multi-metalization possible (test and physics)

But :

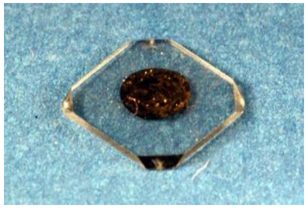
- **3 times less signal** for MIPs (3.6 / 13)
- Difficult to manufacture
- Expensive



2 forms :

Polycristalline

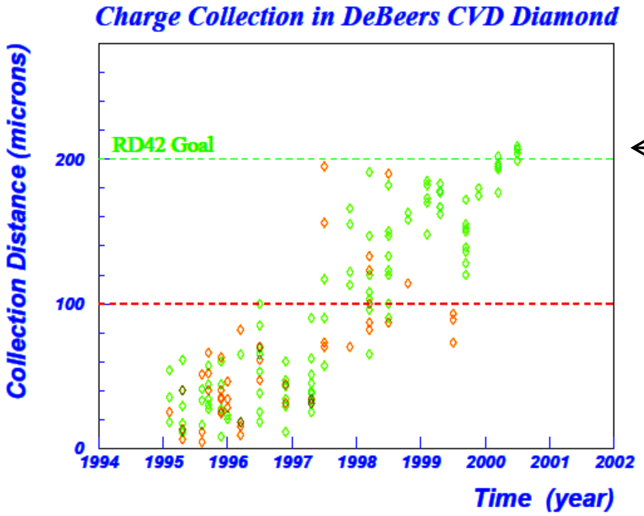
Wafer max.6 inches



Monocrystalline

max : 4 x 4 mm ^{2}

Quality improve →



Charge Collection in DeBeers CVD Diamond

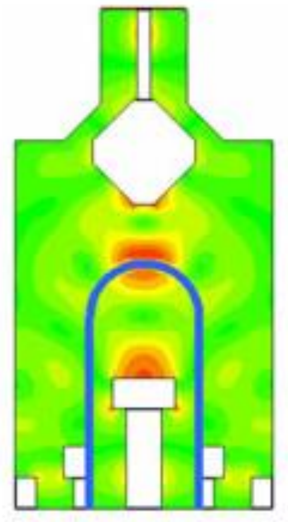
← "Good" diamond

Diamant ?

Natural diamond: Lots of impurities
Lots of defects

Diamant HPHT (High Pressure – High Temperature) 1940
$P > 50\ 000$ bars
$T > 2000$ °C
Monocrystal
Dimension (few mm^2)
Impurities

Diamant CVD (Carbon Vapor Deposition) 1980
Plasma CH4 – H2 (+X…)
$P \approx 0,1$ bar
$T \approx 1000$ °K
Slow deposition on substrate
1 – 50 µm / heure



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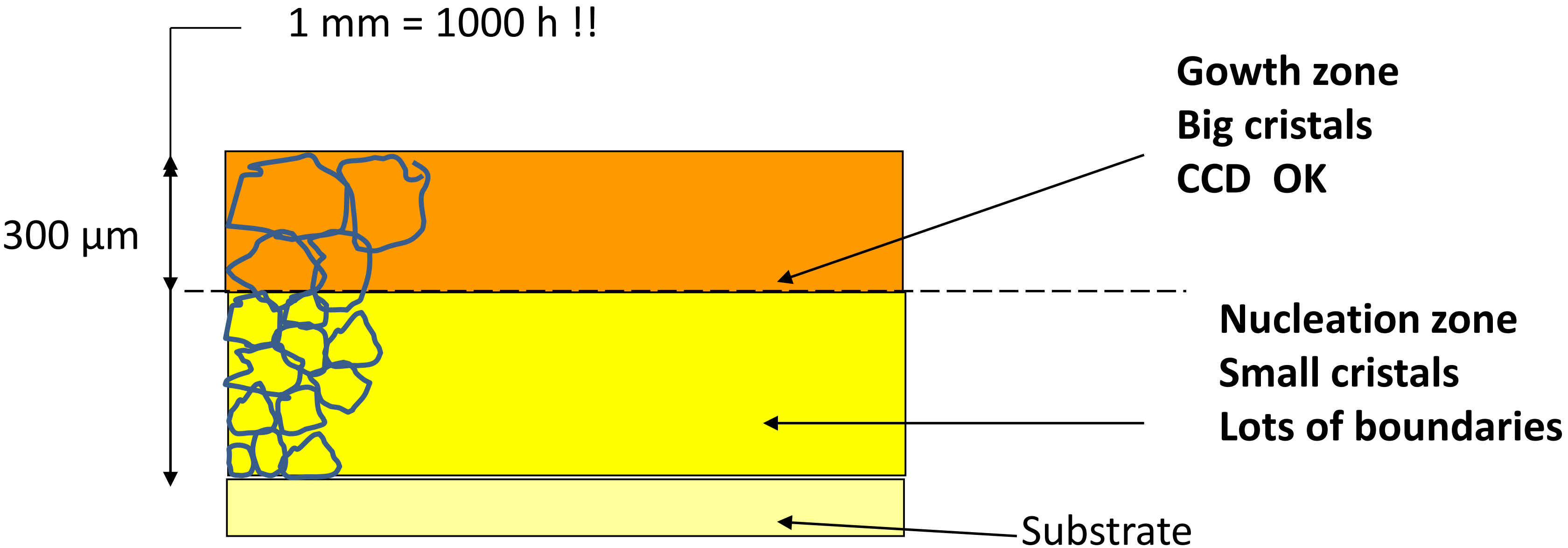
Polycristalline CVD (pCVD)

Grows on any substrate (Si)
Slow process (around 1µm / h)
Cristal bigger along the process
Industrial well controlled process



Gowth zone
Big cristals
CCD OK

Nucleation zone
Small cristals
Lots of boundaries

Substrate

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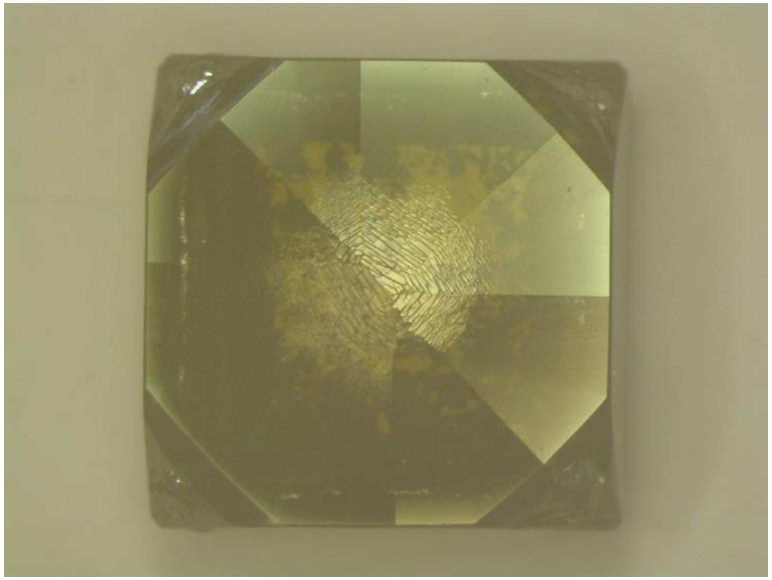
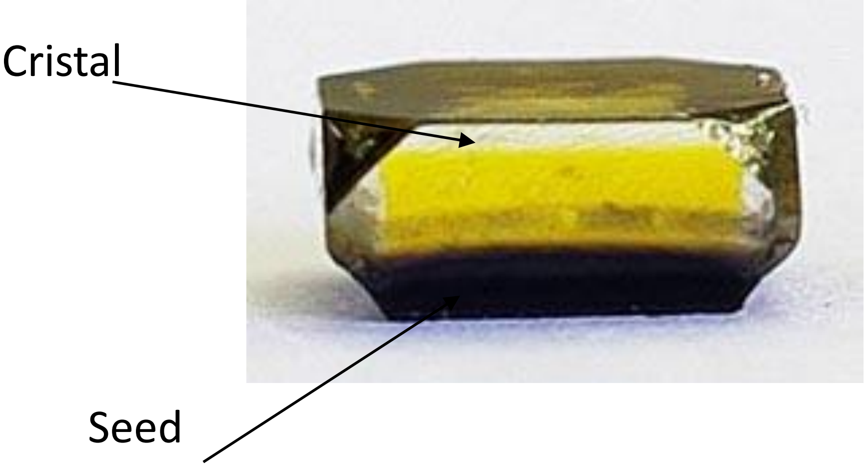
Monocrystalline CVD (sCVD)

Grows only on another monocrystal (HPTHT seed)
small dimensions (typical 4 x 4 mm^2)
faster processus : 25 µm /h)
Very few industrial manufacturers





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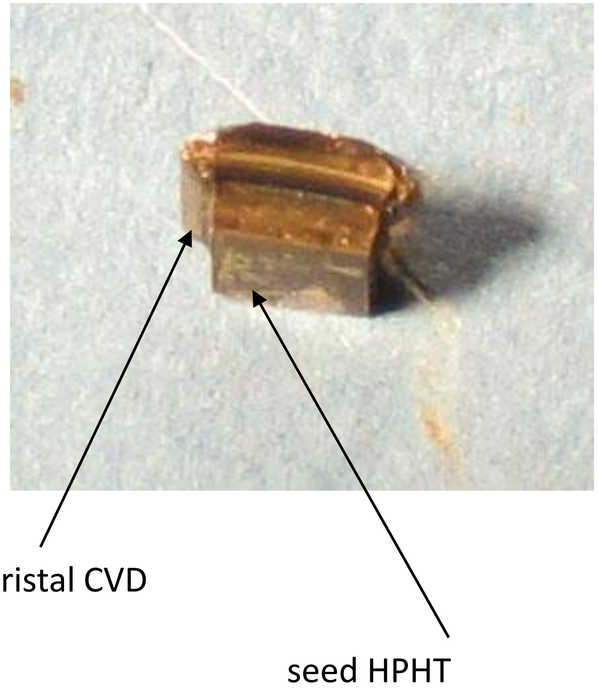
Semiconductors : Diamonds

Finishing :

pCVD : Nucleation (small grains) supppression)

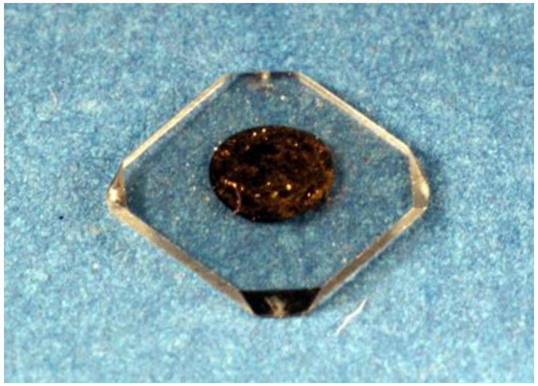
sCVD : Seed suppression



Métalisation (cf bonding)

Cleaning
Re- métalisation

charactérisation (CCD…)



Semiconductors : Diamonds

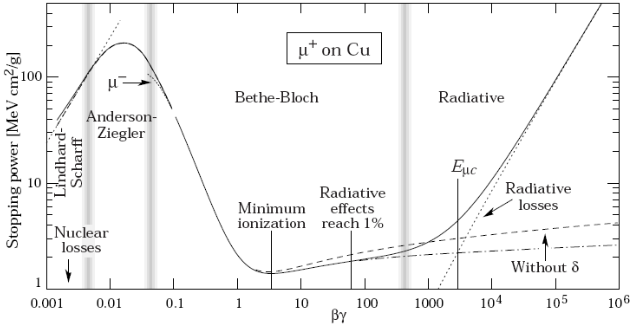
CCD : CHARGE COLLECTION DISTANCE

Energy loss (Bethe-Bloch 1932)

$$-\frac{dE}{dx} = \frac{4\pi}{m_e c^2} \cdot \frac{n z^2}{\beta^2} \cdot \left(\frac{e^2}{4\pi\varepsilon_0}\right)^2 \cdot \left[\ln\left(\frac{2m_e c^2 \beta^2}{I \cdot (1-\beta^2)}\right) - \beta^2\right]$$

Charge transportation (Hecht 1932)

$$CCE = \frac{Q}{Q_o} = \frac{\lambda_e}{L}\left[1 - \exp\left(-\frac{(L - x_o)}{\lambda_e}\right)\right] + \frac{\lambda_h}{L}\left[1 - \exp\left(-\frac{x_o}{\lambda_h}\right)\right]$$



CHARGE COLLECTION DISTANCE :

$\delta = \lambda_e + \lambda_h = (\mu_e \tau_e + \mu_h \tau_h)\, E$

: mean drift distance (mean free path of the carrier)

: distance before e$^-$ and h are trapped somewhere… in a defect ?

