

Laser graphitization for polarization of diamond sensors

F.Fabbrizzi, S.Lagomarsino, L.Nunziati, G.Parrini, S.Sciortino

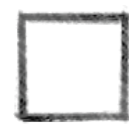
Università di Firenze e INFN Firenze

A. Scorzoni

Università di Perugia e INFN Perugia

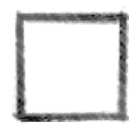
..... one step of the CHIPSODIA project (2011/2012, INFN-IIT coll.)

“New perspectives for the Silicon-On-Diamond material”, RD09



A quick look at CHIPSODIA

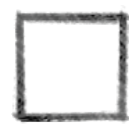
It stems with the novel bonding technique (Appl. Phys. Lett. **96**, 031901 (2010)) of silicon and diamond dies. The bonding is performed by lightening the interface of Si, D dies with 20 ps, $\lambda=355$ nm laser pulses. The surfaces are well clean and polished ($R_a < 10\text{nm}$)



A quick look at CHIPSODIA

It stems with the novel bonding technique (Appl. Phys. Lett. **96**, 031901 (2010)) of silicon and diamond dies. The bonding is performed by lightening the interface of Si, D dies with 20 ps, $\lambda=355$ nm laser pulses. The surfaces are well clean and polished ($R_a < 10$ nm)

Material quality does not influence the bonding: poly vs single crystal is a matter of convenience (price). Most of our experience has been made on "Diamond Detector Ltd" detector grade quality samples.

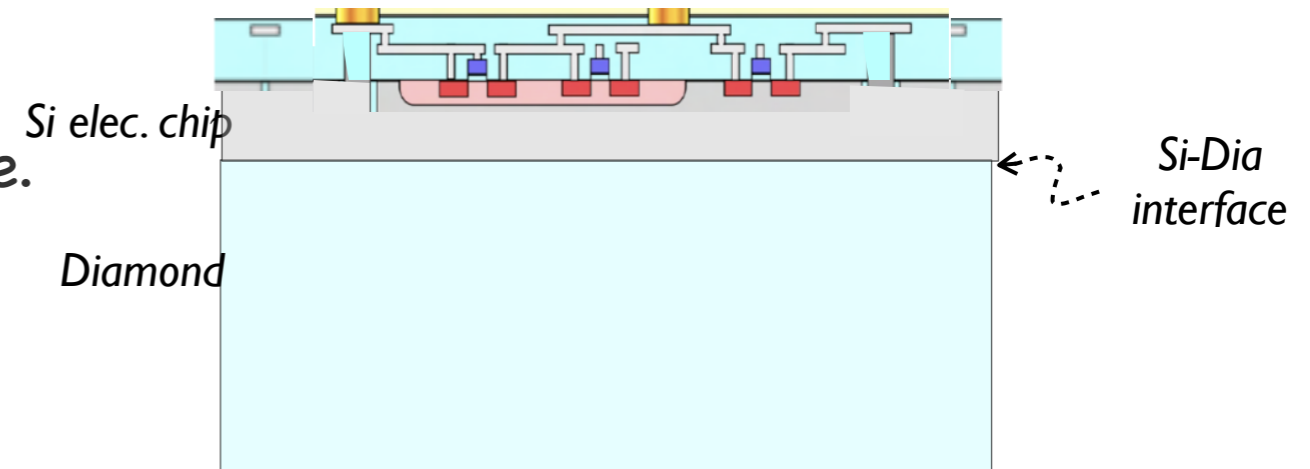


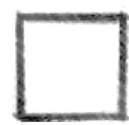
A quick look at CHIPSODIA

It stems with the novel bonding technique (Appl. Phys. Lett. **96**, 031901 (2010)) of silicon and diamond dies. The bonding is performed by lightening the interface of Si, D dies with 20 ps, $\lambda=355$ nm laser pulses. The surfaces are well clean and polished ($R_a < 10$ nm)

Material quality does not influence the bonding: poly vs single crystal is a matter of convenience (price). Most of our experience has been made on "Diamond Detector Ltd" detector grade quality samples.

The goal of CHIPSODIA is to integrate a silicon electronics die with a diamond die to form a monolithic rugged device. This can be both a particle detector (pixel) and a biological device.





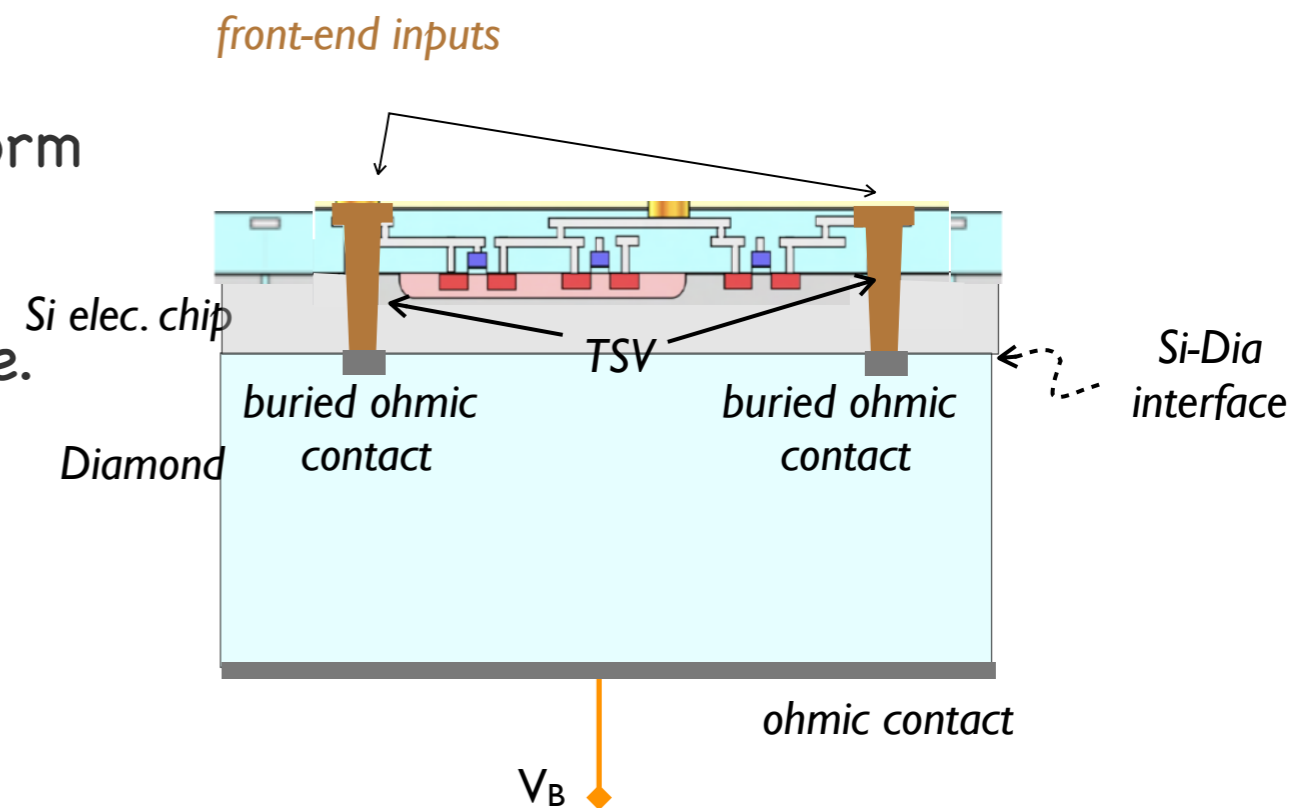
A quick look at CHIPSODIA

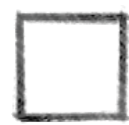
It stems with the novel bonding technique (Appl. Phys. Lett. **96**, 031901 (2010)) of silicon and diamond dies. The bonding is performed by lightening the interface of Si, D dies with 20 ps, $\lambda=355$ nm laser pulses. The surfaces are well clean and polished ($R_a < 10\text{nm}$)

Material quality does not influence the bonding: poly vs single crystal is a matter of convenience (price). Most of our experience has been made on "Diamond Detector Ltd" detector grade quality samples.

The goal of CHIPSODIA is to integrate a silicon electronics die with a diamond die to form a monolithic rugged device. This can be both a particle detector (pixel) and a biological device.

Both Si and Diamond require post bonding process : TSV (Si), buried contacts (D),...
After that the pixel device is ready..... to detect the MIP crossing.





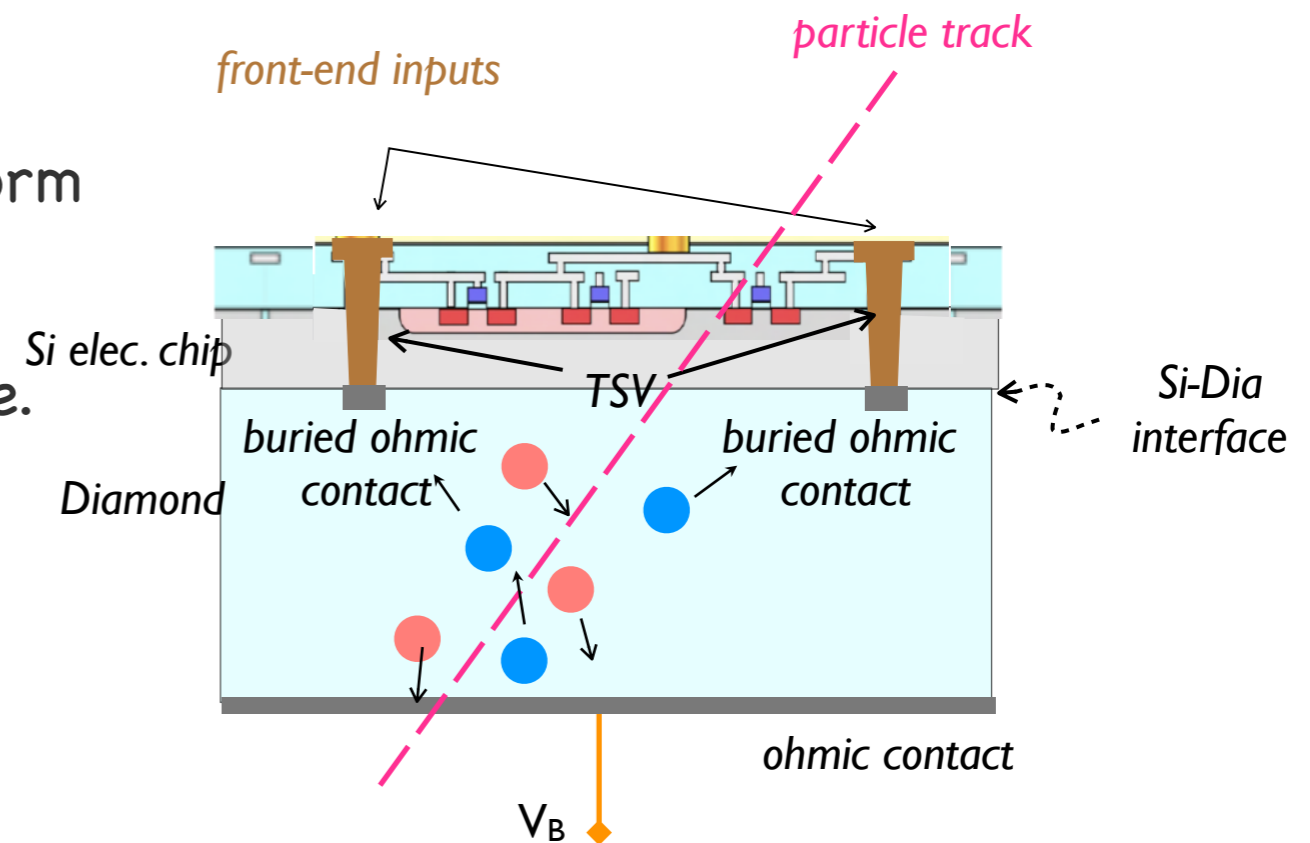
A quick look at CHIPSODIA

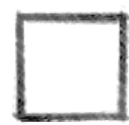
It stems with the novel bonding technique (Appl. Phys. Lett. **96**, 031901 (2010)) of silicon and diamond dies. The bonding is performed by lightening the interface of Si, D dies with 20 ps, $\lambda=355$ nm laser pulses. The surfaces are well clean and polished ($R_a < 10$ nm)

Material quality does not influence the bonding: poly vs single crystal is a matter of convenience (price). Most of our experience has been made on "Diamond Detector Ltd" detector grade quality samples.

The goal of CHIPSODIA is to integrate a silicon electronics die with a diamond die to form a monolithic rugged device. This can be both a particle detector (pixel) and a biological device.

Both Si and Diamond require post bonding process : TSV (Si), buried contacts (D),...
After that the pixel device is ready..... to detect the MIP crossing.





A quick look at CHIPSODIA

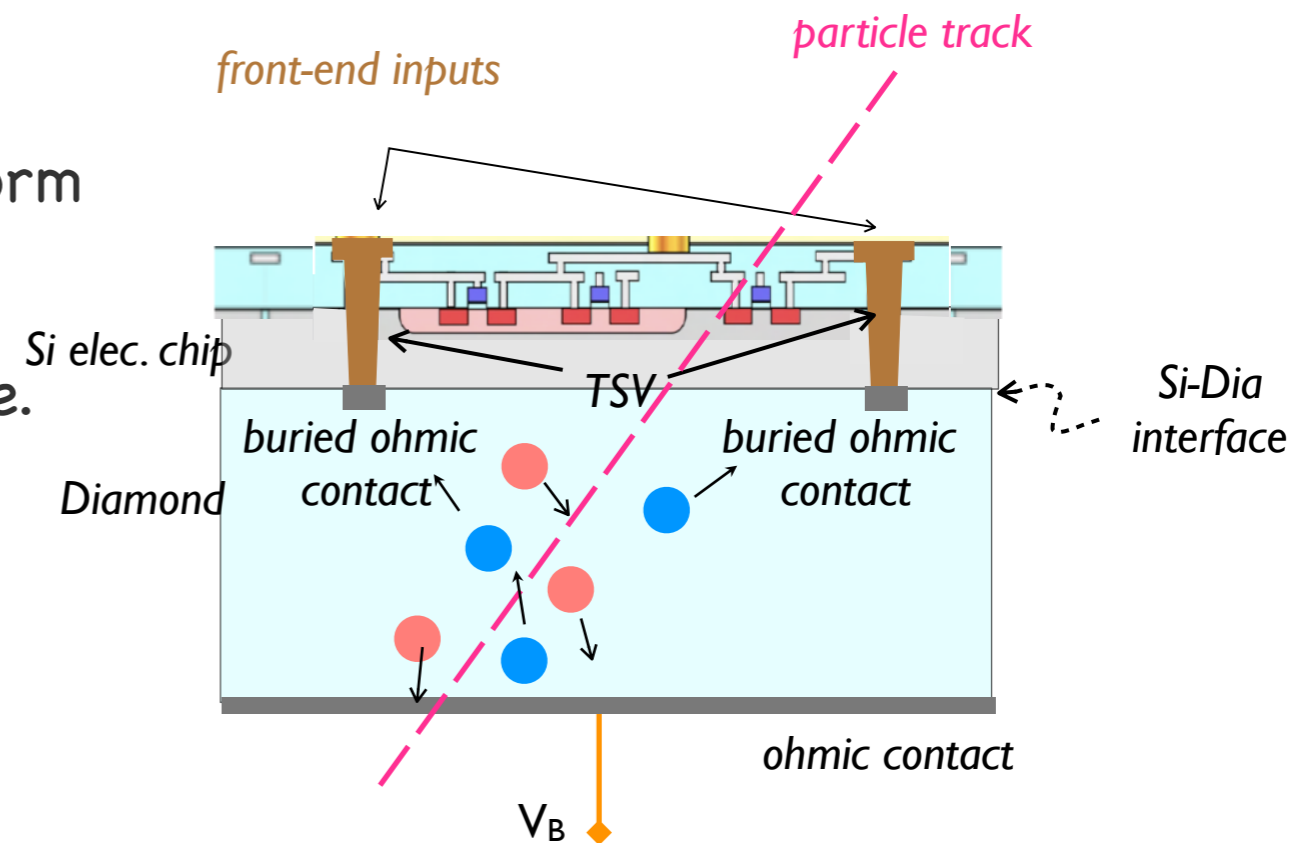
It stems with the novel bonding technique (Appl. Phys. Lett. **96**, 031901 (2010)) of silicon and diamond dies. The bonding is performed by lightening the interface of Si, D dies with 20 ps, $\lambda=355$ nm laser pulses. The surfaces are well clean and polished ($R_a < 10$ nm)

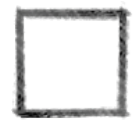
Material quality does not influence the bonding: poly vs single crystal is a matter of convenience (price). Most of our experience has been made on "Diamond Detector Ltd" detector grade quality samples.