μ : mobility

τ : lifetime

E : applied electric field

Si (mono) = 100m

Si (amorphe) =10 µm

Diam = 0(100µm)

CCD : MEASUREMENT : Capacity of Diamond to detect MIPs

Charge Collection Distance :

$$d = (\mu_e . \tau_e + \mu_h . \tau_h).E$$

Collected charge:

$$Q = \frac{d}{L} Q_0$$

Pairs / MIP : 3600 / 100 µ diamant)

CCD : Measurement of diamond quality (pCVD ou sCVD)

$$\boxed{d = \frac{N_{\text{électrons}}}{36}}$$

For particle detection :

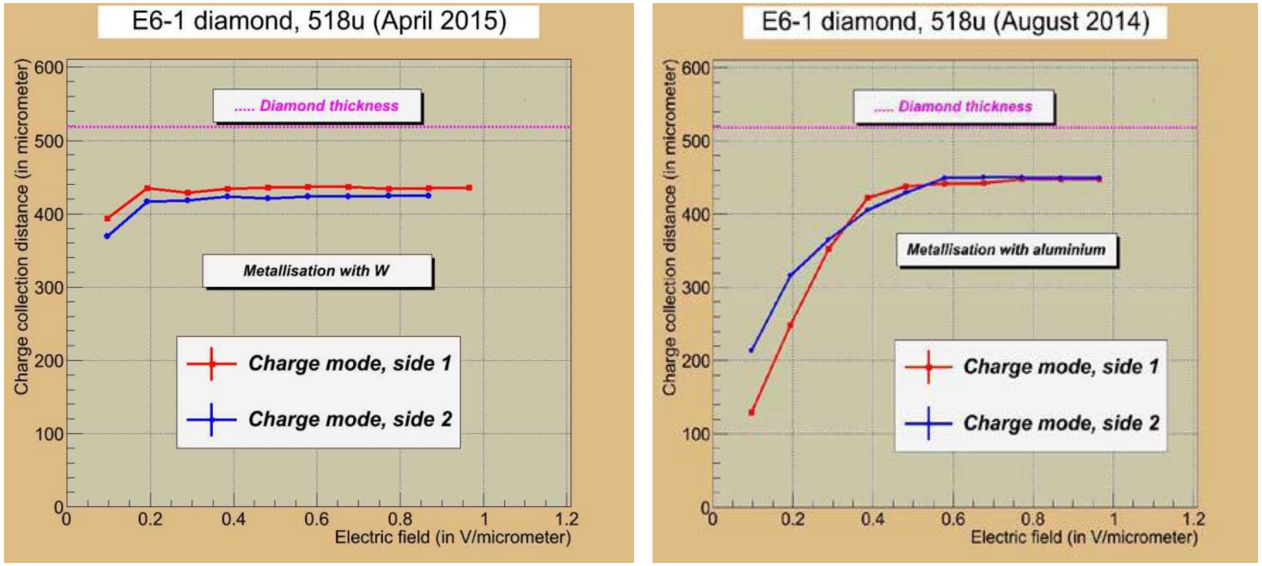
$N_{\text{électrons}}$ (MIP) ~ 10 000

imposes CCD = 277.7 µm

For "transparency"

Thickness ~300 µm

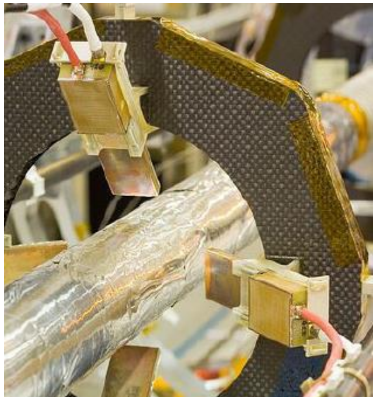
Then CCD ~ 100 % of thickness !!



CCD OK, but different shapes ?

APPLICATIONS IN HEP EXPERIMENTS

Beam Conditions Monitors
Beam Loss Monitors
BaBar
CDF
ATLAS – CMS - LHCb



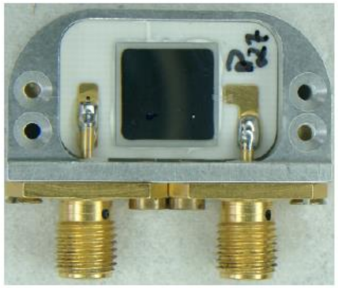
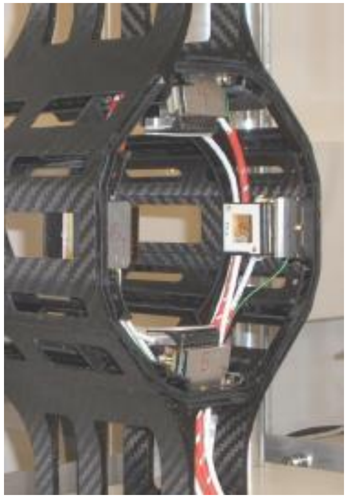
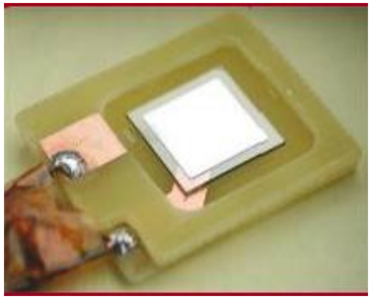


Figure 20: A photograph of the final module used by CMS for its BLM system.

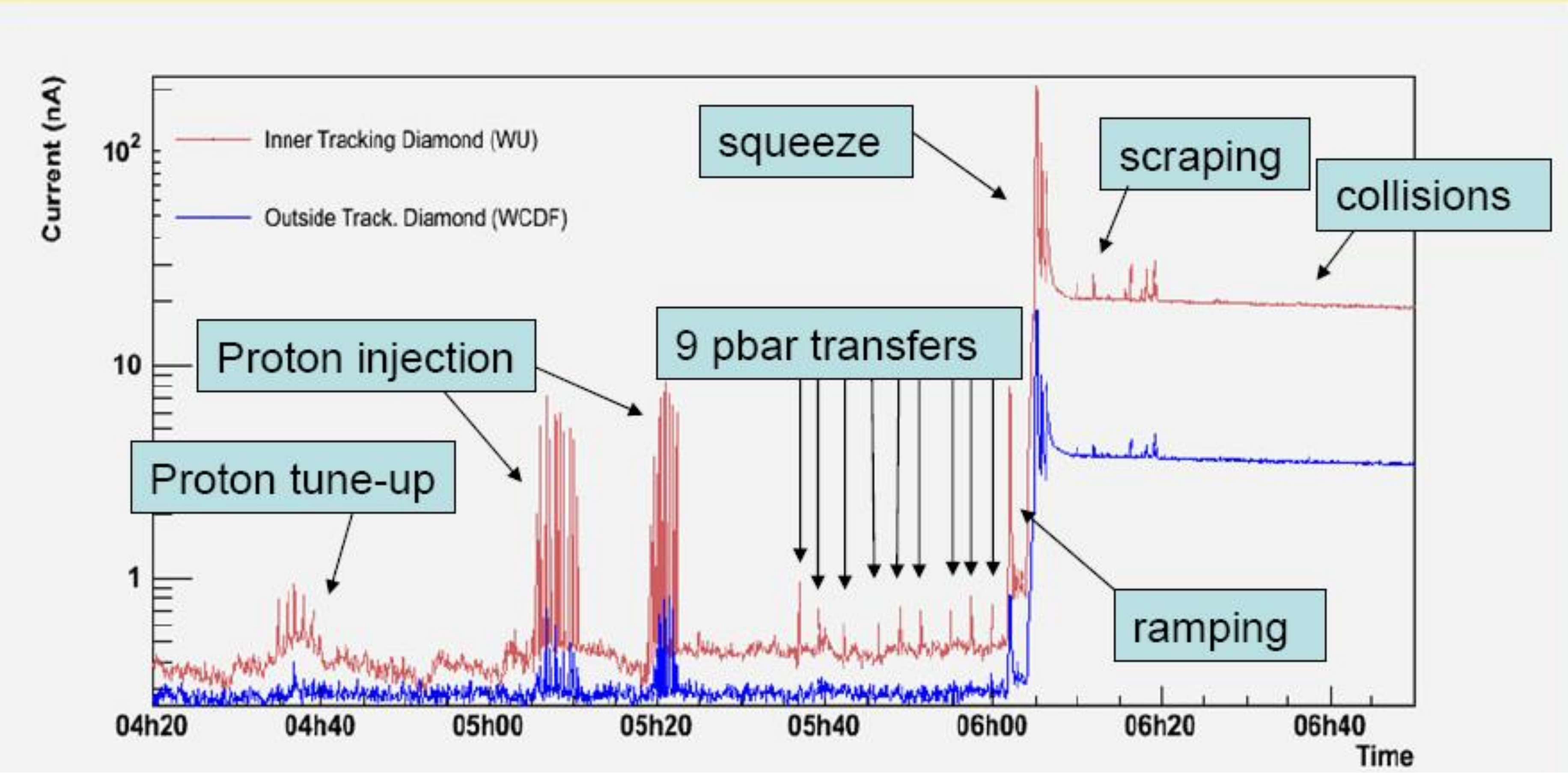


Principe :
Continuous current measurement
with field E ~ 1 V / µ



MAPS vertical integration
(Heat Dissipation)
Since 2007

Semiconductors : Diamonds



CDF at Fermilab

Conclusion : Diamond is OK for beam condition monitor

Idea in 1995 : Diamond is more resistant than Silicon
can it be used for tracking in very difficult conditions ?