The goal of CHIPSODIA is to integrate a silicon electronics die with a diamond die to form a monolithic rugged device. This can be both a particle detector (pixel) and a biological device.

Both Si and Diamond require post bonding process : TSV (Si), buried contacts (D),... After that the pixel device is ready..... to detect the MIP crossing.

This talk concerns the experimentation on the technique we want to use for the (buried) diamond contacts.





Laser graphitization on diamond

The Diamond engraving and graphitization by means of a laser beam is a well known fact!

Diamond Inscription , Romance and Practicality

Wavelengths either below or above the absorption threshold of Diamond (≈ 225 nm) can be used.



(GIA Gem Trade Laboratory)

Long wavelengths allow to penetrate the Diamond and to graphitize either dot shaped or column shaped volumes.

Graphite is a conductor ($\approx 1 \div 500$ m Ω .cm) and it can be the solution of our buried contact issue.

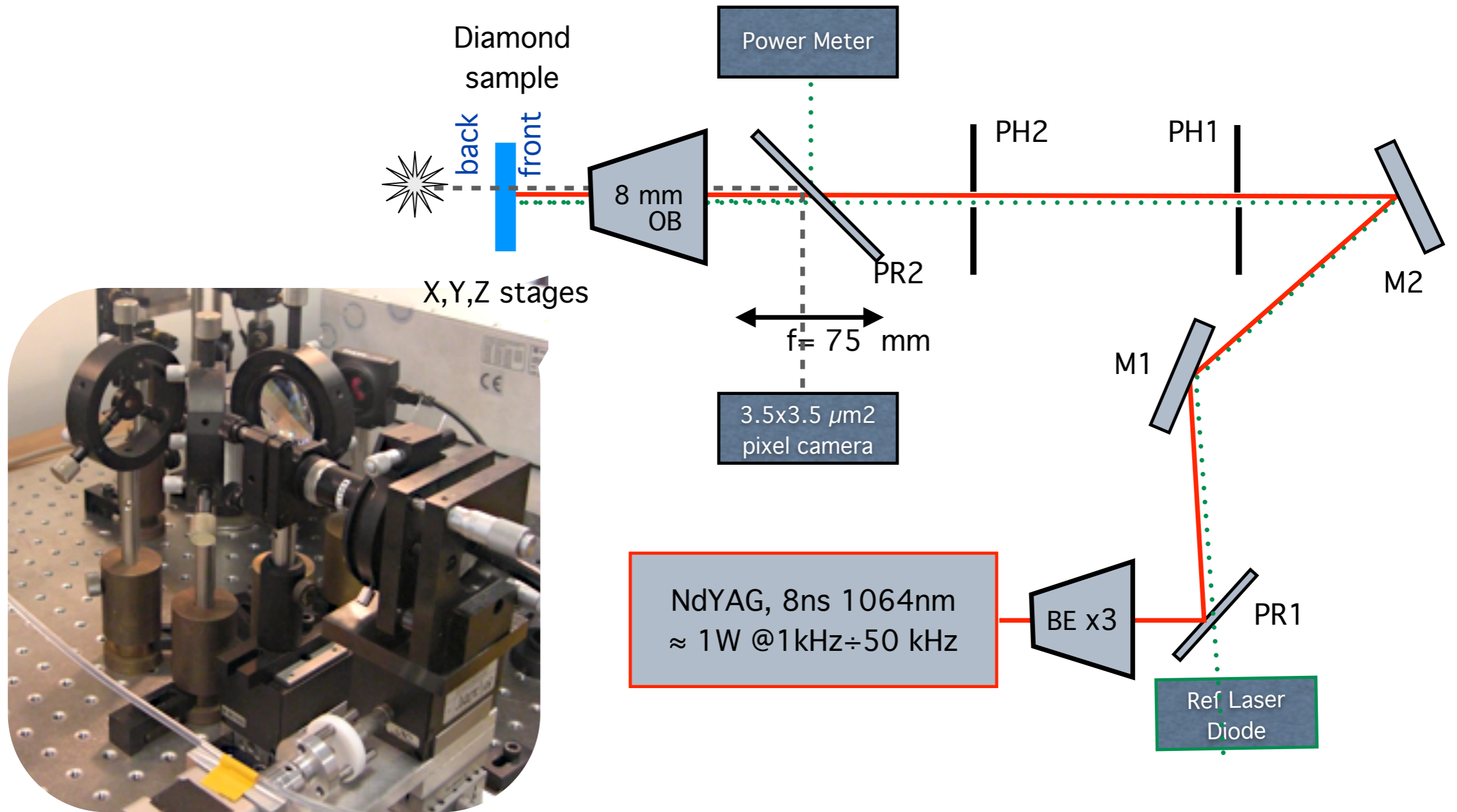
Some concerns are about the integrity of diamond due to different specific volumes.

Some concerns are on the quality of the laser produced graphite.

But our main issue is the electrical quality of the graphite-D contact and its stability.

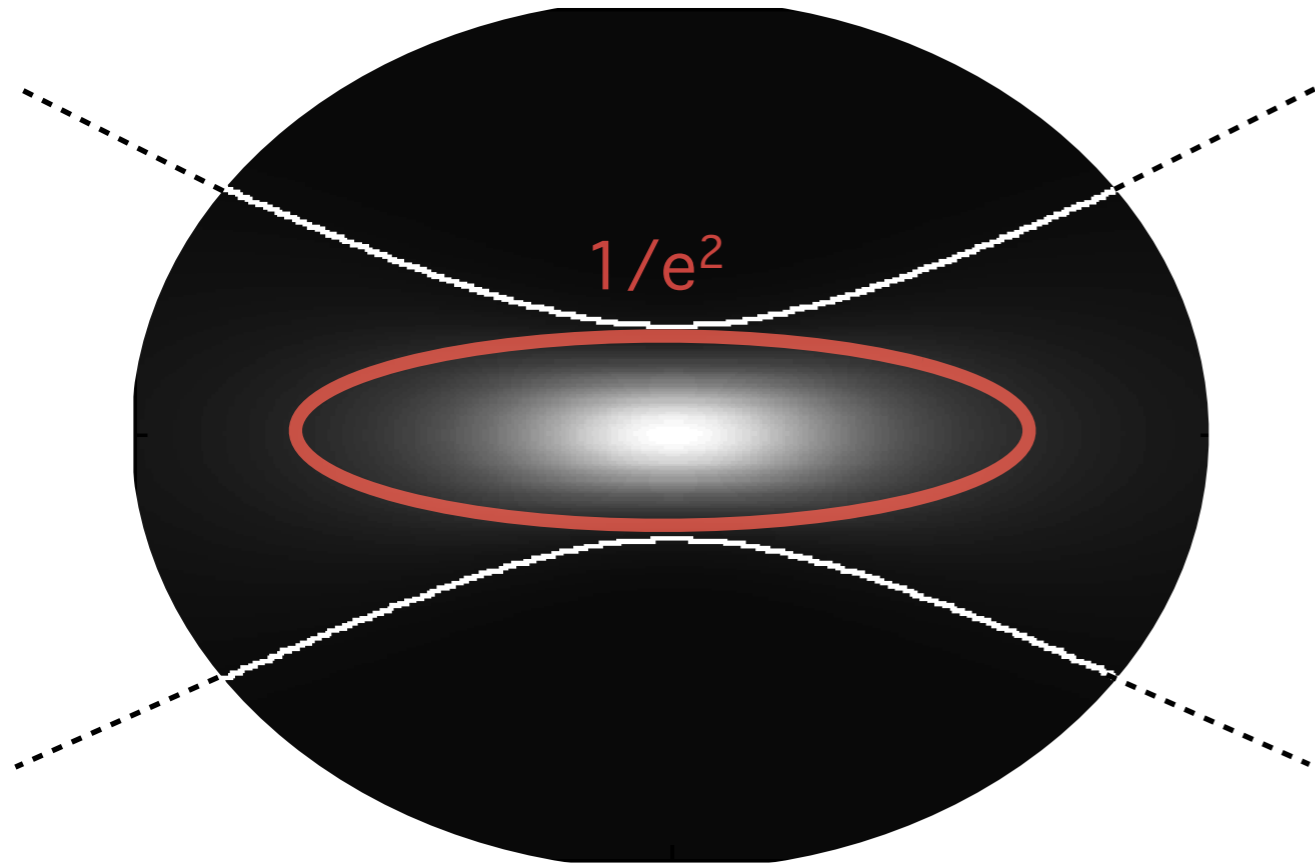
Its reputation in literature is a bit controversial anyway we rely on the graphitization as the solution for the buried contacts.

Graphitization set-up

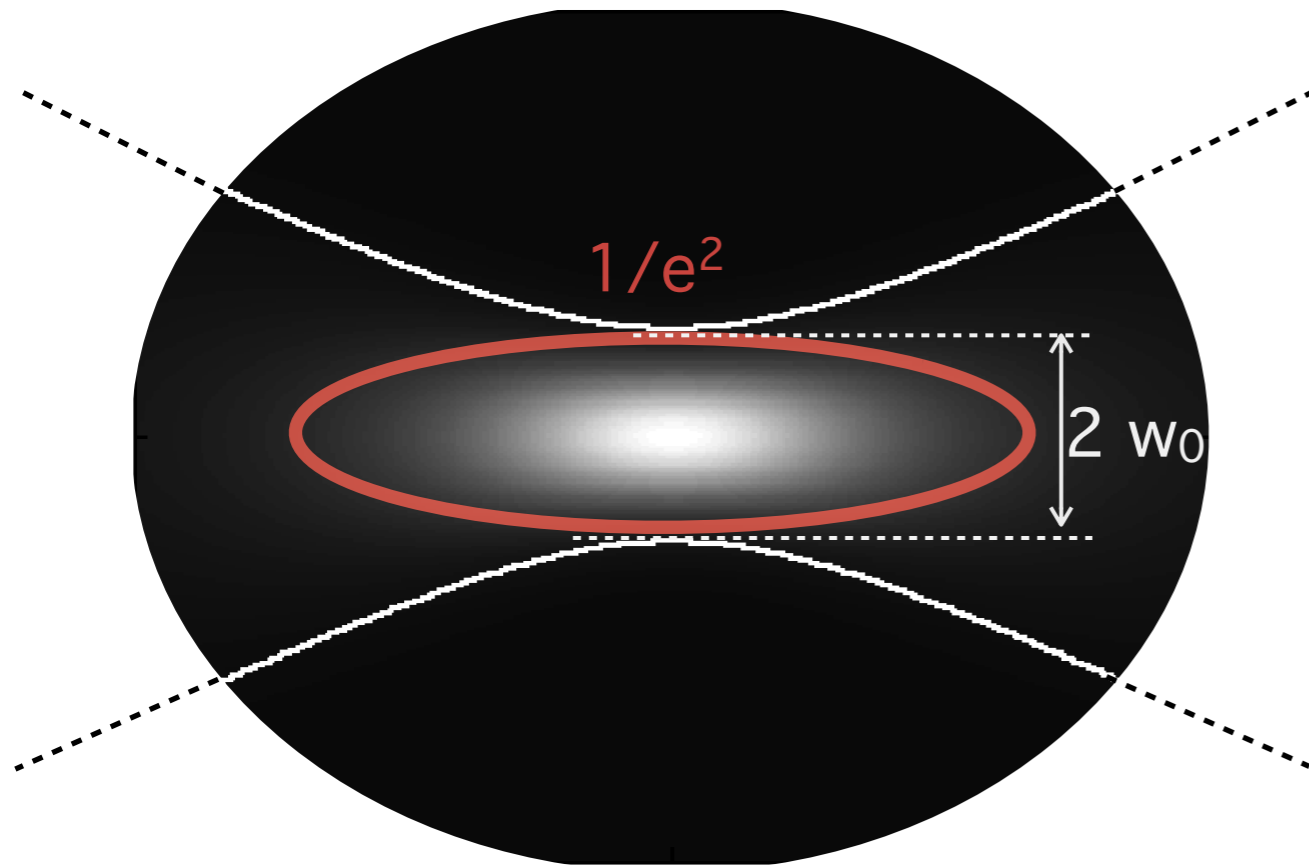


Experimentation with 100 fs @ 800 nm Laser is on going

beam spot dimension: waist diameter, depth of focus



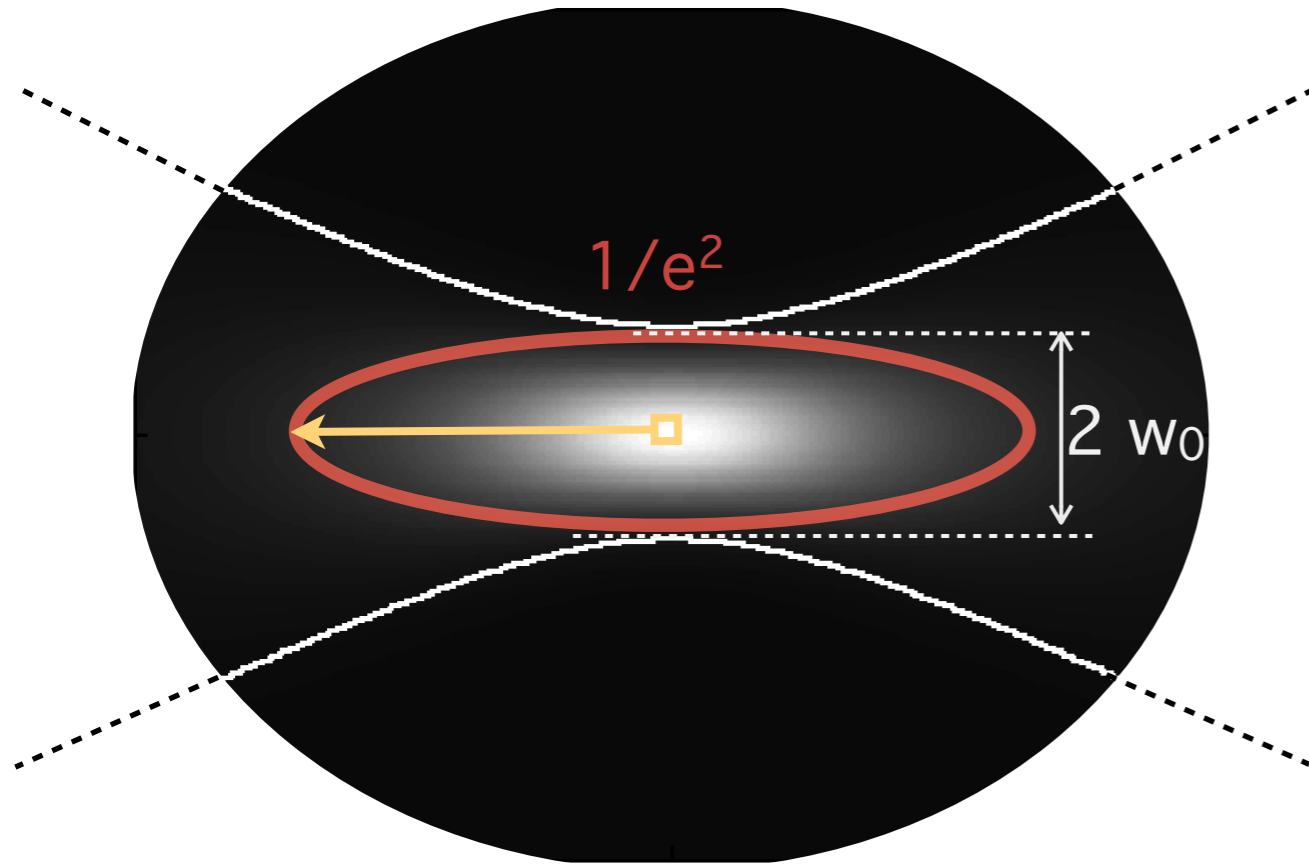
beam spot dimension: waist diameter, depth of focus



Air

$$2 w_0 = \frac{2\lambda}{\pi} \times (f / \#) \approx 3.5 \mu m$$

beam spot dimension: waist diameter, depth of focus

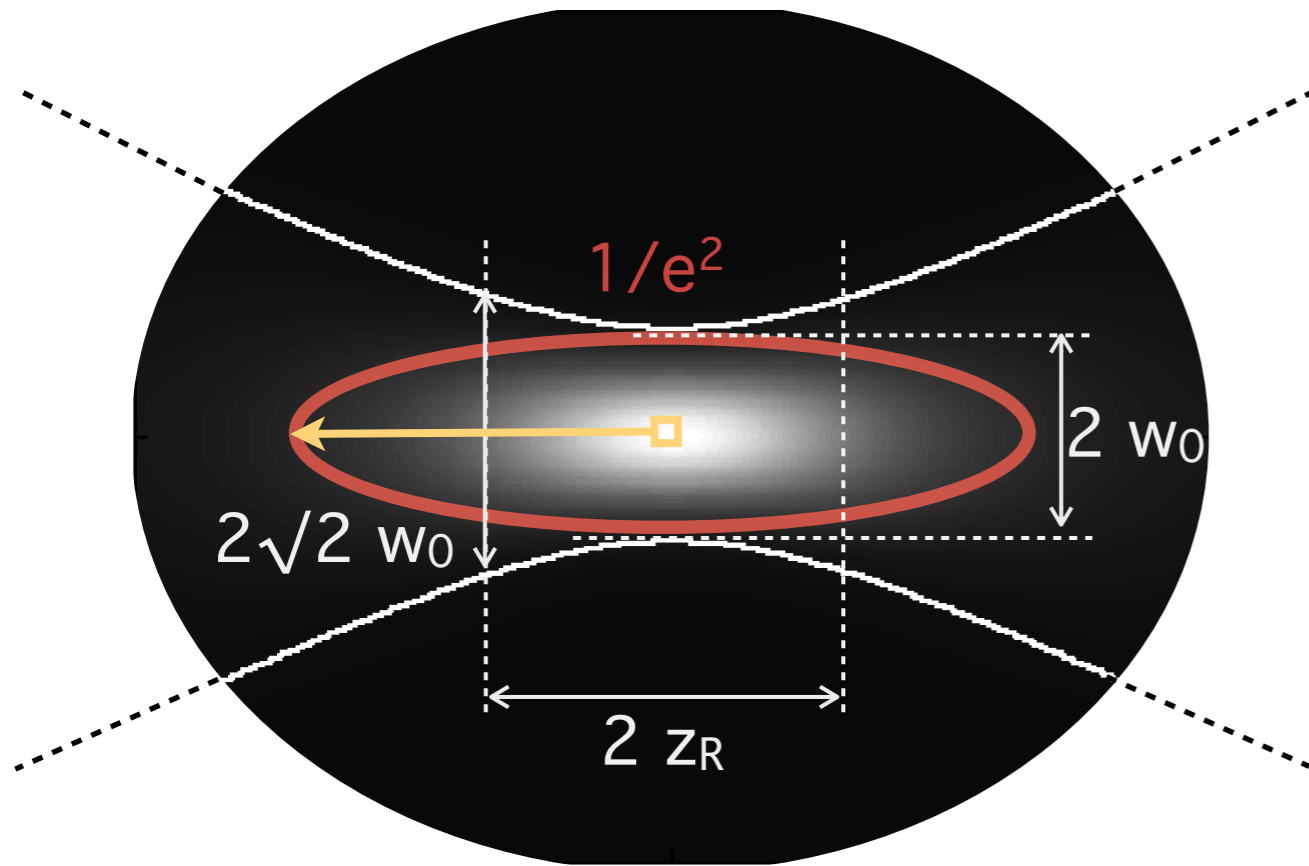


Air

$$2 w_0 = \frac{2\lambda}{\pi} \times (f / \#) \approx 3.5 \mu m$$

$$2 z_R = \frac{2\pi}{\lambda} \times w_0^2 \approx 19 \mu m$$

beam spot dimension: waist diameter, depth of focus

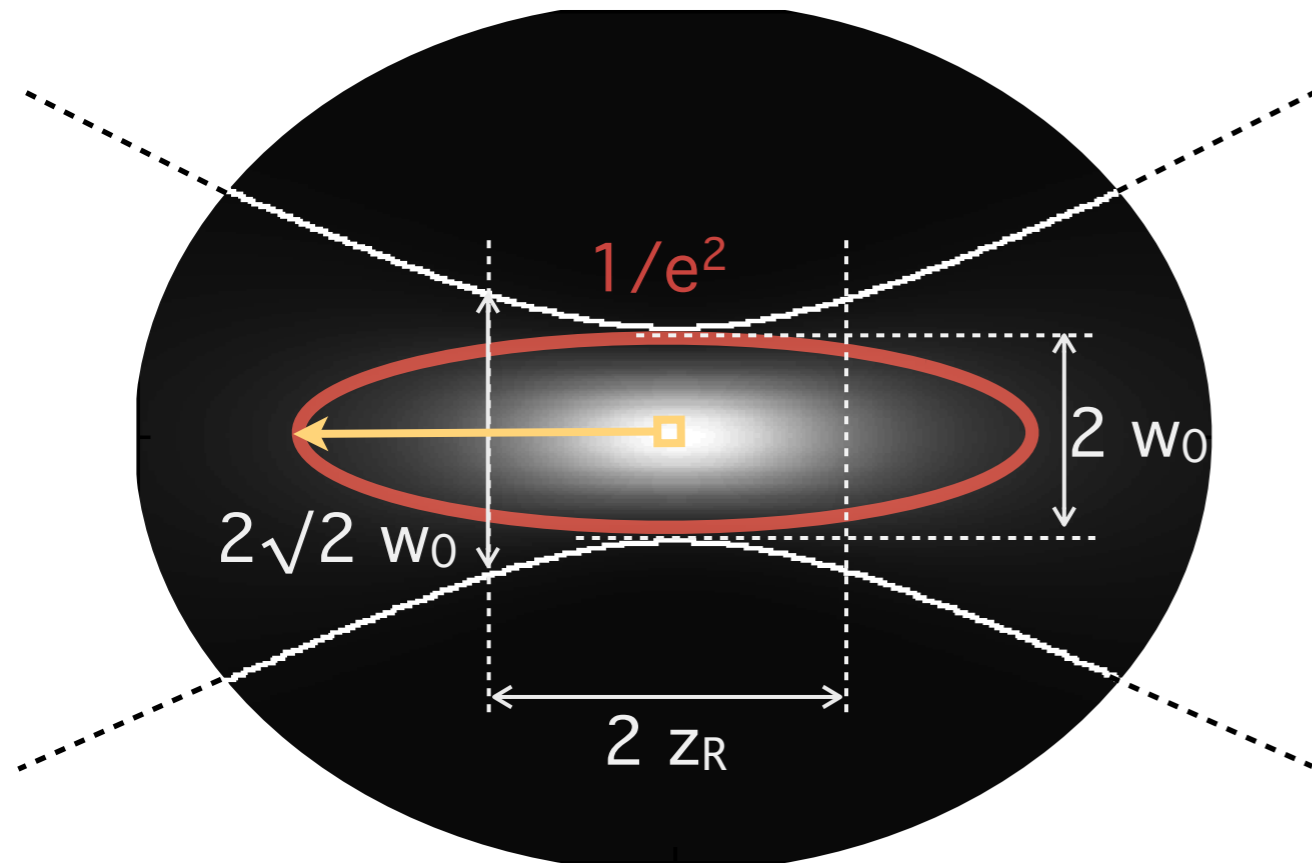


Air

$$2 w_0 = \frac{2\lambda}{\pi} \times (f / \#) \approx 3.5 \mu m$$

$$2 z_R = \frac{2\pi}{\lambda} \times w_0^2 \approx 19 \mu m$$

beam spot dimension: waist diameter, depth of focus



Air

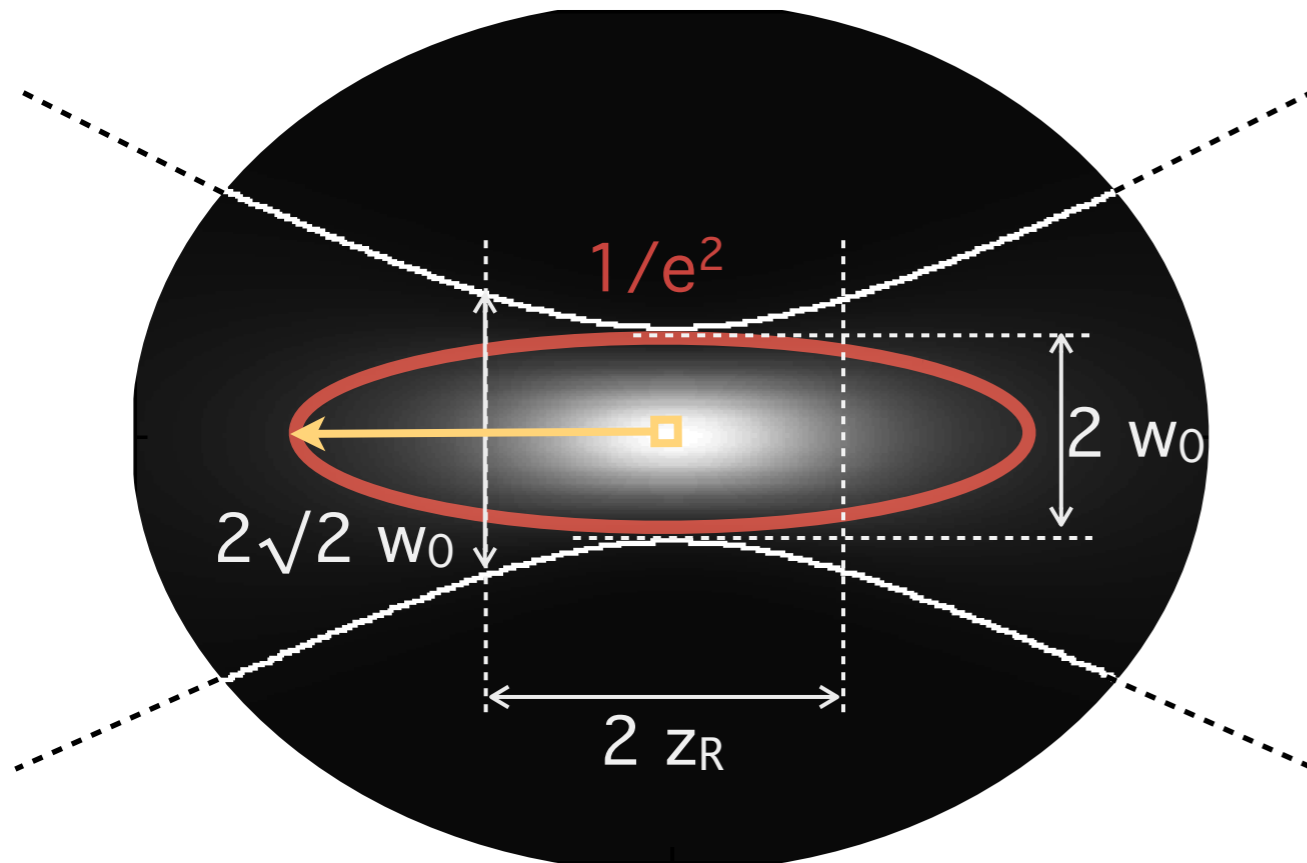
$$2w_0 = \frac{2\lambda}{\pi} \times (f/\#) \approx 3.5 \mu m$$

$$2z_R = \frac{2\pi}{\lambda} \times w_0^2 \approx 19 \mu m$$

Diamond

only DOF increases ($n_D \approx 2.42$): $2z_R \approx 46 \mu m$

beam spot dimension: waist diameter, depth of focus



Air

$$2w_0 = \frac{2\lambda}{\pi} \times (f/\#) \approx 3.5 \mu\text{m}$$

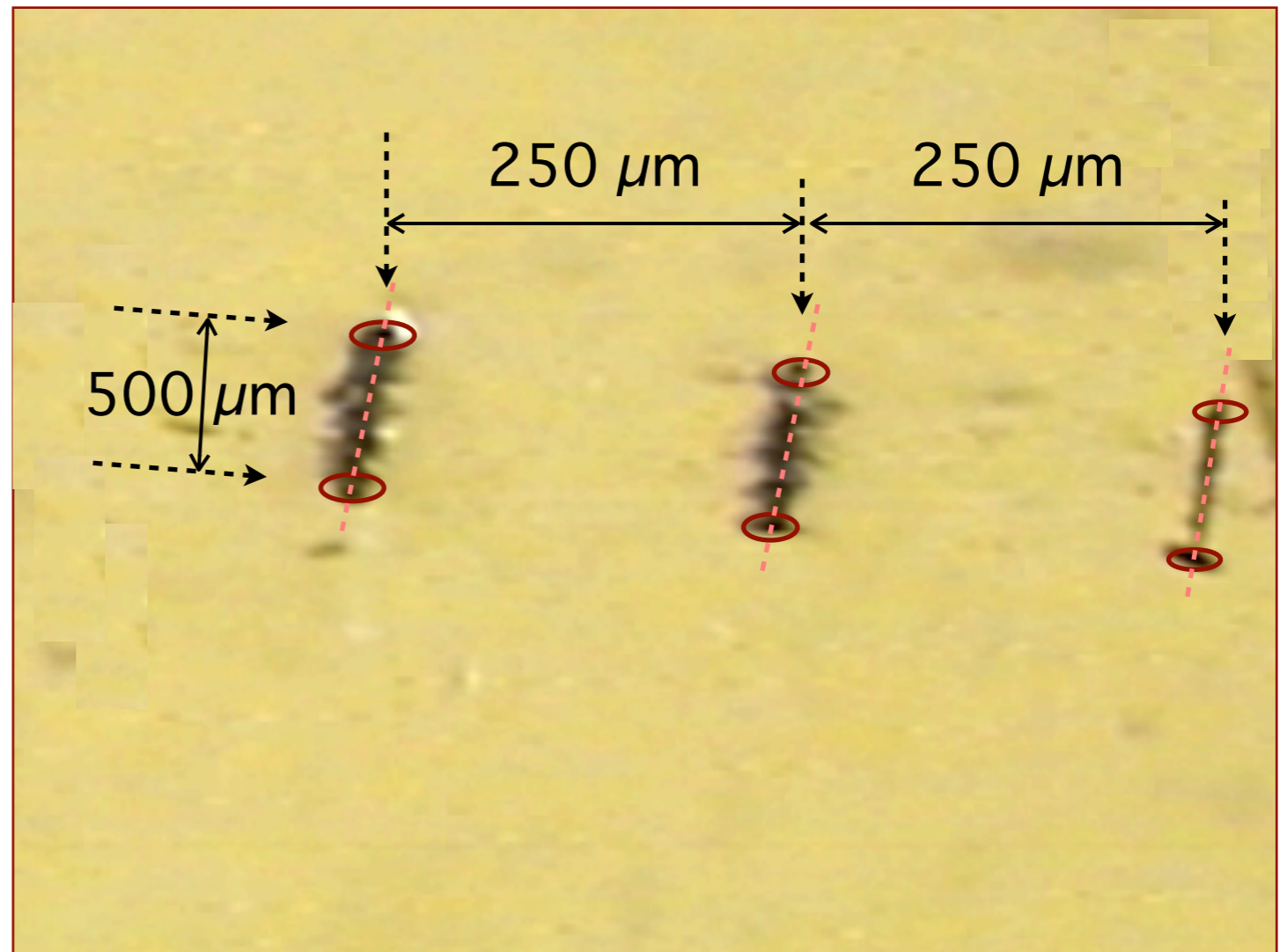
$$2z_R = \frac{2\pi}{\lambda} \times w_0^2 \approx 19 \mu\text{m}$$