LHC Phase II : **Φ at 4 cm ~1.4 ÷ 1.6 10^{16} n_{eq}/cm^2 mainly charged**
Φ at r> 60cm ~1 ÷ 3 10^{14} n_{eq}/cm^2 mainly neutral

Present Tracker Layers radii

L= 8 x 10^{34}

High fluence regime Charged particles predominant

Medium fluence regime Combined Charged and neutral particles

Low fluence regime Neutral particles predominant

r (cm)	ϕ X 10^{13} cm^{-2}	% charged	% neutral
4,30	1509,97	84,5	15,5
7,10	622,76	82,0	18,0
11,00	297,74	78,7	21,3
22,00	117,71	69,2	30,8
32,00	72,39	63,6	36,4
41,00	54,55	59,1	40,9
49,00	43,40	55,8	44,2
58,00	35,47	51,8	48,2
74,50	19,35	26,0	74,0
82,50	17,27	21,9	78,1
90,50	15,78	18,5	81,5
98,50	14,33	13,9	86,1
114,50	12,79	8,4	91,6

Data and simulations agree

Not a clear picture

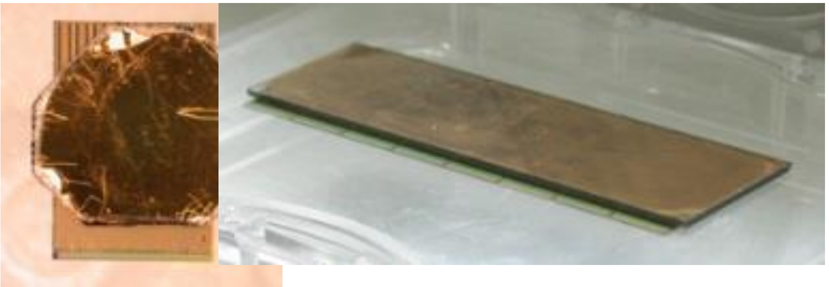
Mixed irradiation
new options/scenario

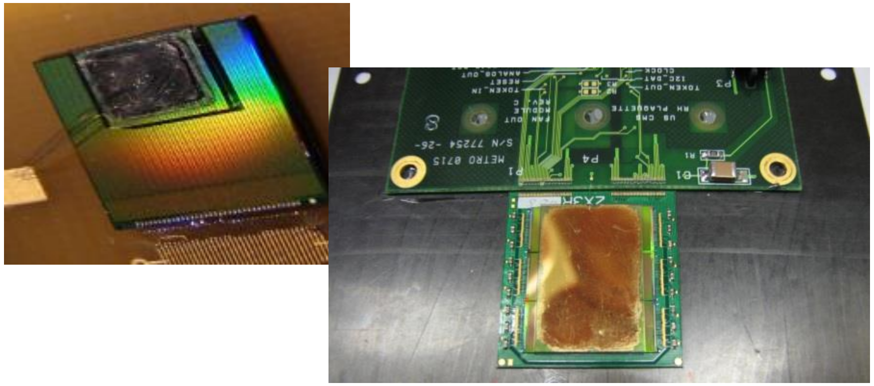
A.Messineo / conveners CERN June 3rd SUWG meeting

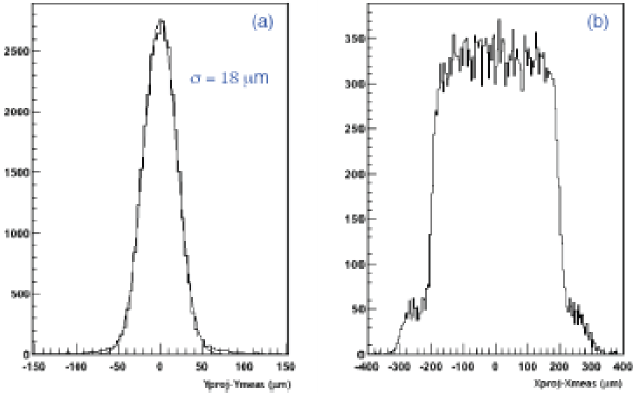
CVD diamond already tested as pixel sensors

By ATLAS (pCVD)

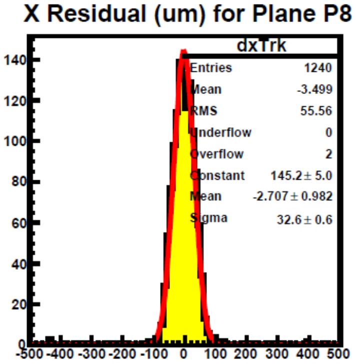
By CMS (sCVD)

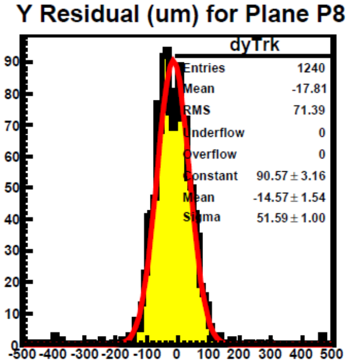






Correct…

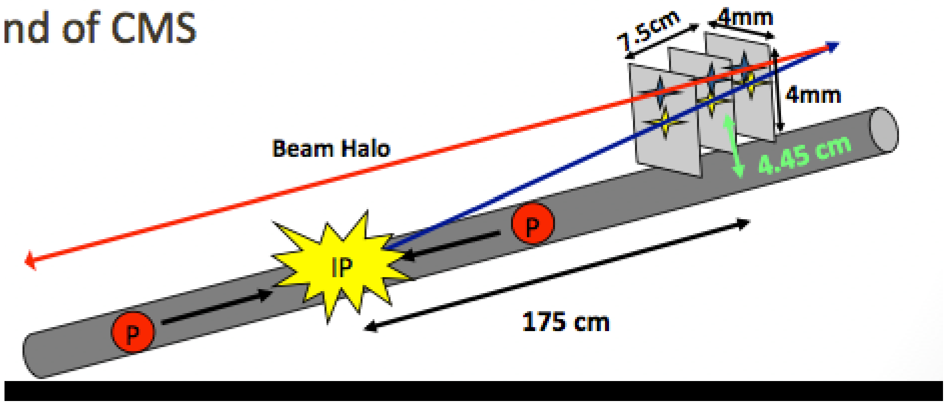


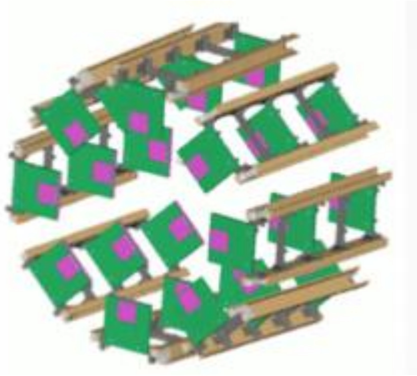


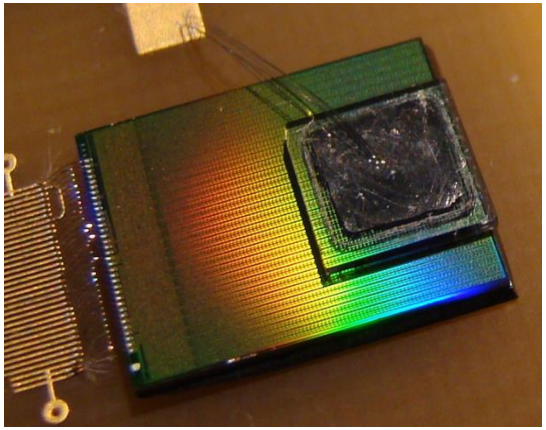
But only once…

Semiconductors : Diamonds

THE CMS PLT





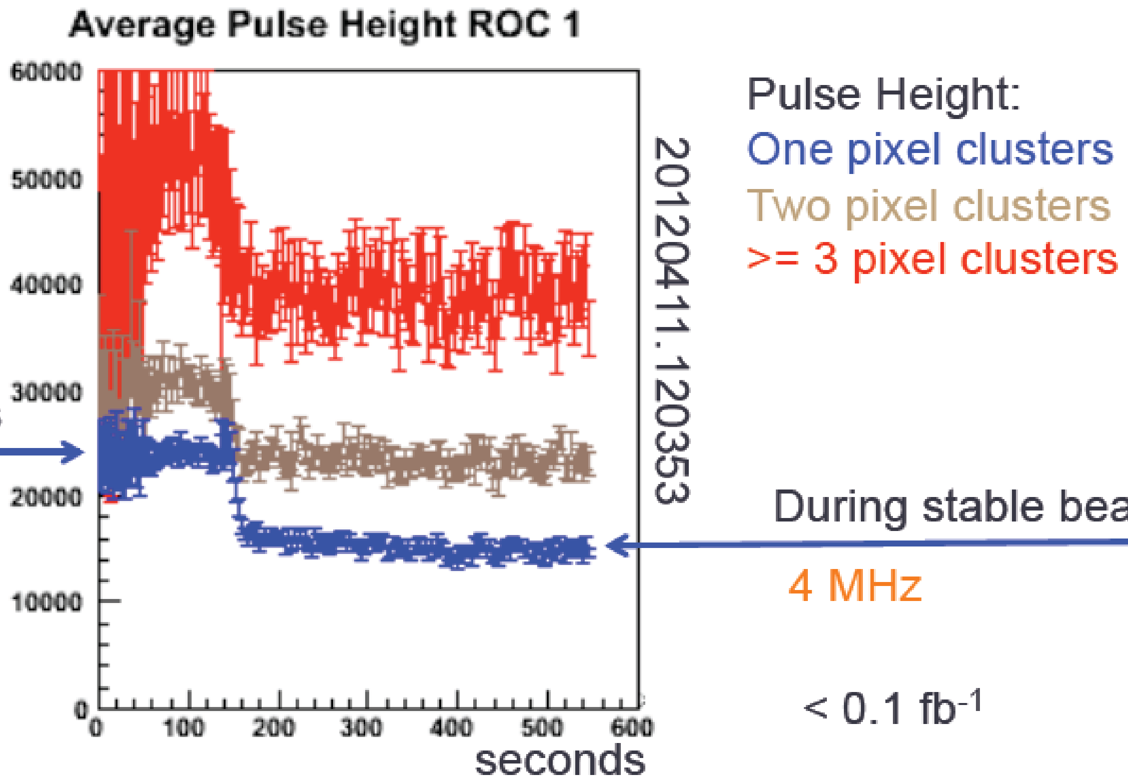


"Normal" way :
- Ordering Monocrystals at the Industry
- Measuring CCD
    - OK : Pixellisation and use
    - Not OK : return to manufacturer

Sucess rate : around 64 %...

Tests beams at Fermilab :
OK – efficiency
- Resolution

Semiconductors : Diamonds



Before stable beams

(100) Hz

During stable beams

4 MHz

< 0.1 fb^{-1}

Failure …

Signal (more than 600 seconds) in absence of particles.

No results

No publication

Silence….

Diamond is not considered in ATLAS and CMS anymore…

Semiconductors : Diamonds

Why ? - Industrial manufacturers (industrial secrets)
- Characterisation test : CCD measurement for a short time.
- lack of information within the community.
- Particle physicists are NOT solid state physicists.

The fundamental problems is : Understanding what makes a ''good'' diamond

MONODIAM-HE Project (ANR-12-BS05-0014)

Work with a laboratory expert in growing diamonds (LSPM – Paris)
- growing **sCVD** and comparison with industrial sCVDs
- Understanding the important parameters
- Improving the quality (for tracking at HE)

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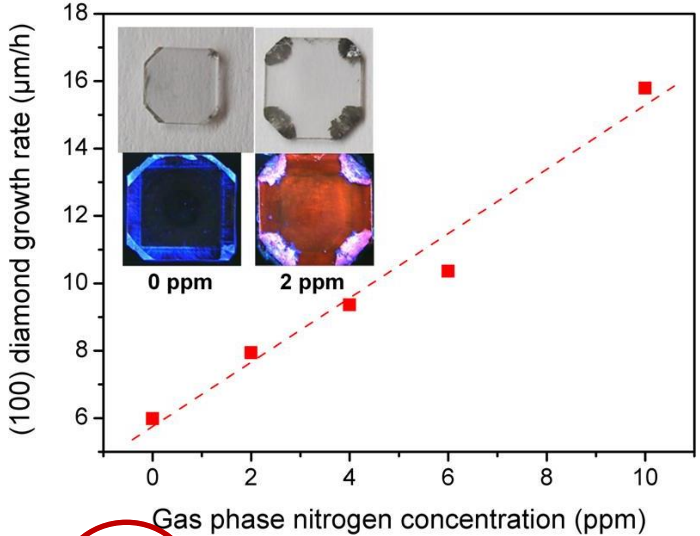
Answering some questions :

- Important parameters for growing diamond (the recipe)
- Important parameters for preparing the diamond
- Important parameters to watch

- **Nitrogen contens**
- **Surface finishing**
- **Metallisation**
- **Long term**

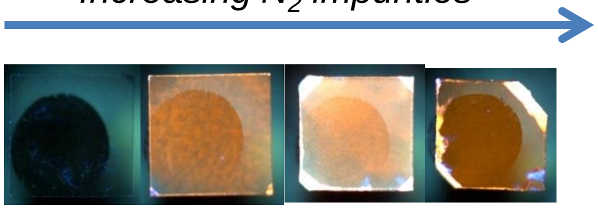
1. Growing conditions : Nitrogen impurities

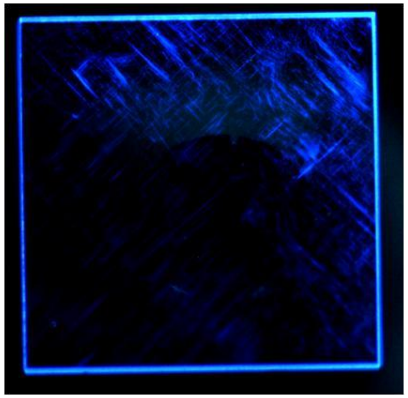
Adding Nitrogen :
- Strong effect on growth rates (up to 100 µ/h)
  - Twinning at the edges
- Limitation of twinning at the surface
- Effect on CCD
  - (incorporation of N2 in the crystal)



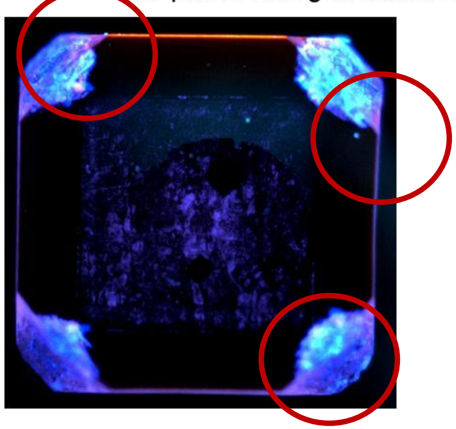
Prototypes made at LSPM with variable N2 contents

Increasing N_2 impurities



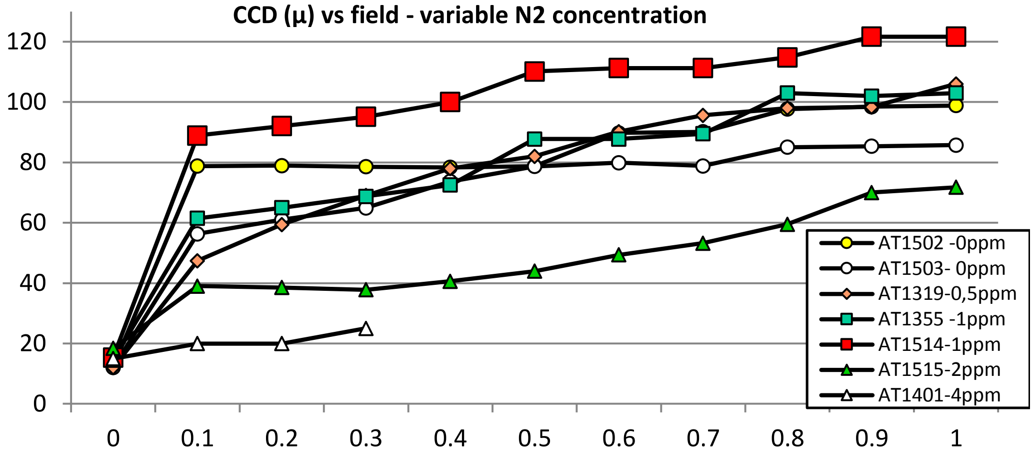


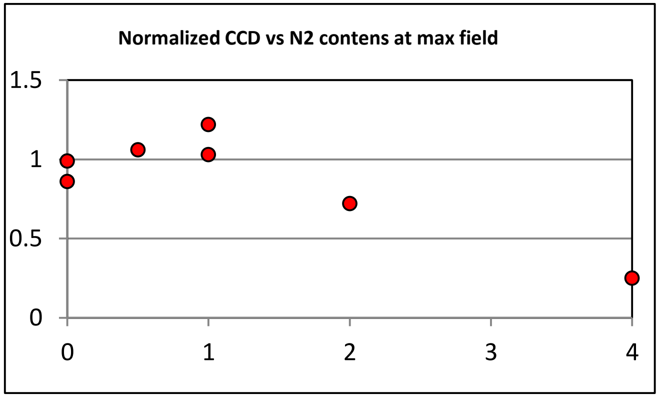
Industrial sCVD
(no N2 ?)



Laboratory sCVD
(2 ppm N2)

thickness	N2 cont.	CCD max.	CCD Norm.	HT Lim
518	0	512	99	500
582	0	499	86	500
500	0,5	530	106	500
430	1	412	103	400
452	1	550	122	500
571	2	410	72	500
518	4	130	25	150





Study made on several prototypes
- same laboratory (LSPM)
- same growing protocol
- same finishing
- same metallisation
- **variable N2 contents**

Optimal : 0 to 1 ppm

2. Surface finishing

Observation :

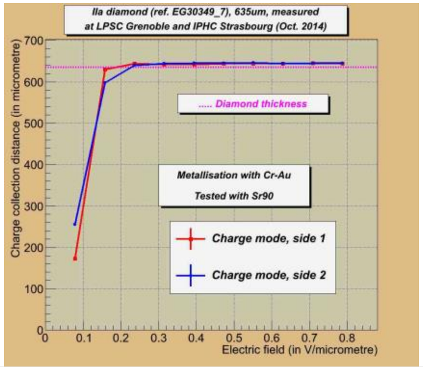
2 possible problems :

- CCD not correct (less than 10000 e^-)
- High voltage limitation (less than 1V/µ)

Possibility ?

CCD related to defects (traps) in the bulk
HV limitation due to surface problems

Evaluation of the quality of a diamond detector :
Measurement of the CCD using MIP (^{90}Sr)
Need : 10 000 e^- : CCD ≈ 280 – 300µ
≈ 100% for a 300µ sCVD

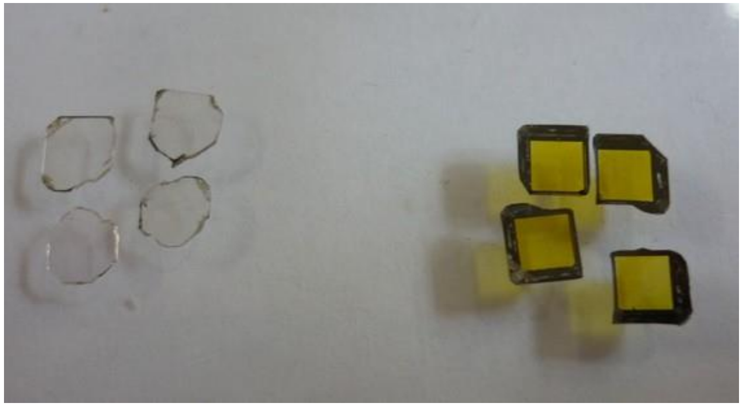


Example :
Industrial sCVD
Thickness : 635 µ
CCD about 600 µ

Use 4 LSPM prototypes sCVD (MM7- 1 to 4)

grown under the same conditions
in the same reactor
at the same time
prepared the same way (same company)
    laser cut to separate the HPHT seed
    precise polishing
Cleaned
metallised (Cr-Au) in laboratory
Measured (CCD and HV limts)

Use 1 industrial (good) sCVD (IND-1)



Reprocessing :

MM7-1 : precise re-polishing by another company (specialized in pCVD)
MM7- 2 : re-etched by RIE at laboratory
MM7- 3 : Terminated by VUV (172nm) in O2 flux
MM7- 4 : untouched . For calibration
IND - 1 : badly re-polished (on purpose)

And metallisation Cr-Au

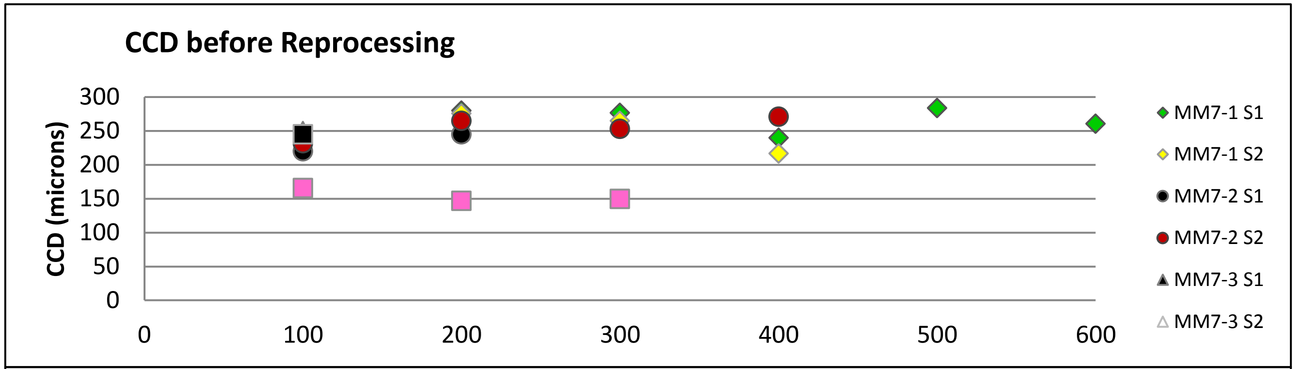
On HV Limits

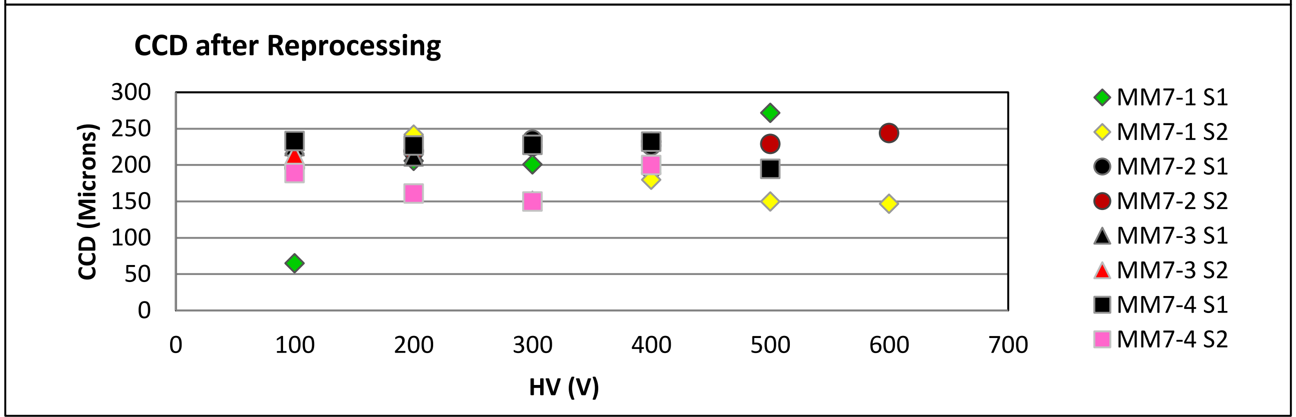
	Before reprocessing		**After reprocessing**		**Observation**
sCVD	Side 1	Side 2	Side 1	Side 2	
IND - 1	500V	600V	500V	300V	degradation
MM7-1	600V	400V	600V	400V	same
MM7-2	200V	400V	400V	500V	**Improvement**
MM7-3	100V	100V	300V	100V	**Little improvement**
MM7-4	500V	350V	500V	400V	same