Diamond

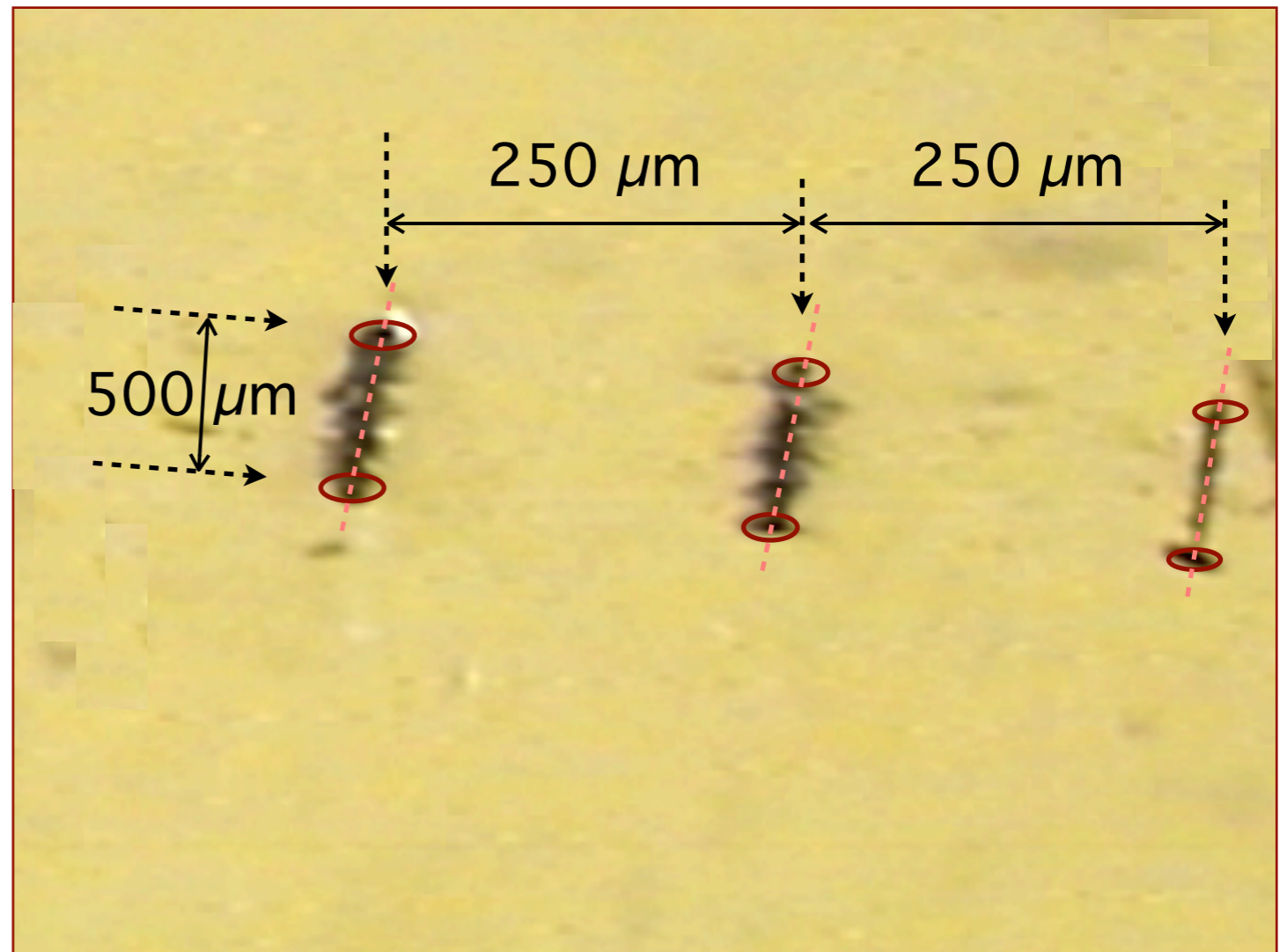
only DOF increases ($n_D \approx 2.42$): $2z_R \approx 46 \mu\text{m}$

The distribution of the focus intensity is important to determine the shape of the graphitization volume. Other parameters are the energy ($< \text{few } \mu\text{J/pulse}$), the scanning velocity ($\leq \text{few mm/s}$) and the pulse repetition rate (10 KHz).

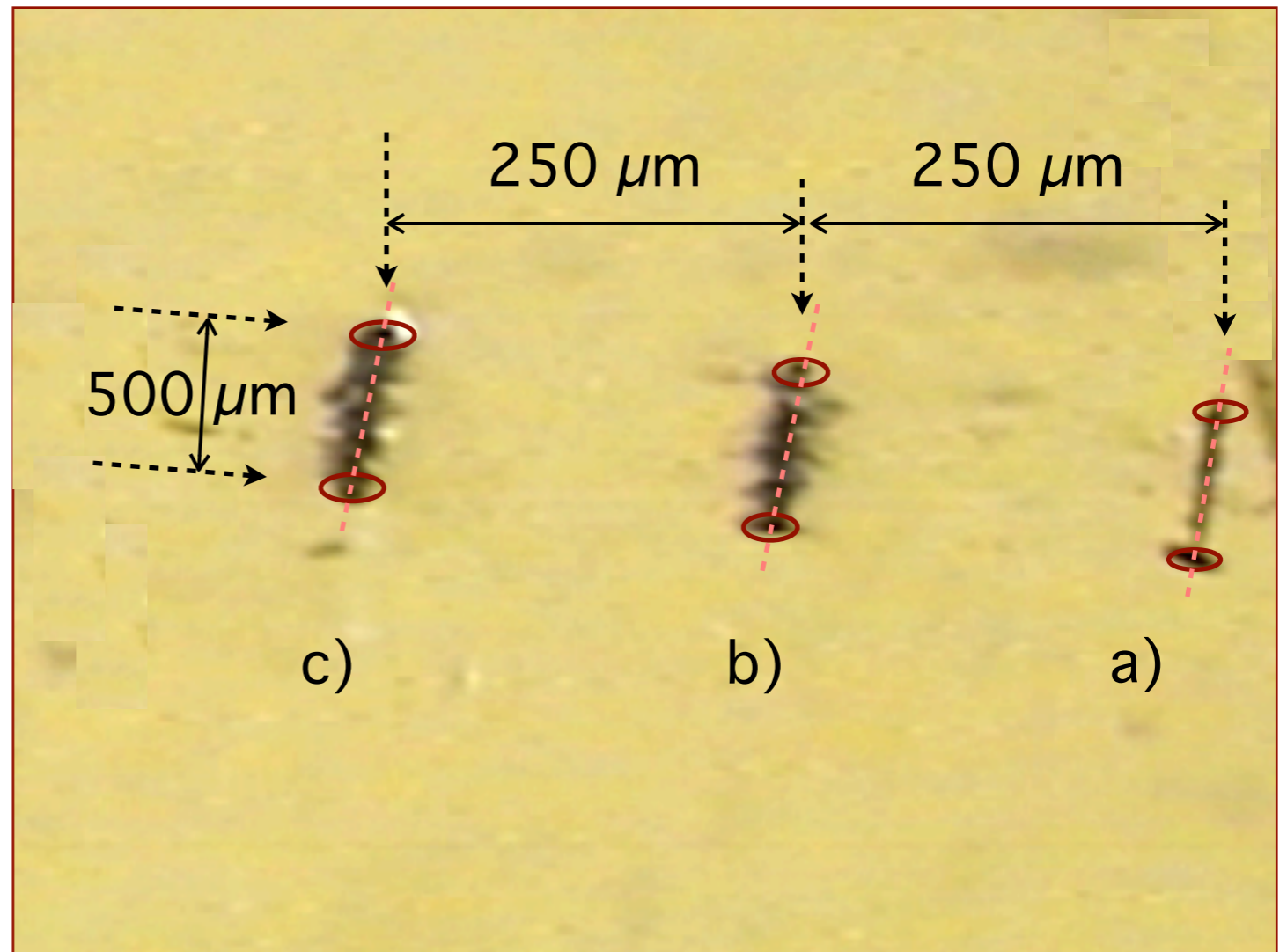
example of graphite objects (500 μm poly diamond): columns



example of graphite objects (500 μm poly diamond): columns



example of graphite objects (500 μm poly diamond): columns



Electrical resistivity obtained from column experimentation

$$\rho \approx 50 \div 250 \text{ m}\Omega \cdot \text{cm}$$

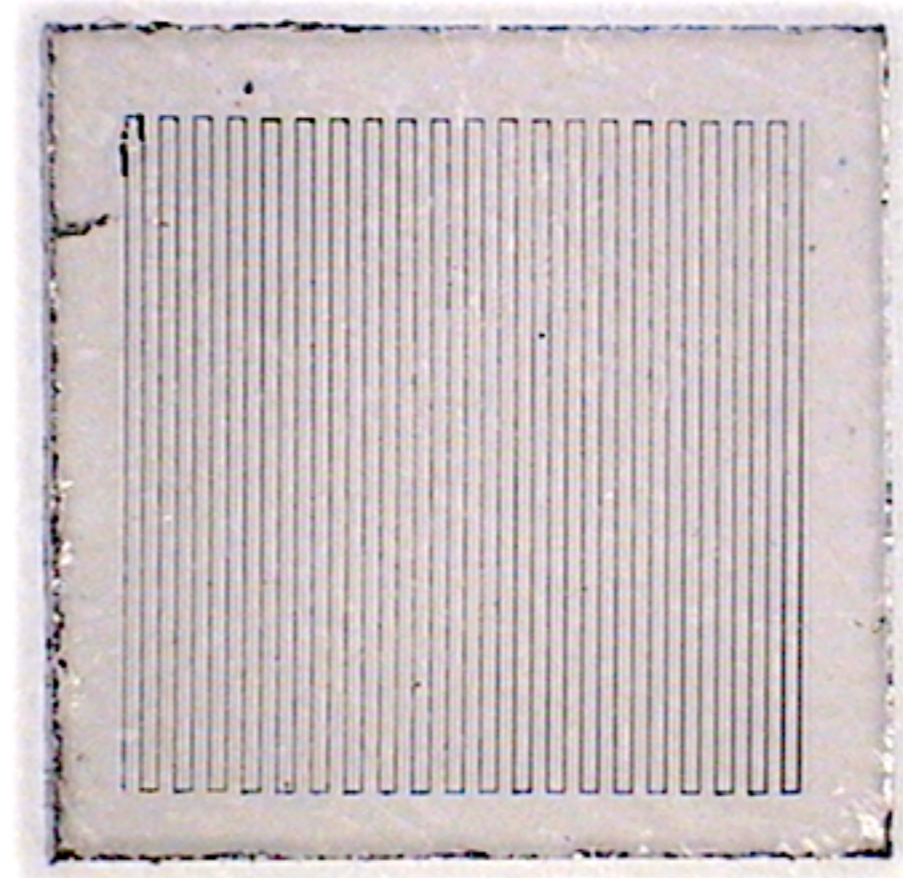
(main uncertainty due to the effective cross section)

	$\mu\text{J}/\text{pulse}$	$\varnothing/\mu\text{m}$	s/cm^2	$\text{R}/\text{K}\Omega$
a)	7	$9 \div 8$	$6.00\text{E}-07$	46
b)	13	$16 \div 13$	$1.70\text{E}-06$	7.3
c)	17	$19 \div 19$	$2.80\text{E}-06$	3.8

The Greek fret pattern

A long graphite zigzag track has been carved on the sample surface (both faces).

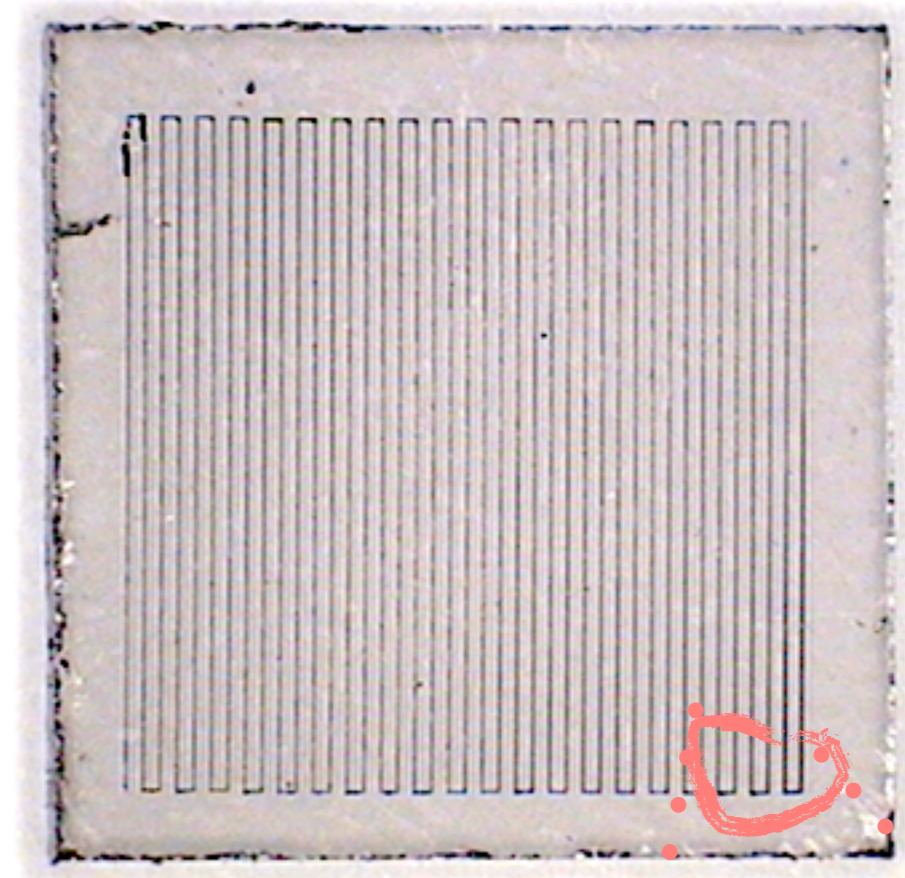
5x5 mm², 500 μm thick sample



The Greek fret pattern

A long graphite zigzag track has been carved on the sample surface (both faces).

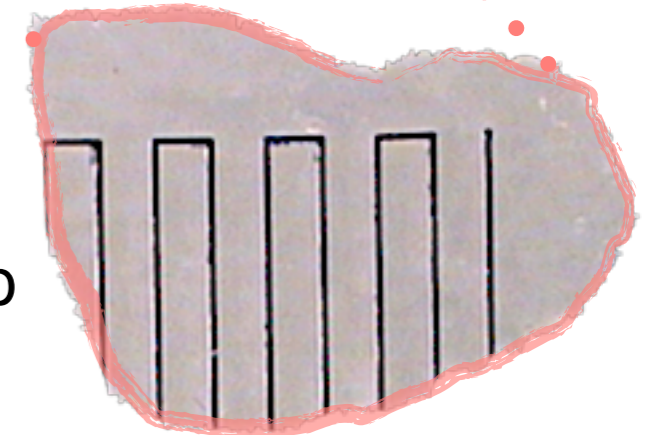
5x5 mm², 500 μm thick sample



100 μm pitch

9 μm width

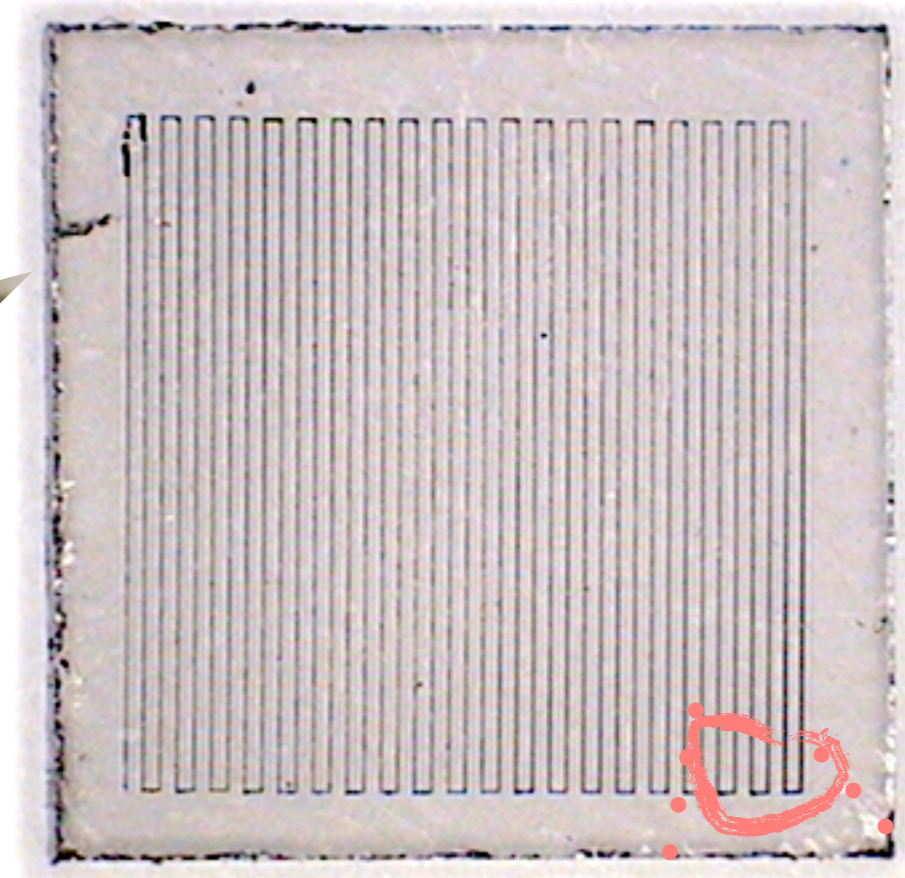
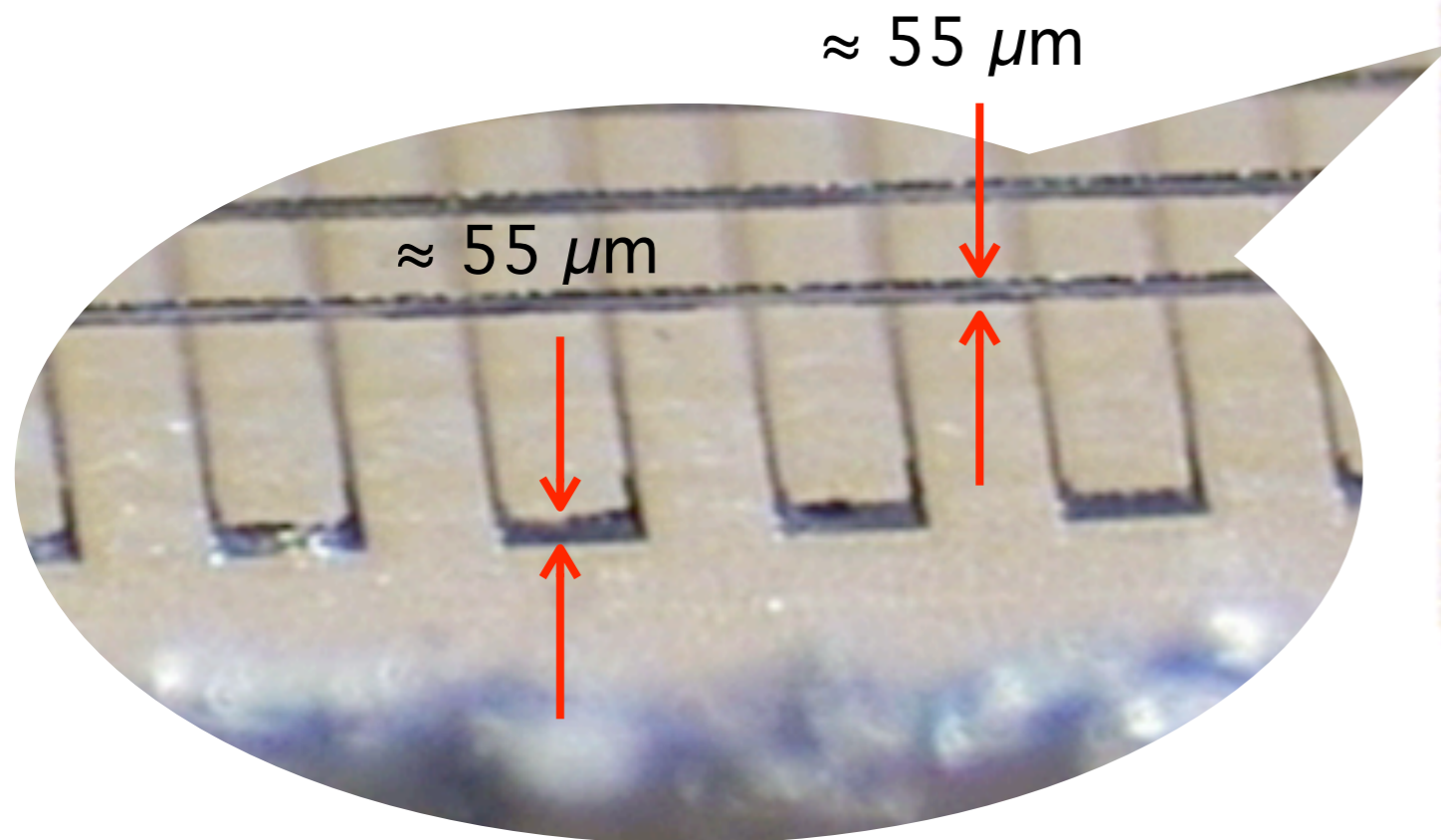
≈ 55 ± 5 μm deep



The Greek fret pattern

A long graphite zigzag track has been carved on the sample surface (both faces).

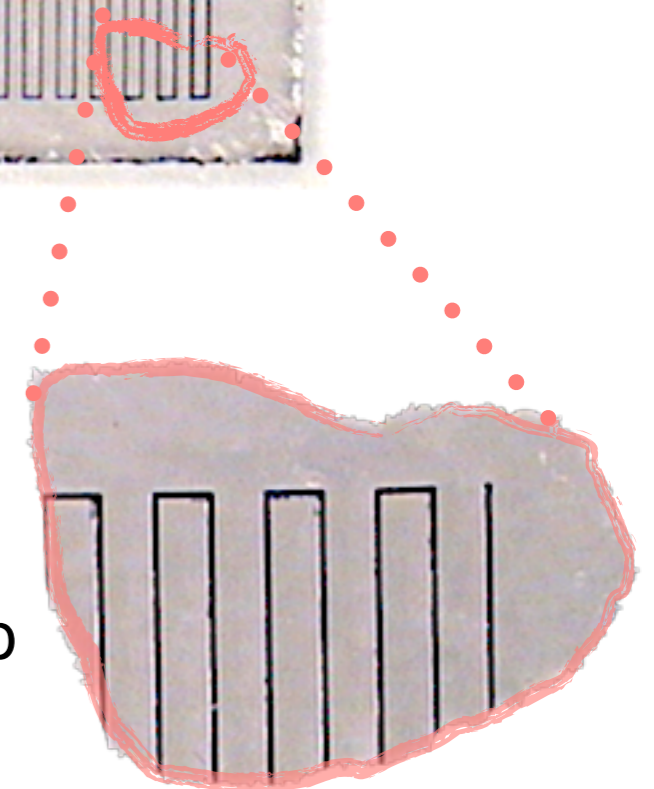
5x5 mm², 500 μm thick sample



100 μm pitch

9 μm width

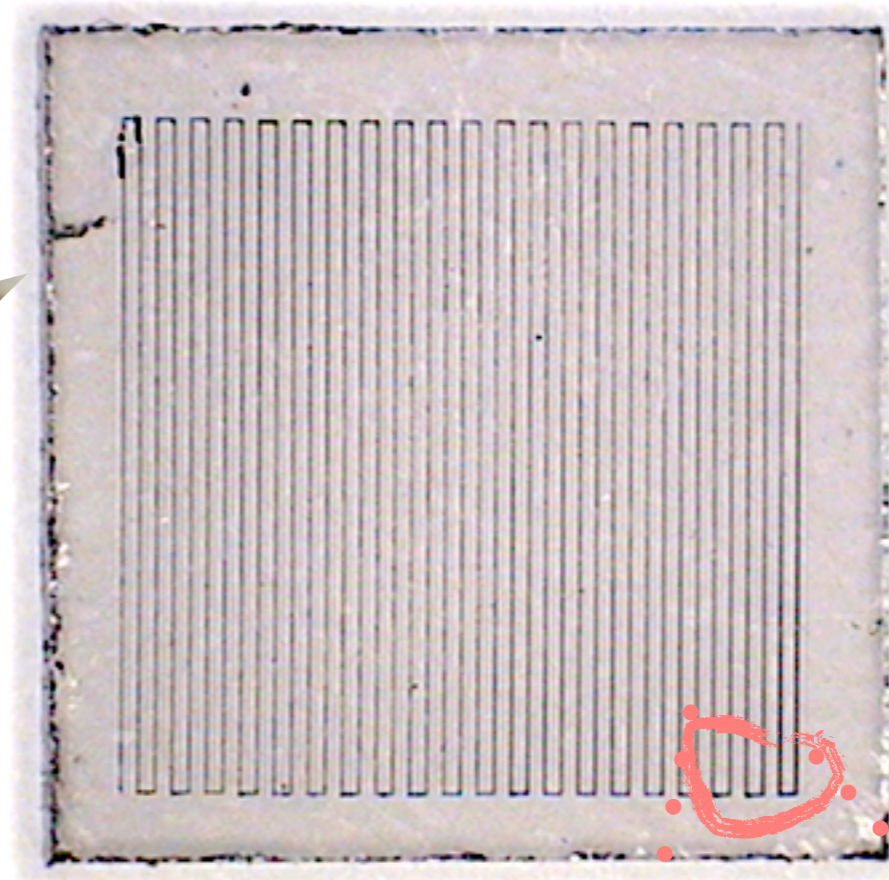
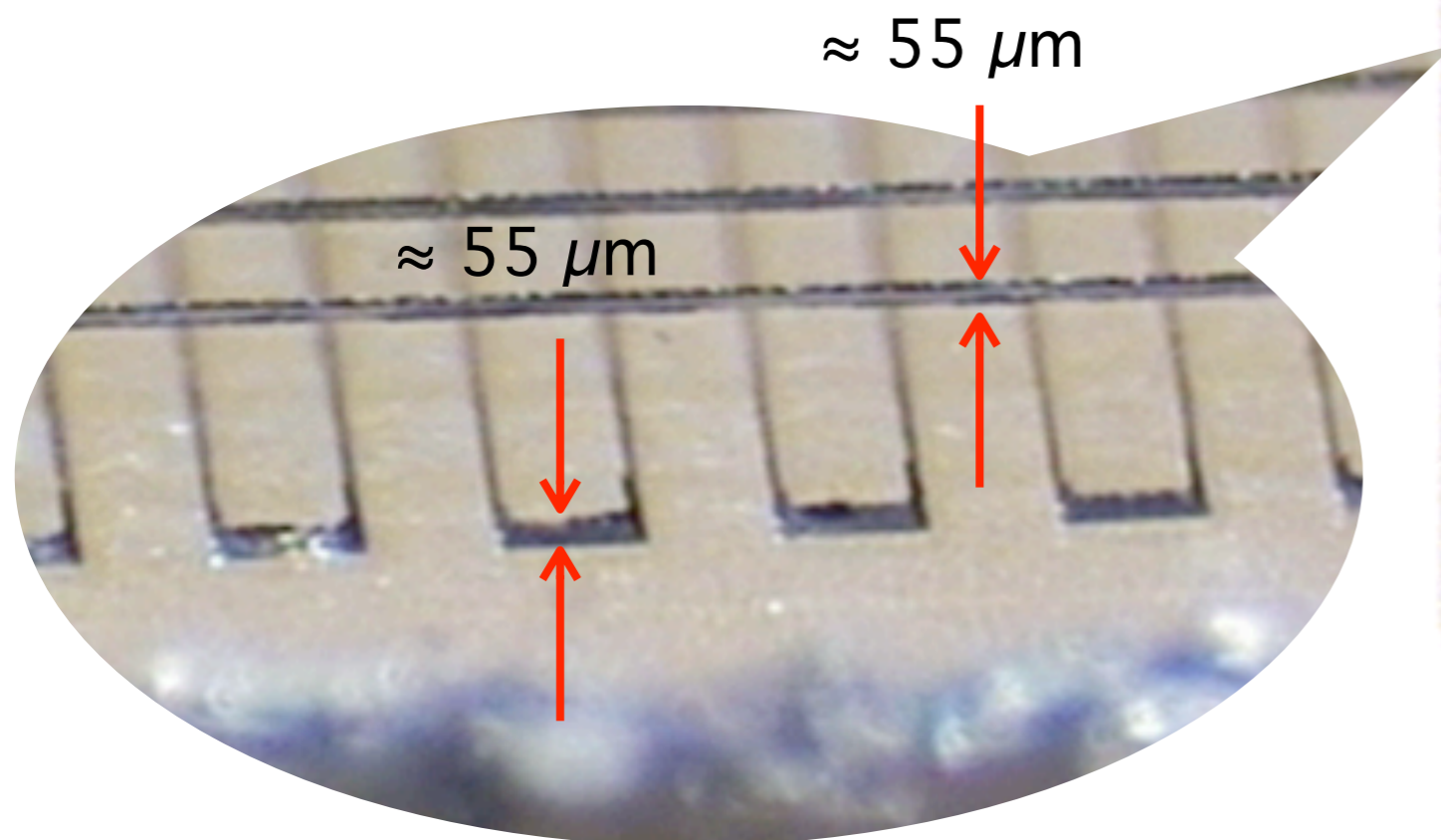
$\approx 55 \pm 5 \mu\text{m}$ deep



The Greek fret pattern

A long graphite zigzag track has been carved on the sample surface (both faces).