As good as it is , polishing may not be enough…

Reactive Ion etching
Ozonization
(and probably very agressive cleaning)

Seems to be having an effect on the HV limitation


CCD before Reprocessing


CCD after Reprocessing

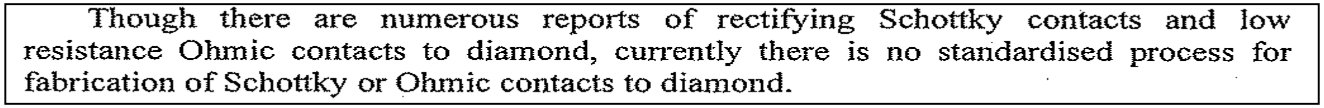
Reprocessing
has a little effect on CCD
(given the measuerment
uncertainties)

Conclusion :

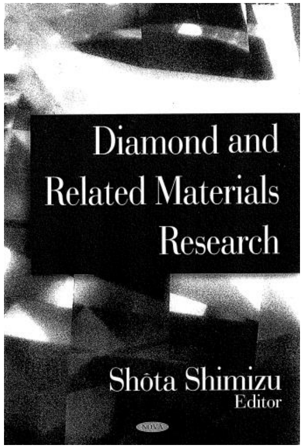
- CCD is related to Bulk quality
- HV limitation is related to surface quality and **may be improved.**

3. Metallisation

Metallisation needed for contacts (wire bonding or bump-bonding)
Early prototypes showed a Schottky Diode Behaviour
Extensive researches on diamond contacts
see, for example «



Though there are numerous reports of rectifying Schottky contacts and low resistance Ohmic contacts to diamond, currently there is no standardised process for fabrication of Schottky or Ohmic contacts to diamond.



Use Two (industrial) reference detectors
metalised Cr-Au
Tested on different benches

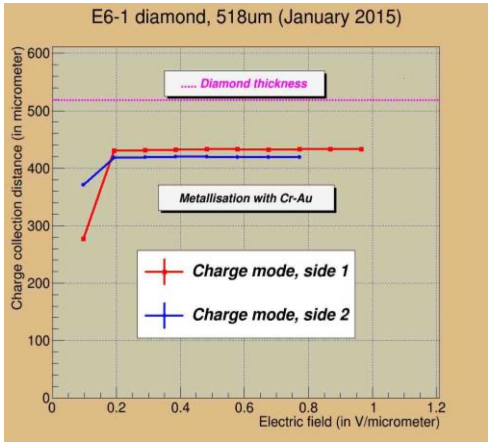
Cleaned
Metallisation (pulverisation)
Tests : **Cr- Au**
W
Cu
Al
In (Cu-In)

At LSPC-Grenoble

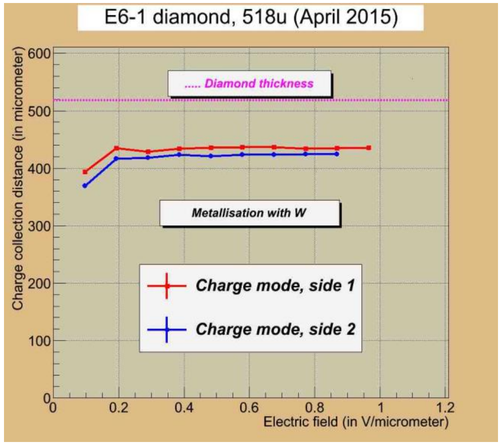
Metal deposition by
by microwave plasma-assisted sputtering
Cleaning by
2 steps of plasma-assisted cleaning

At Icube - Strasbourg

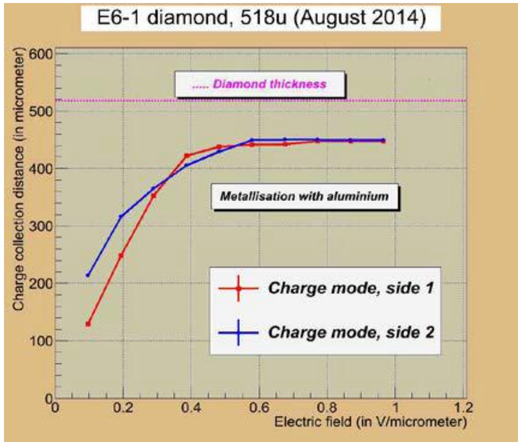
Metal deposition by
Vacuum evaporation
Cleaning by
Hot H_2S0_4 – KNO_3



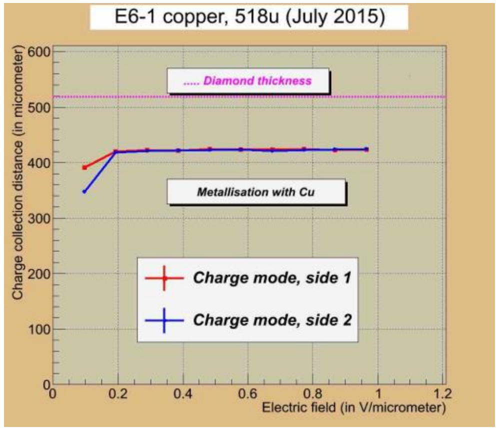
Cr-Au



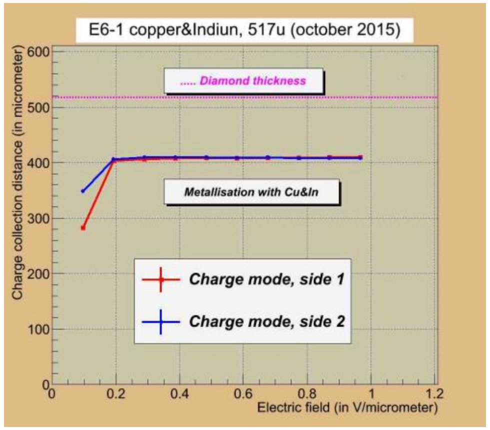
W



Al

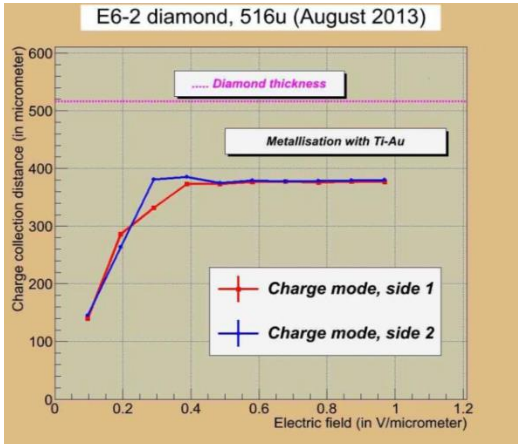


Cu

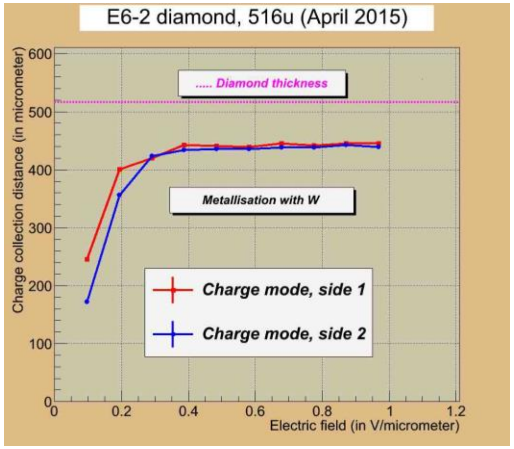


Cu-In

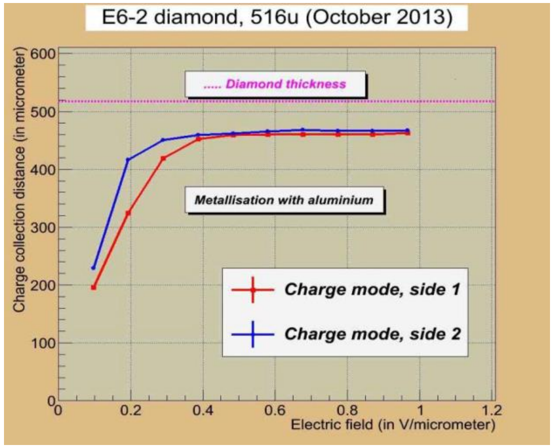
Cr-Au :	HT limits	500V / 400V
	CCD	440 µm / 420 µm
W :	HT limits	500V / 450V
	CCD	440 µm / 420 µm
Al :	HT limits	500V / 500V
	CCD	440 µm / 440 µm
Cu :	HT limits	500V / 500V
	CCD	425 µm / 425 µm
Cu-In :	HT limits	500V / 500V
	CCD	410 µm / 410 µm



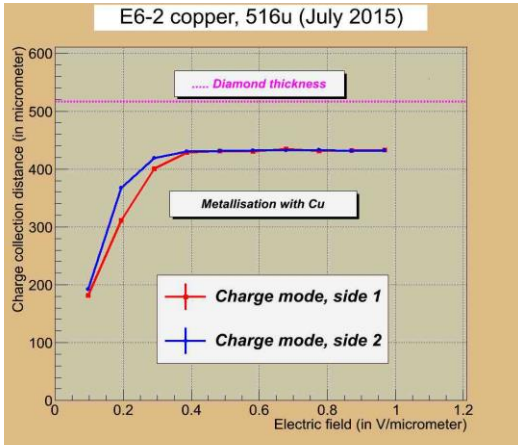
Ti-Au



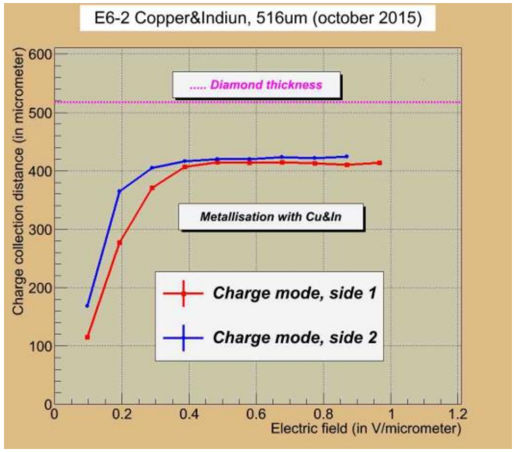
W



Al



Cu



Cu-In

Ti-Au :	HT limit	500V / 500V
	CCD	380 µm / 380 µm
W :	HT limit	500V / 500V
	CCD	425 µm / 425 µm
Al :	HT limit	500V / 500V
	CCD	460 µm / 460 µm
Cu :	HT limit	500V / 500V
	CCD	410 µm / 410 µm
Cu-In :	HT limit	500V / 450V
	CCD	410 µm / 420 µm

Various metallisations have **very little effect** on CCD

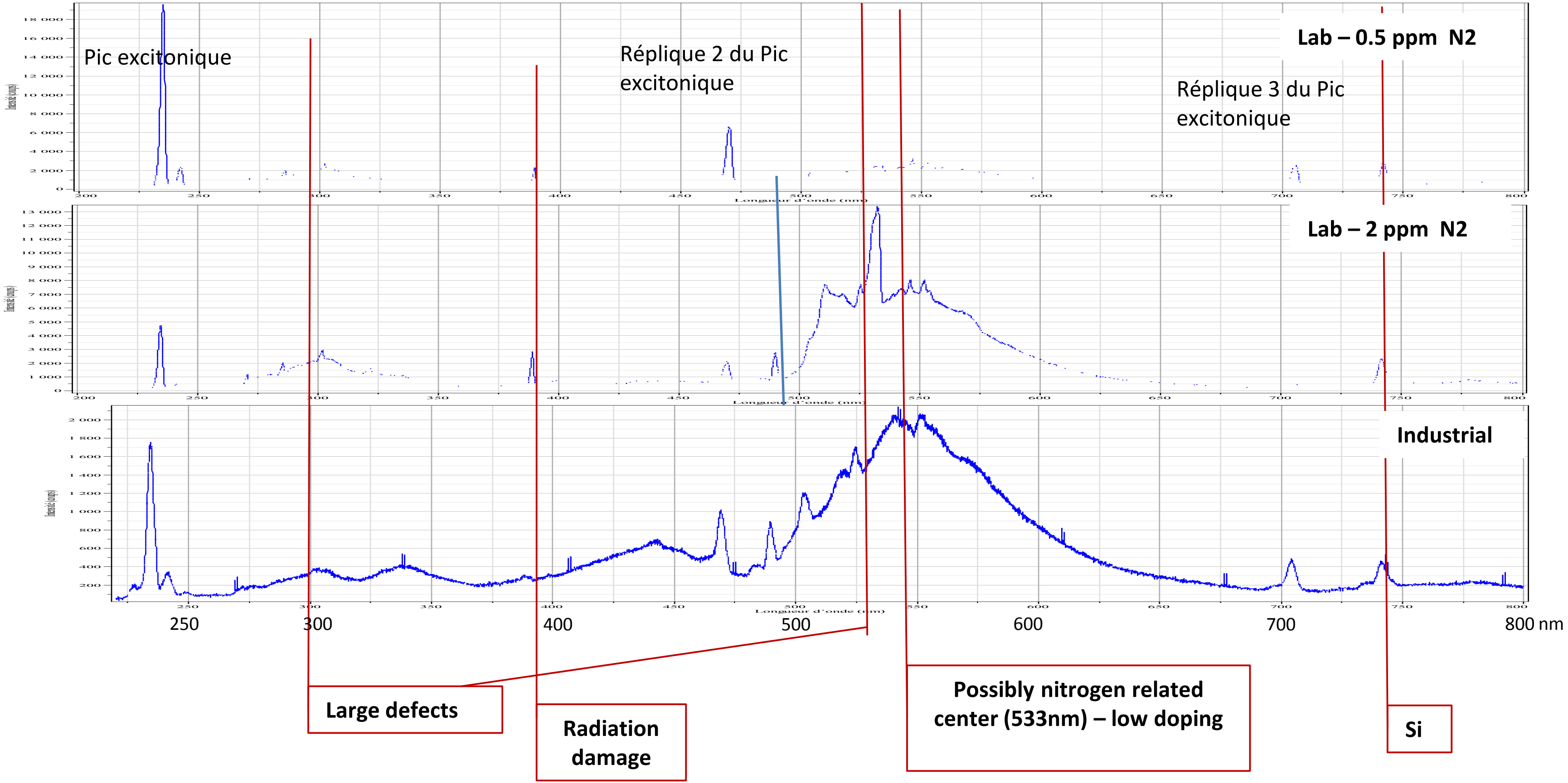
(Schottky element already stabilized by cleaning ?)

HT limit related to **surface quality**

4. Bulk Studies

By Cathodo-luminescence at low temperature
(electron beam 10kV, 7nA – T=110°K)

Lab – 0.5 ppm N2 : Limit HT, limited CCD
Lab – 2 ppm N2 : HT OK, CCD OK
Ind - ? : HT OK, CCD OK



Pic excitonique

Réplique 2 du Pic excitonique

Réplique 3 du Pic excitonique

Lab – 0.5 ppm N2

Lab – 2 ppm N2

Industrial

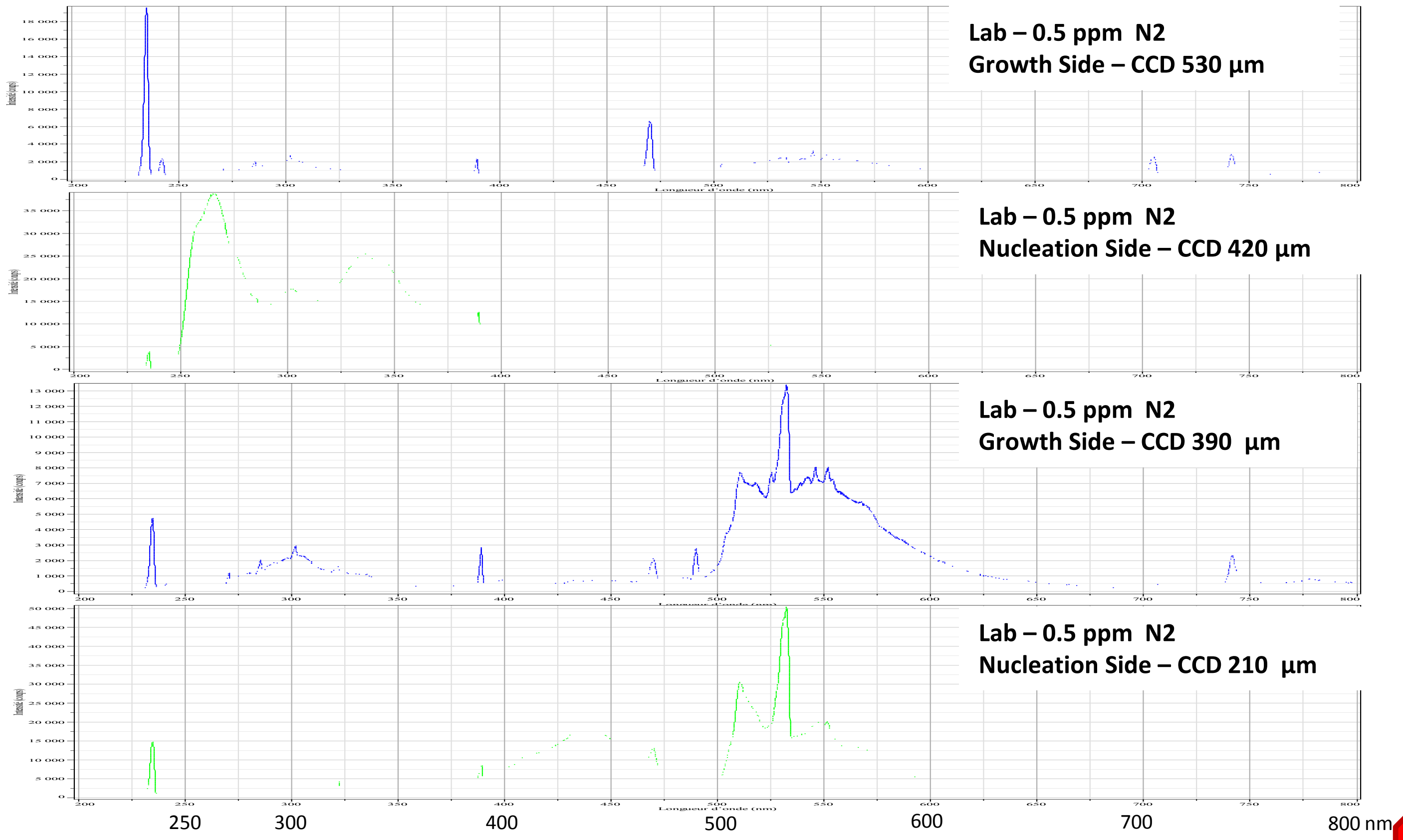
Large defects

Radiation damage

Possibly nitrogen related center (533nm) – low doping

Si

One can see clearly the presence of Nitrogen
There are extended defects in all diamonds
Test under source (74 MBq) induces damages: Radiation hardness ?

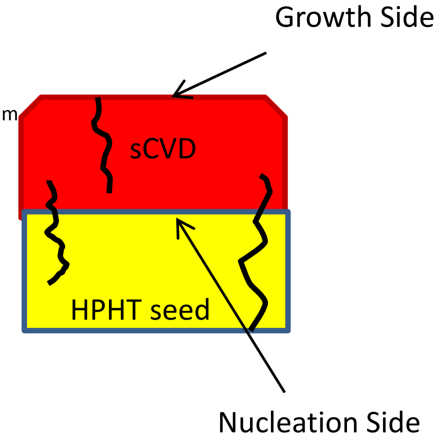


Lab – 0.5 ppm N2
Growth Side – CCD 530 µm

Lab – 0.5 ppm N2
Nucleation Side – CCD 420 µm

Lab – 0.5 ppm N2
Growth Side – CCD 390 µm

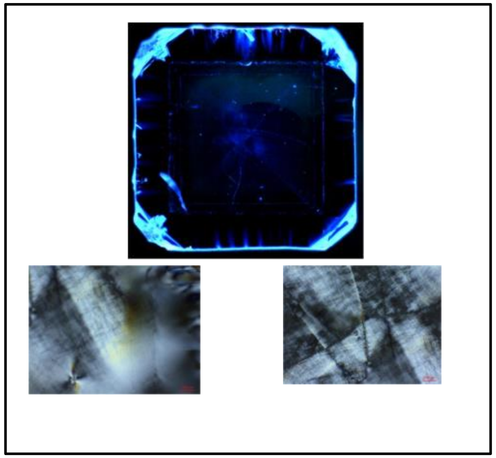
Lab – 0.5 ppm N2
Nucleation Side – CCD 210 µm



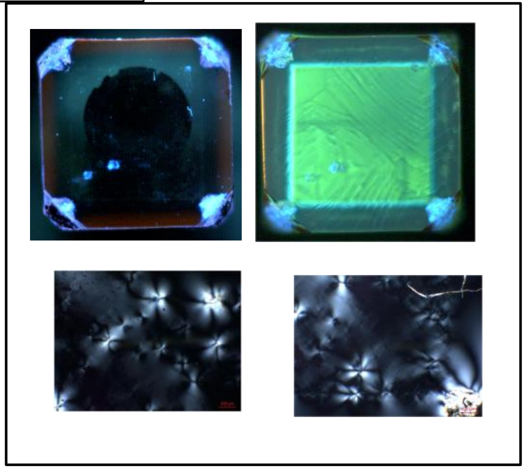
More extended defects on the nucleation side (HPHT mirror effect)
Even the sCVD are asymetrics (known for the pCVDs)
(with consequences on the CCD)

5. Bulk and surface Studies

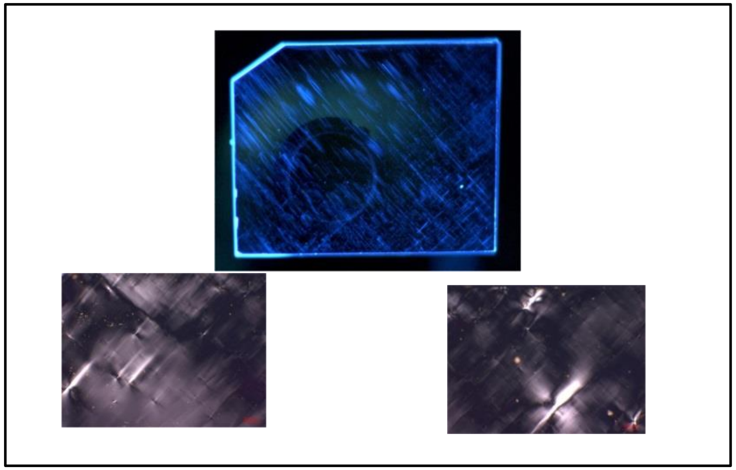
By Images (Fluorescence / polarised light / X-ray Tomography)