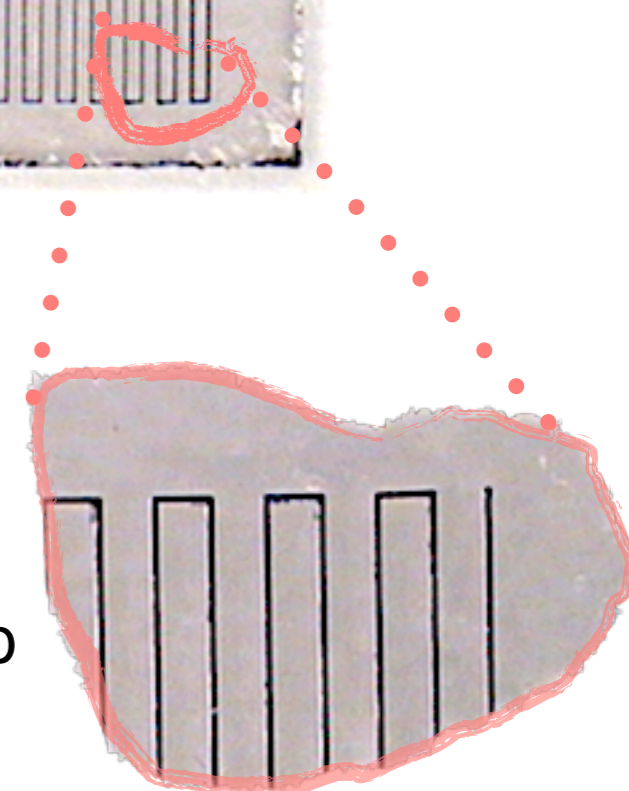
5x5 mm², 500 μm thick sample



100 μm pitch

9 μm width

$\approx 55 \pm 5 \mu\text{m}$ deep

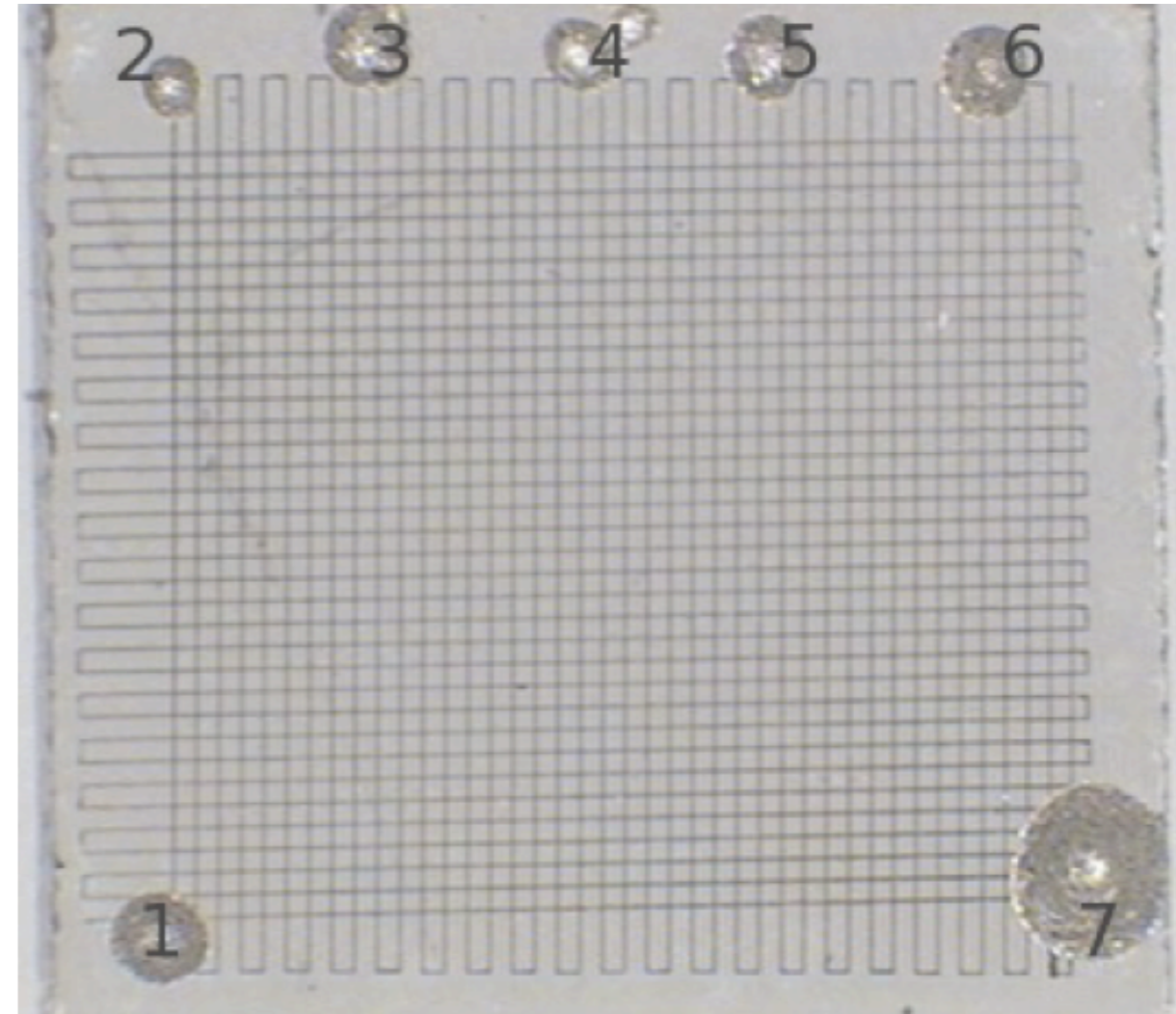
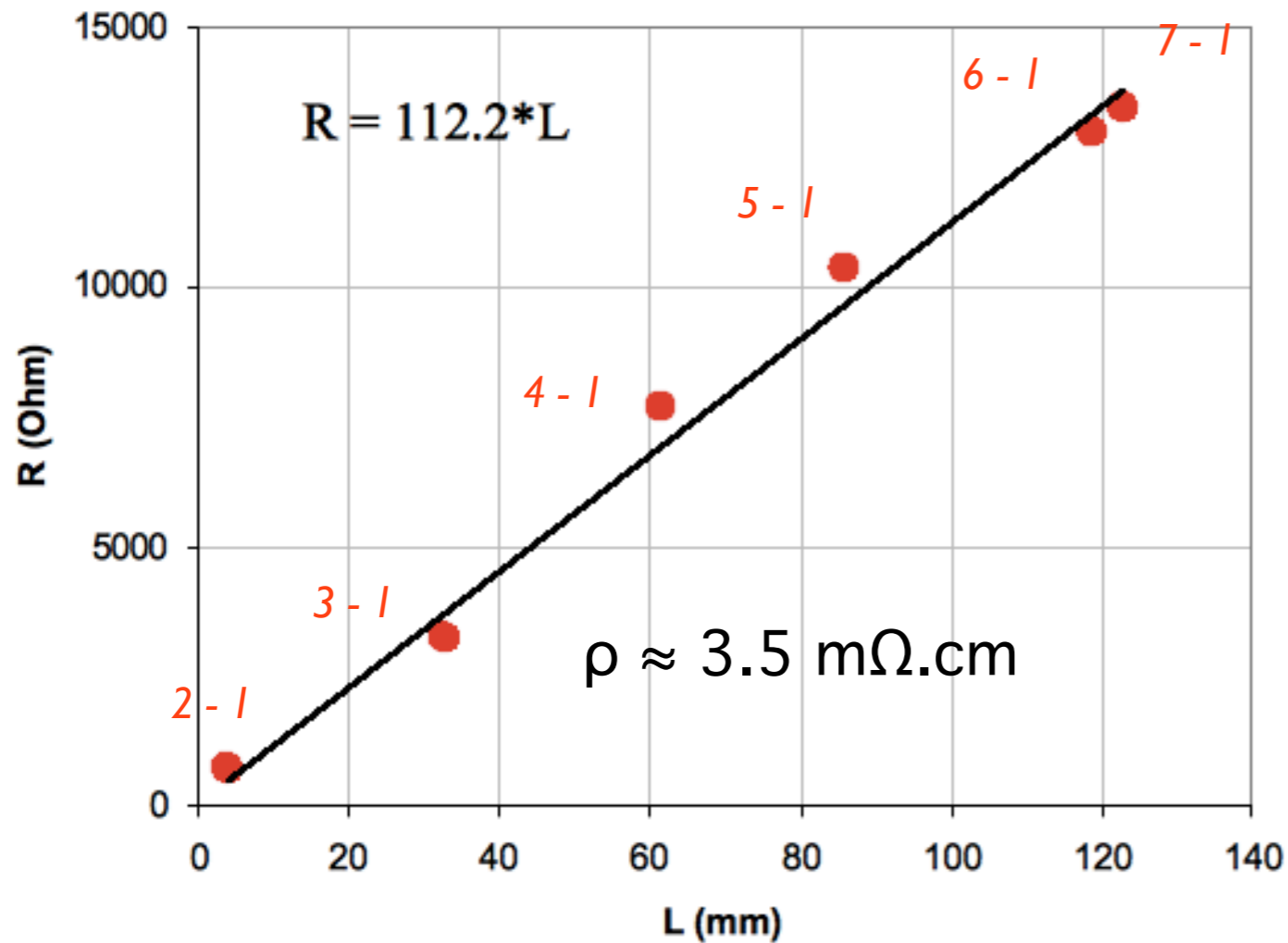


Geometry suitable to study graphite-diamond contact as detector electrodes.

The same geometry could be done with shorter laser wavelengths to minimize the track depth.

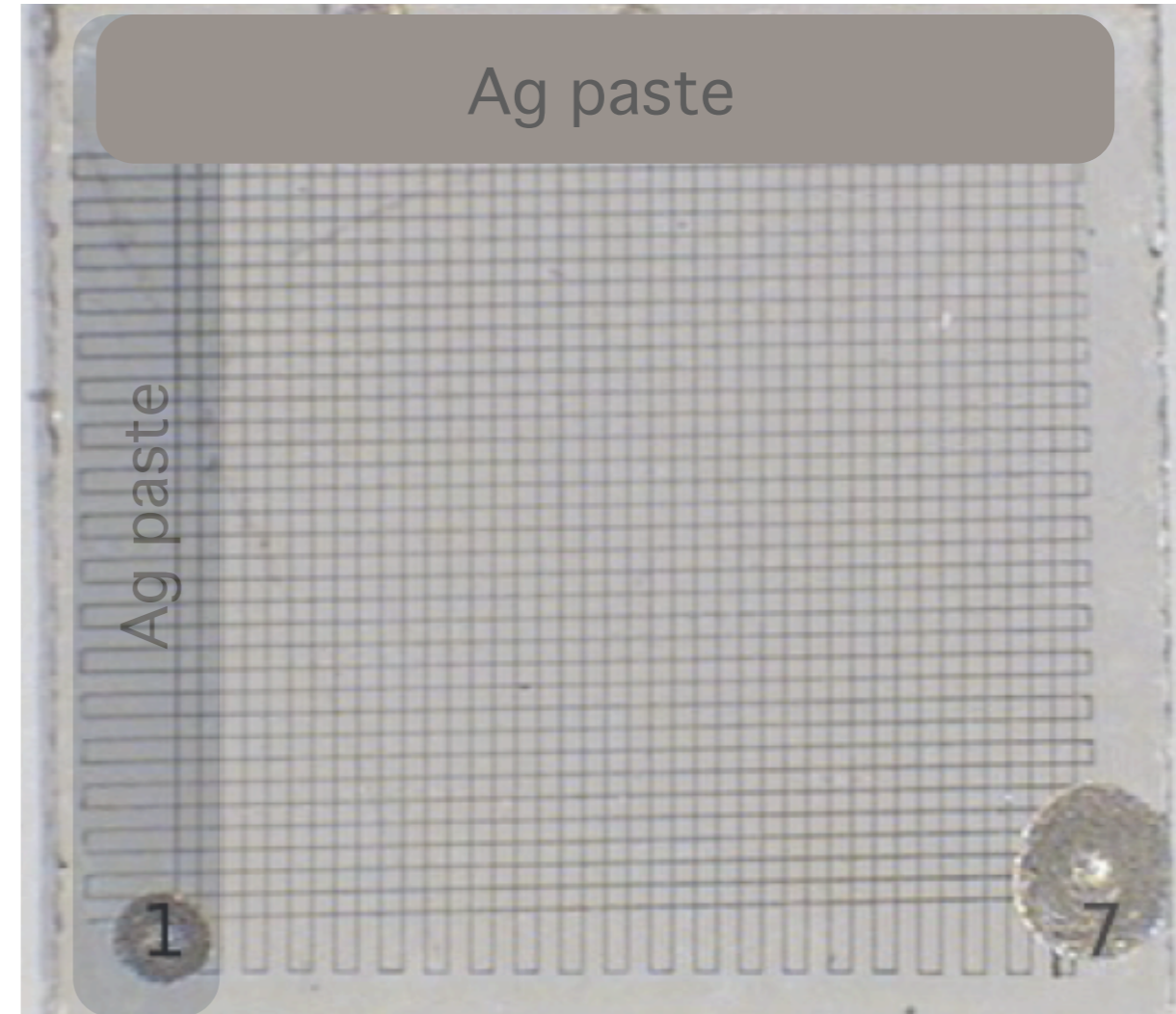
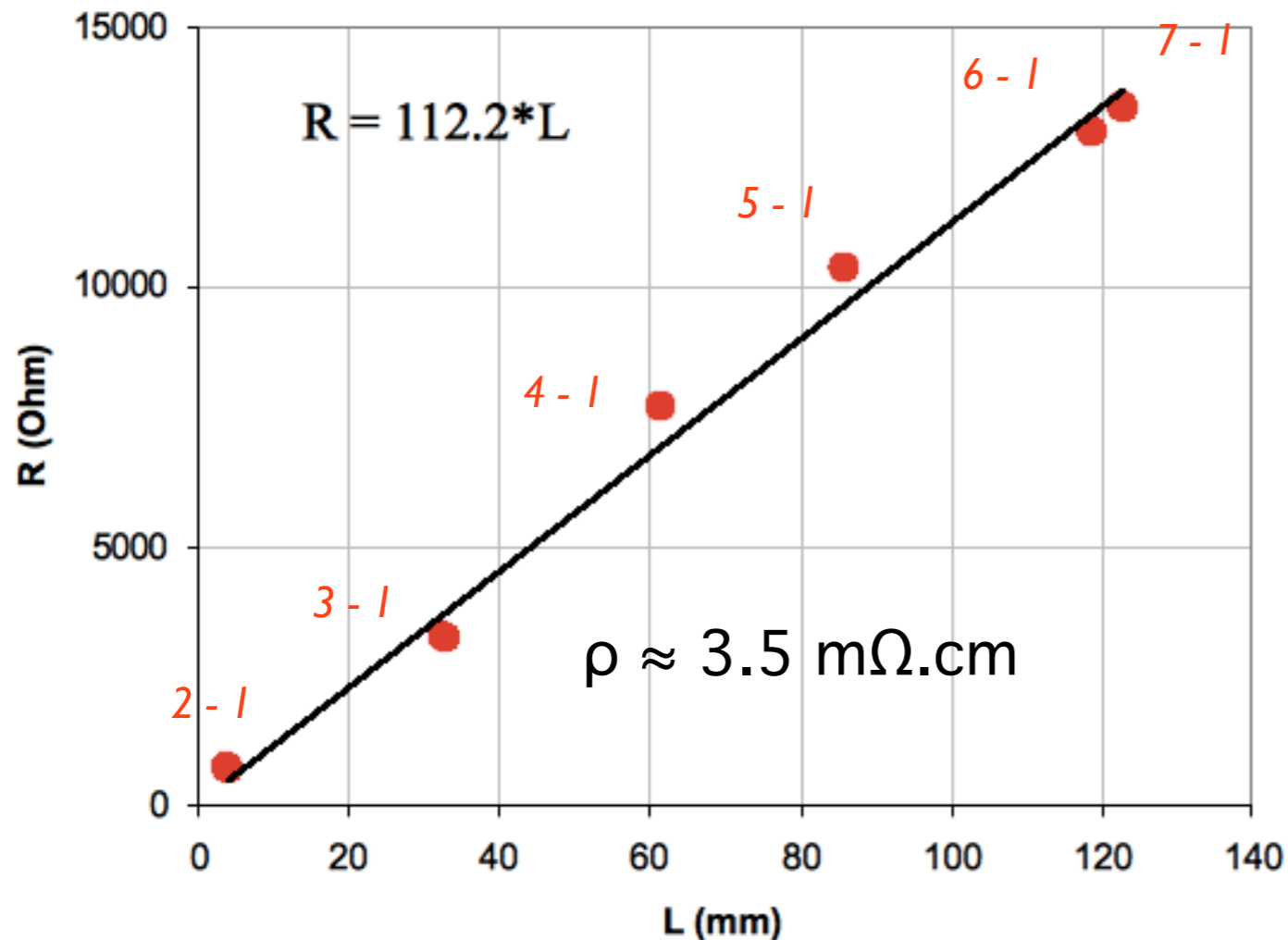
The graphite Greek Fret electrode

The track resistance per unit length has been measured to be $\approx 112 \Omega/\text{mm}$
(resistivity $\approx 3.5 \text{ m}\Omega\cdot\text{cm}$ if track depth $\approx 55 \mu\text{m}$)



The graphite Greek Fret electrode

The track resistance per unit length has been measured to be $\approx 112 \Omega/\text{mm}$
(resistivity $\approx 3.5 \text{ m}\Omega\cdot\text{cm}$ if track depth $\approx 55 \mu\text{m}$)



The two GF electrodes overlap of about 0.59 of the sample surface and the measure of the coupling capacitance $C_d \approx 2.24 \text{ pF}$.