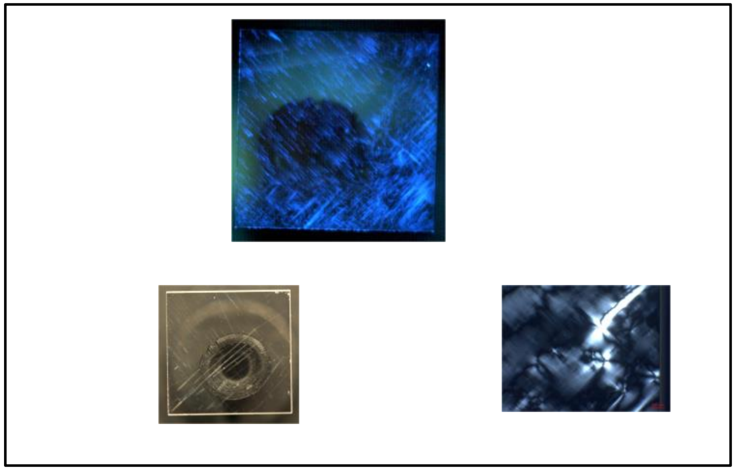
LSPM – 0 ppm N2
results on CCD OK



LSPM – 1 ppm N2
results on CCD OK



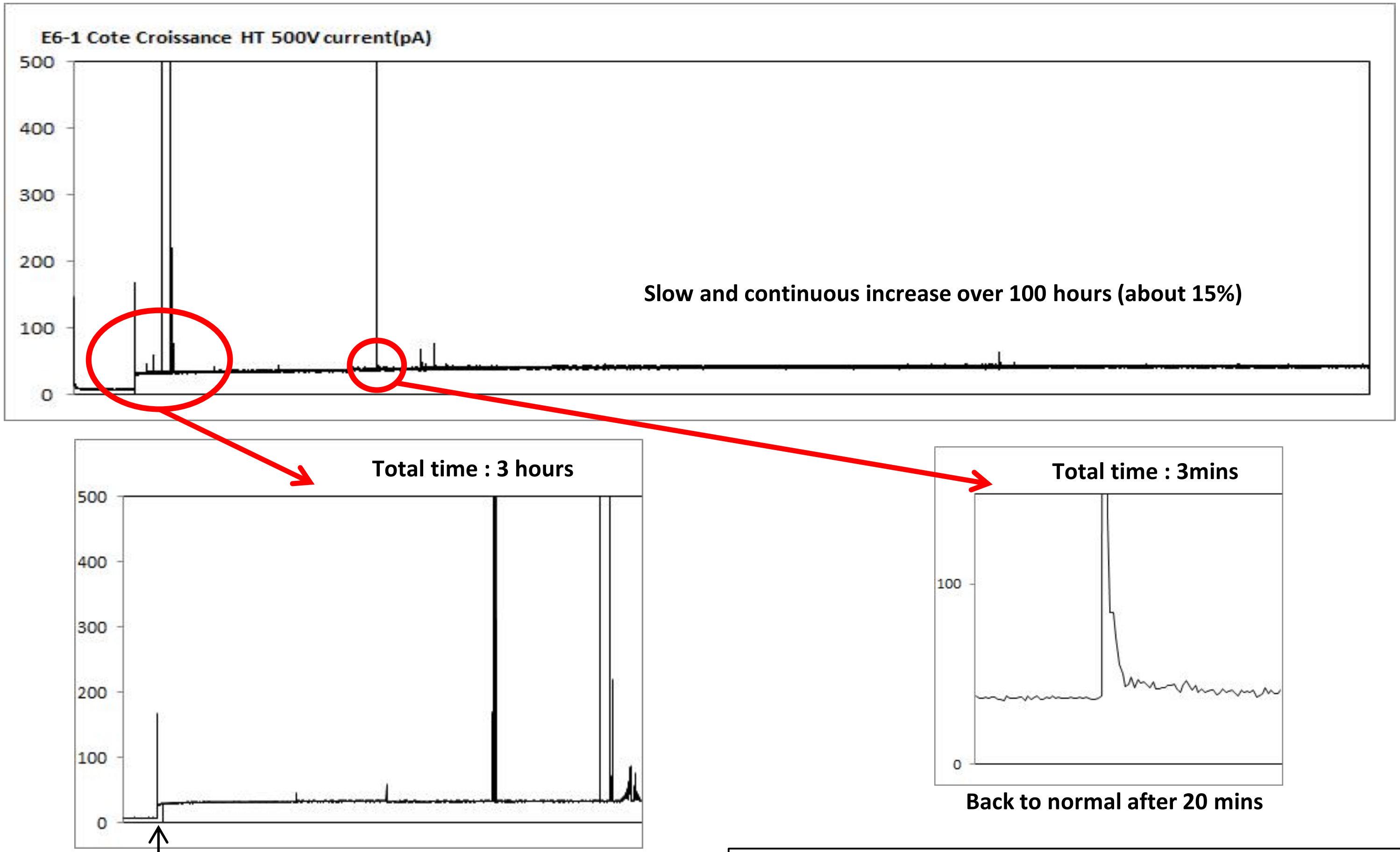
Industry
results on CCD very good



All sCVD show defects
- deep dislocations
- bad polishing
- N2 incorporation
- …

6. Long term effects – ‘’Polarisation’’ ?

Long term measurement leakage current at max field (≈1 V/µm)



Slow and continuous increase over 100 hours (about 15%)

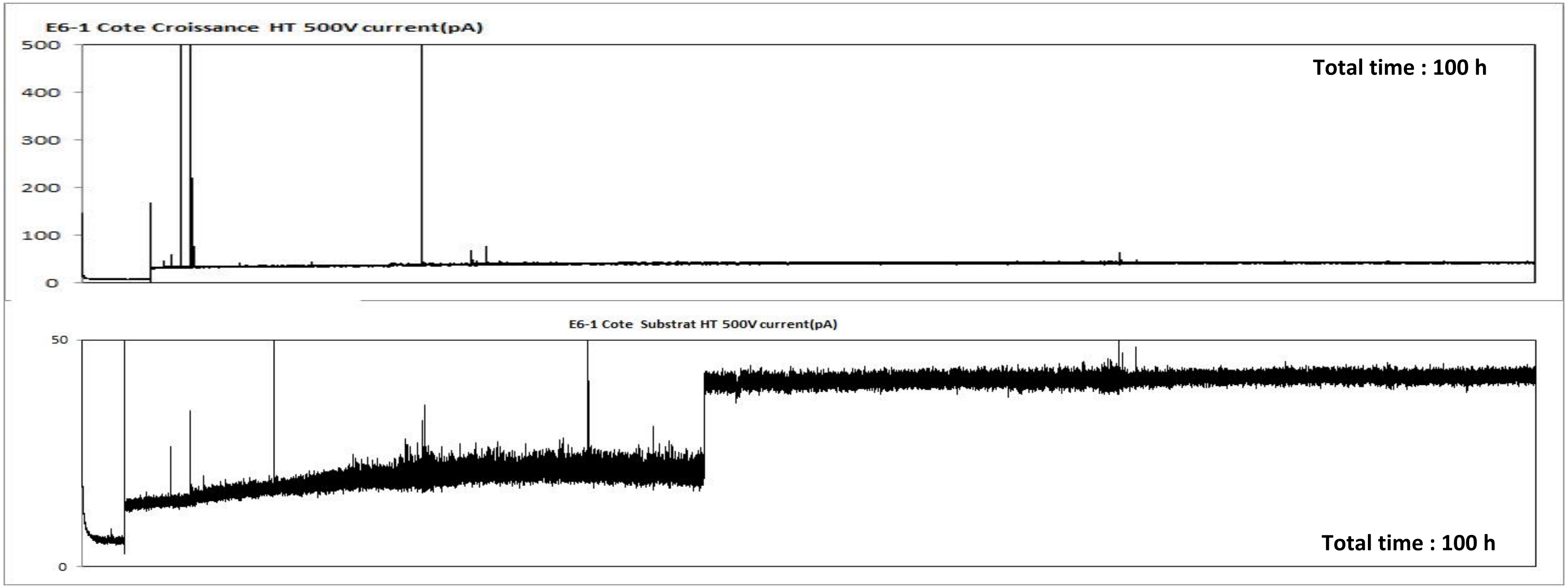
Total time : 3 hours

Total time : 3mins

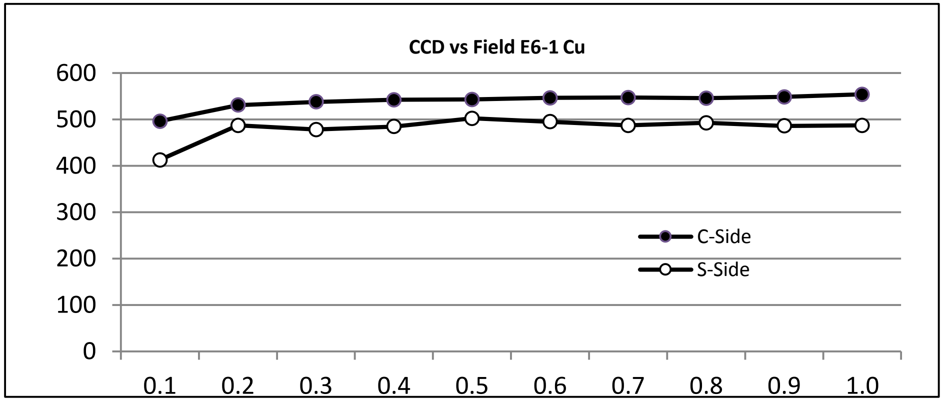
Back to normal after 20 mins

74 MBq source

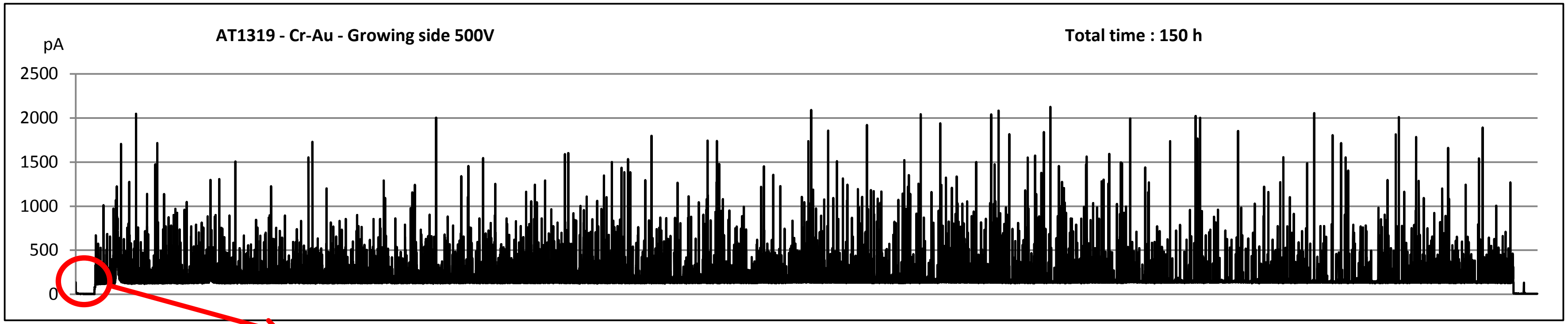
Clear source effect
‘’ slow ‘’ discharges (charging – discharging of traps)