Contact impedance expected is $\approx 1/j\omega C_d + R$ with $R < 30 \Omega$ ($\approx 10 \Omega$?)

poly diamond detectors, comparison

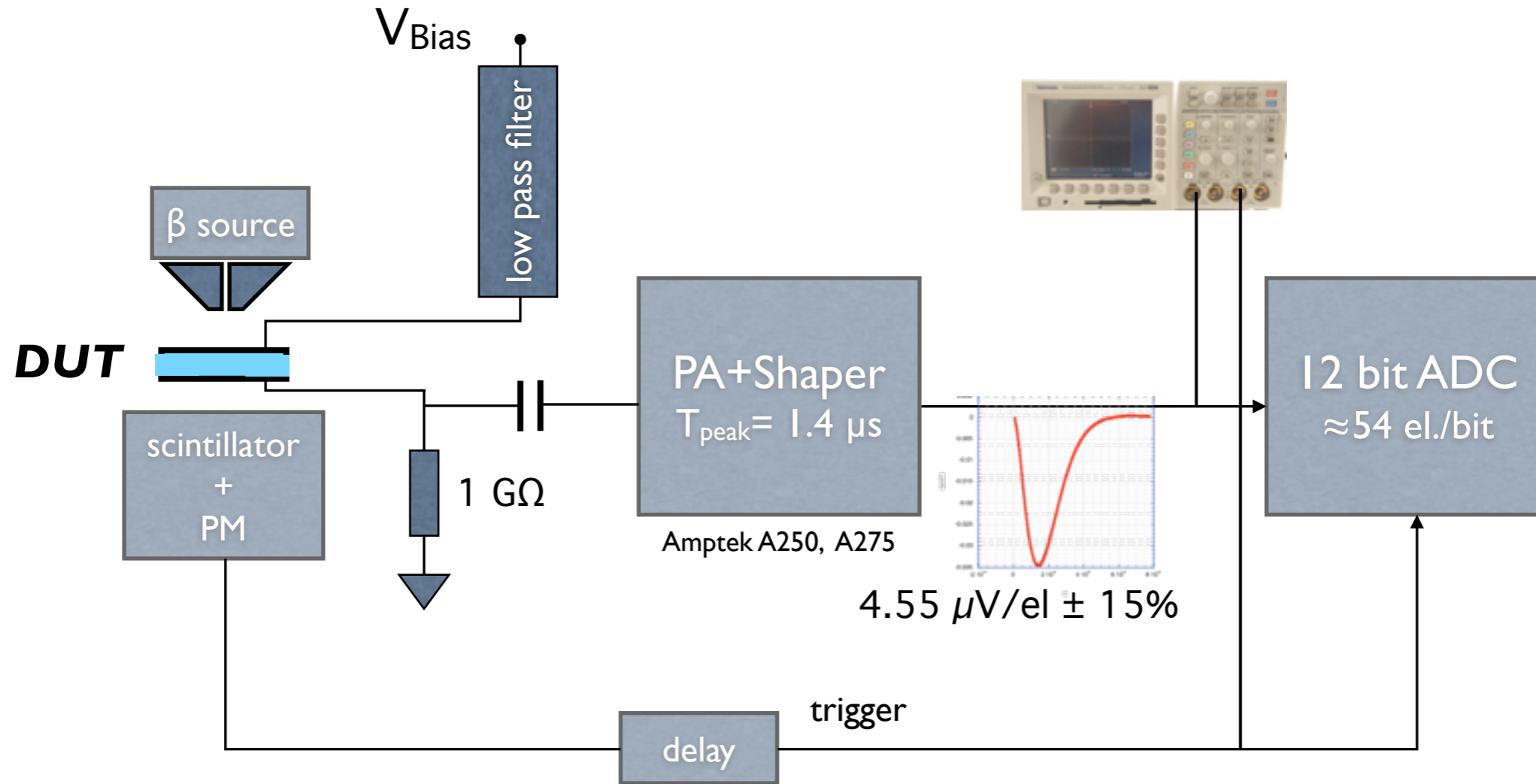
The “greek fret” detector (GF) has been compared with an other one of the same geometrical thickness (500 μm), of the same batch (Diamond Detectors) but equipped with Ti-Au plane electrodes. The extension of the plane electrodes with respect to the sample surface is $\approx 85\%$ in the last case while $\approx 59\%$ in the former.

Also the sensitive thickness of the GF detector is smaller ($\approx 20\%$) due to the depth of the graphite tracks ($\approx 390 \mu\text{m}$).

The measured capacitances are 2.24 pF (GF) and 2.56 pF (Ti-Au). From their ratio we obtain an independent evaluation of the GF sensitive thickness:

397 μm which well consistent with the previous one.