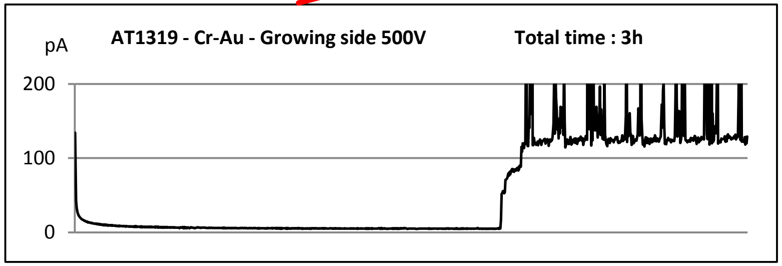
For a ''good'' (industrial) sCVD



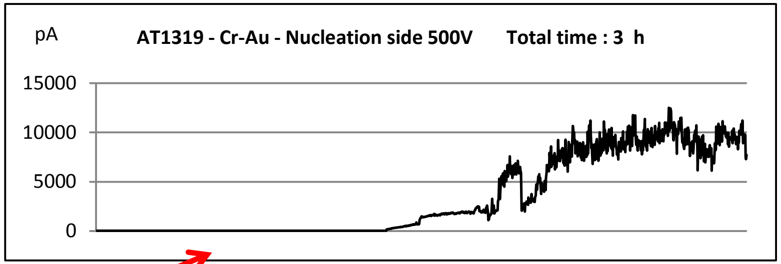
Difference between sides
Lower CCD on the nucleation side
More defects (traps) on the nucleation side



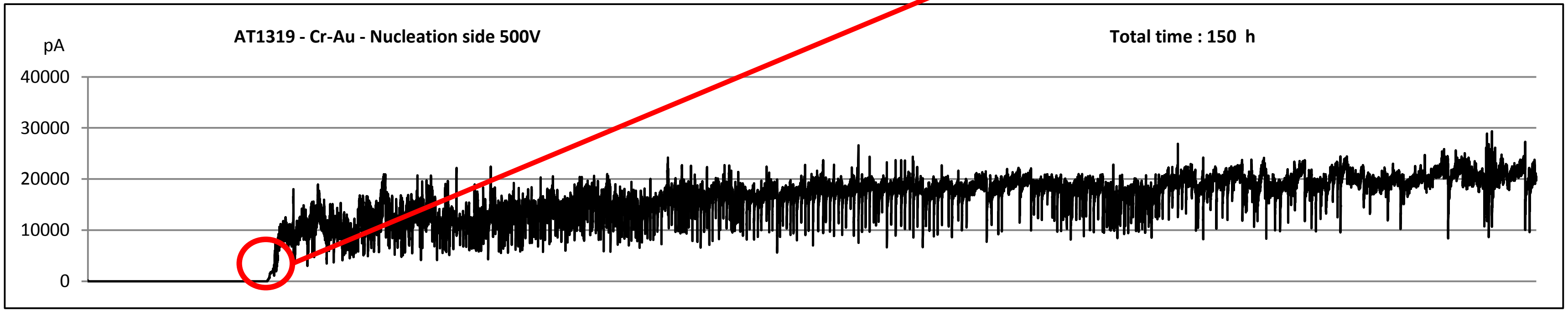
AT1319 - Cr-Au - Growing side 500V **Total time : 150 h**



AT1319 - Cr-Au - Growing side 500V **Total time : 3h**



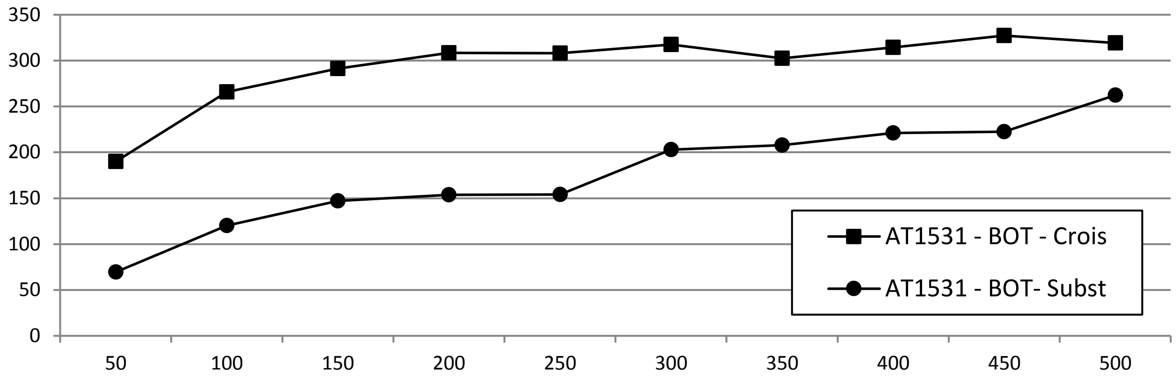
AT1319 - Cr-Au - Nucleation side 500V **Total time : 3 h**



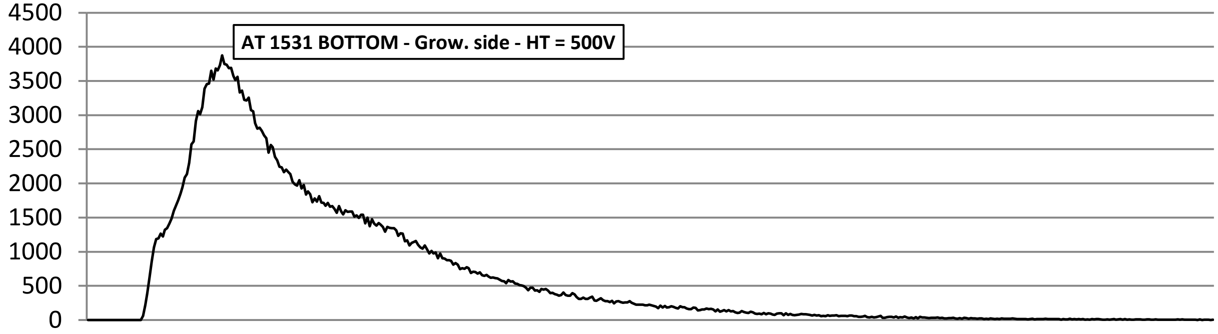
AT1319 - Cr-Au - Nucleation side 500V **Total time : 150 h**

Strong difference between sides
More defects (traps) on the nucleation side
Could be a way to evaluate the defects rate ??

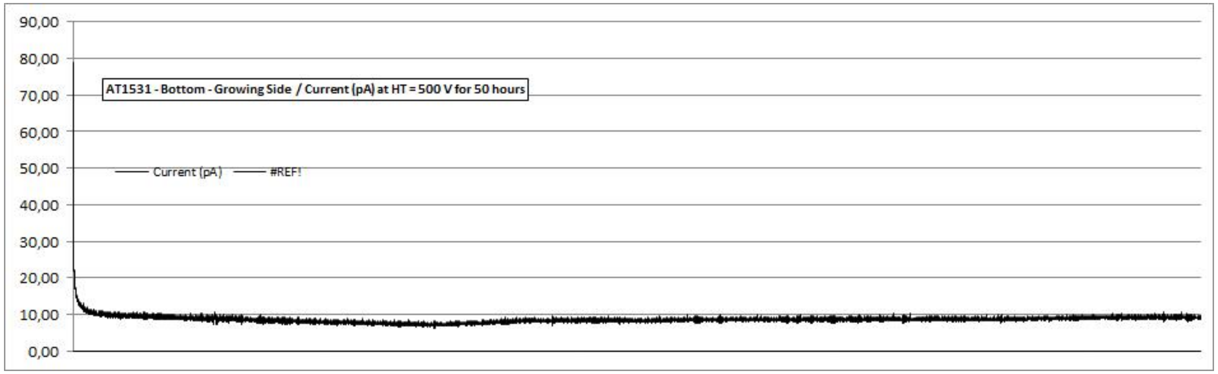
One GOOD DIAMOND :



CCD vs HV



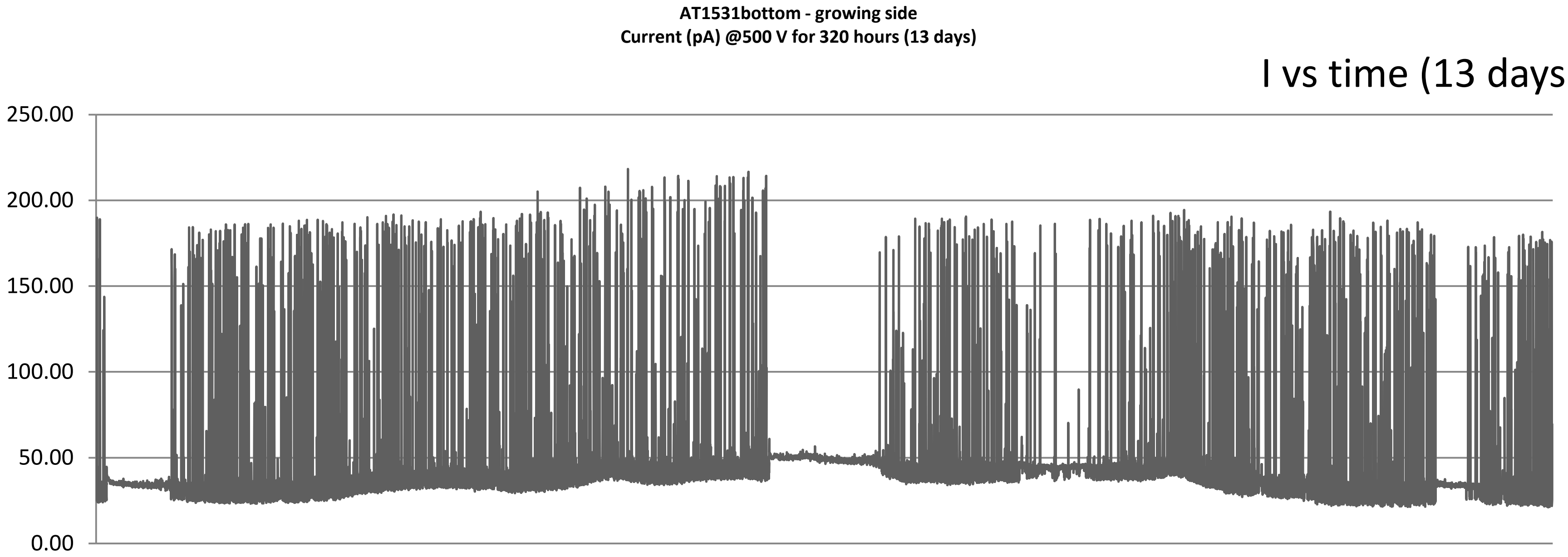
MIP detection



Good stability (50h)

BUT ….

I vs time (13 days)



Current peaks around 200 pA (not destructive, but annoying)

- Stabilisation after some minutes.
- Stable from 20 minutes to 31 hours
  from 148 and 172 hours

EXPLANATION ???

(148 to 172 h : Week end)

Conclusion and prospectives (?)

We have started to adress fundamental problems :

Diamond bulk effects

- impurities (Nitrogen – Boron)
- bulk defects importance
  - defects rate have to be understood, and under control
  - **some ideas exist :**
    - **differential growing, immediate anealing, disorientation)**

Diamond surface effects

- surface finishing
- metallisation
  - **problem considered as solved (?)**

The main lesson :

True :

Developpements in the Research Community may lead to Industrial Developpements.

Wrong :

Objects developped in the Industrial World can be easily used in the Research Community.