the test set-up

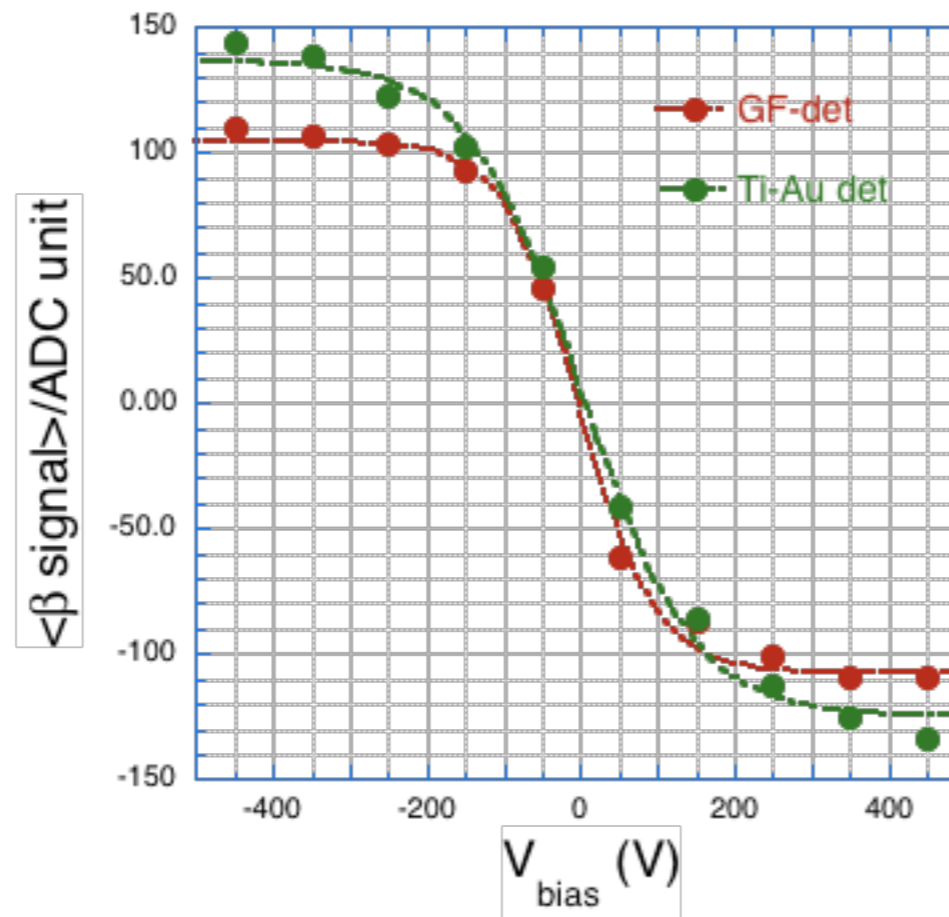


signal positive with $V_{bias} < 0$

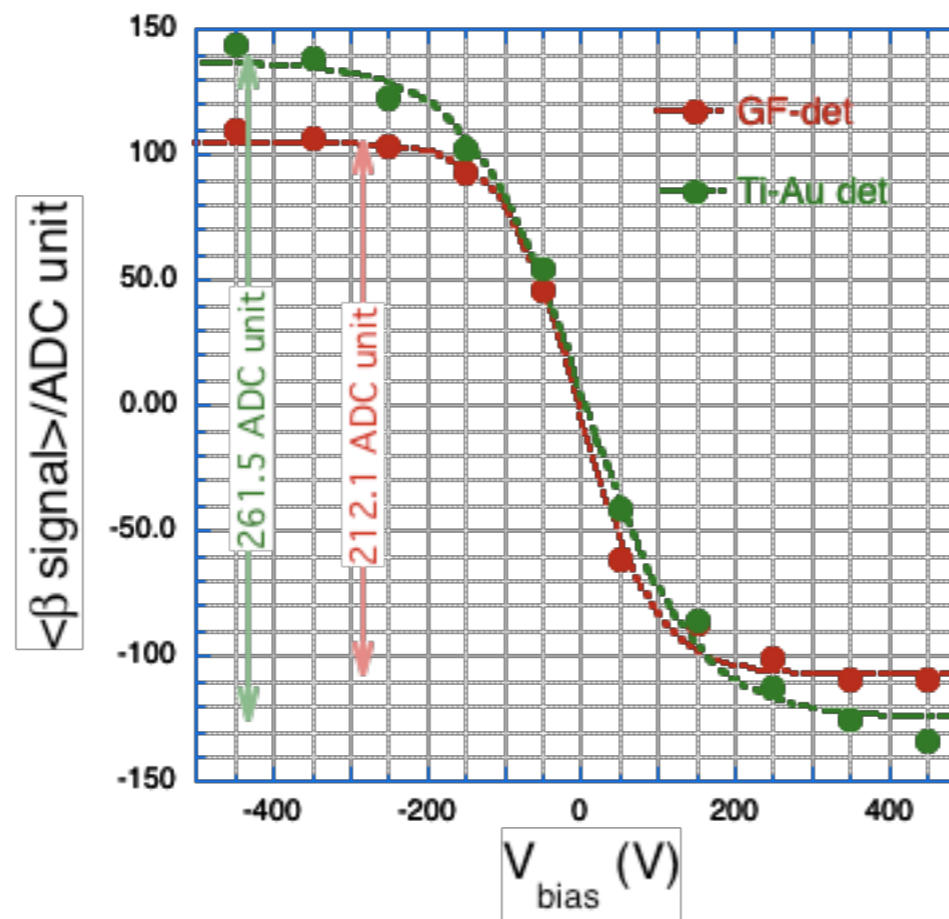
signal negative with $V_{bias} > 0$

Pre-Amp equivalent input serial noise resistance : 60÷70 Ω

Results of measurements: signal+noise



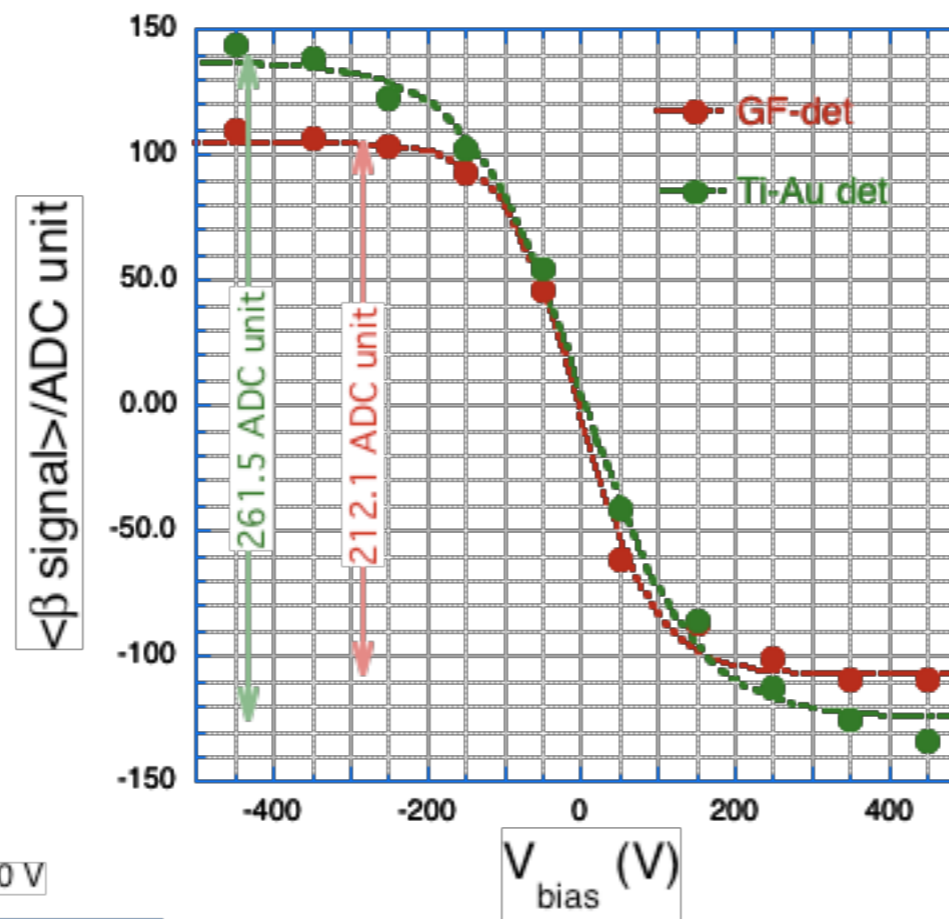
Results of measurements: signal+noise



$$261.5/212.1 \approx 1.23$$

$$(500/390 \approx 1.28)$$

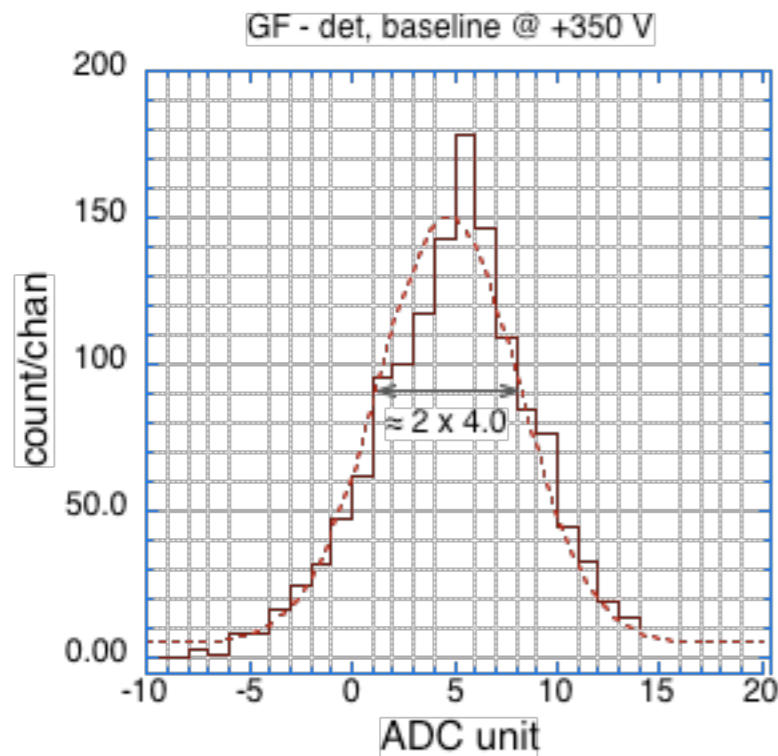
Results of measurements: signal+noise



$$261.5/212.1 \approx 1.23$$

$$(500/390 \approx 1.28)$$

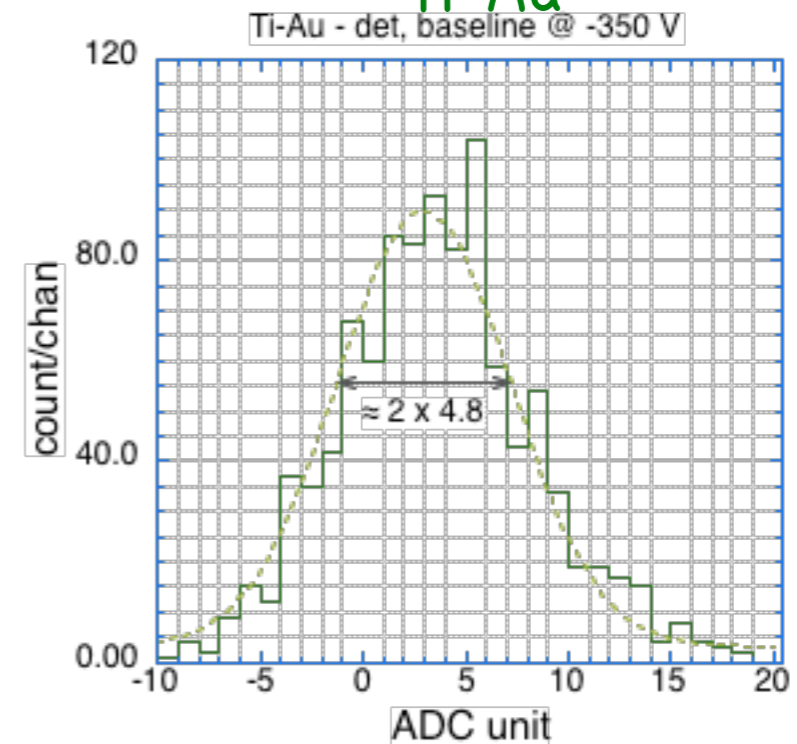
GF



$$4.8/4 = 1.2$$

$$(2.56/2.24 = 1.15)$$

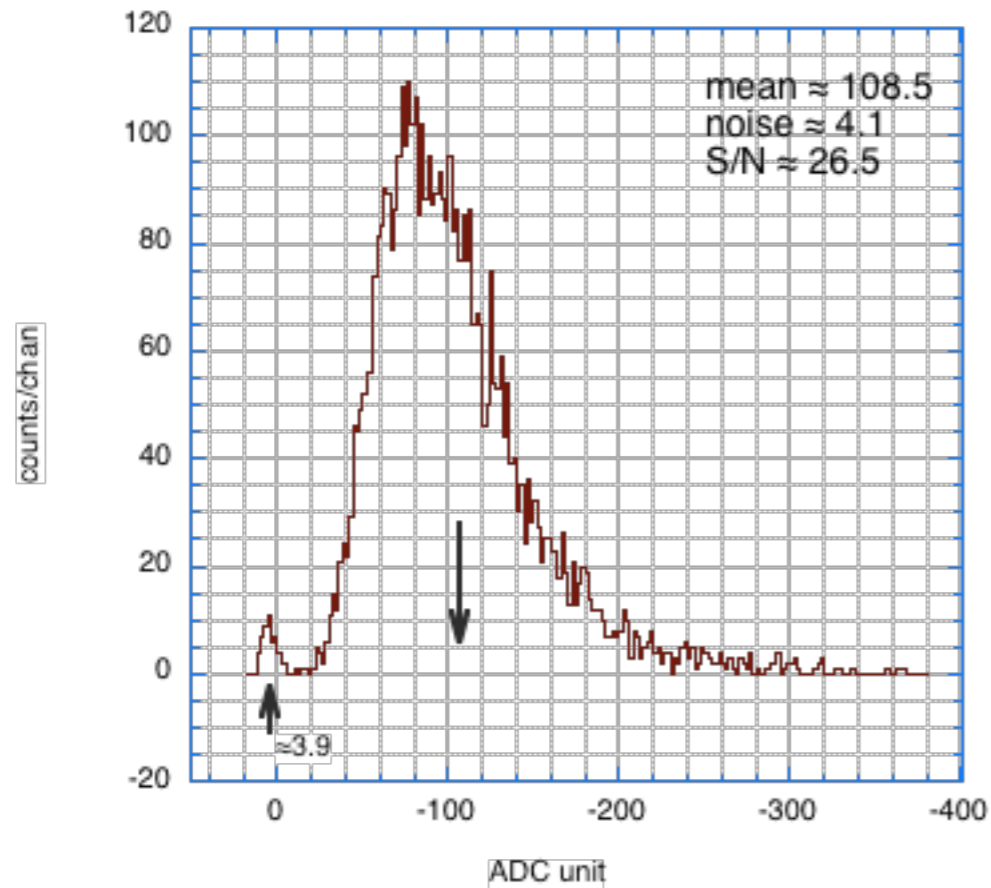
Ti-Au



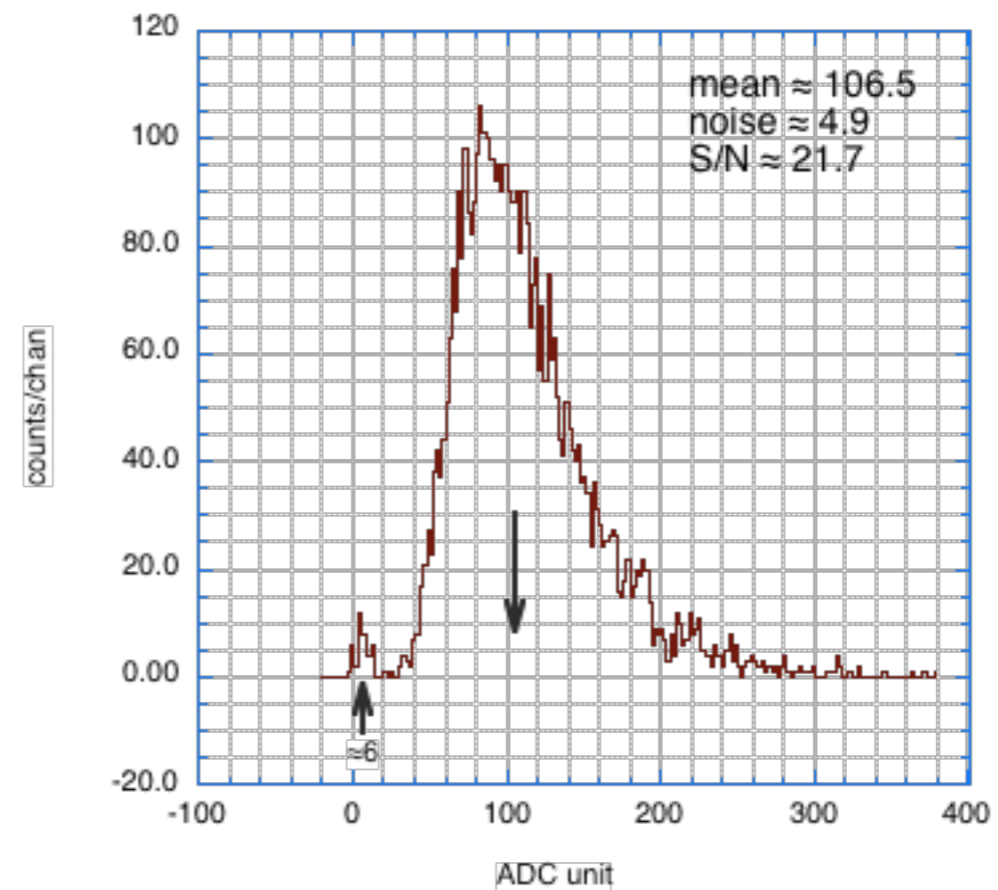
Results of measurements: β straggling

G F

+350 V



-350 V



Response of the graphite electrode detector at symmetric saturation voltages

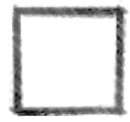
Conclusions

We have compared two diamond detectors made of the same quality material but with different electrode technique, the classical Ti-Au and the surface laser graphitization without metal covering.

The two detectors show small differences in charge collection and noise which however are ascribable to their different electrode geometry.

We must go ahead in the experimentation but the conclusion is the feasibility of laser graphite contacts on diamond to collect particle signals and the reliability of the graphite technique for the Chipsodia devices.

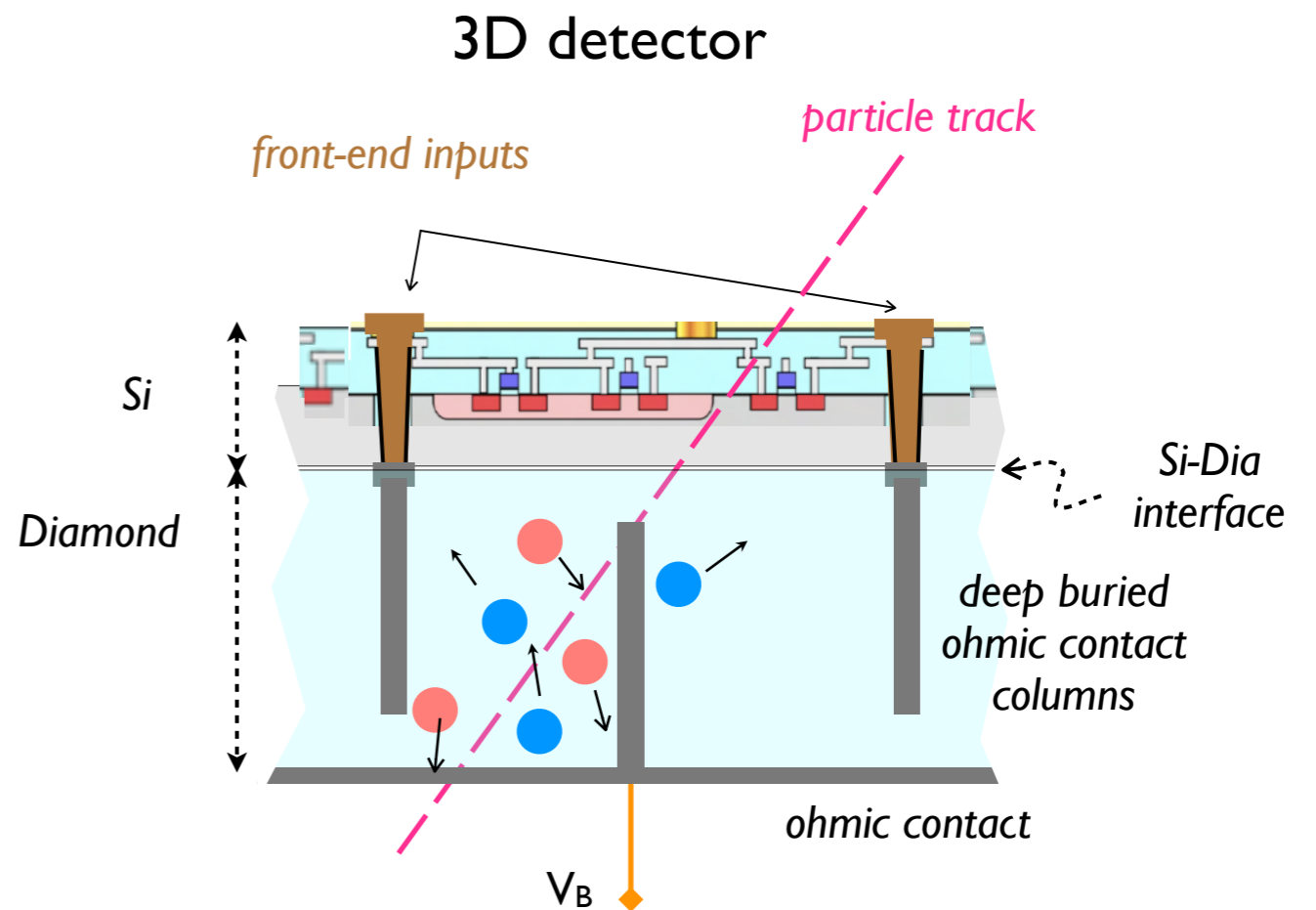
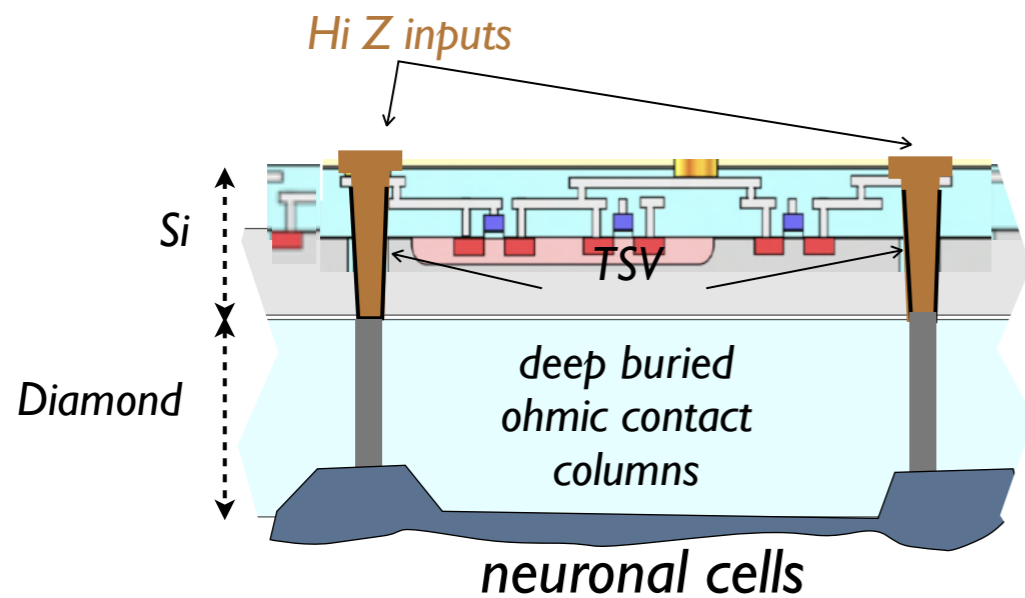
Back Up



SoD device concepts

The buried ohmic contact issue is the key stone for a variety of applications of a SoD architecture.

Below two designs considered by the CHIPSODIA collaboration: one more for MIP tracking (3D) and one for biological studies/applications



Graphyte, Diamond

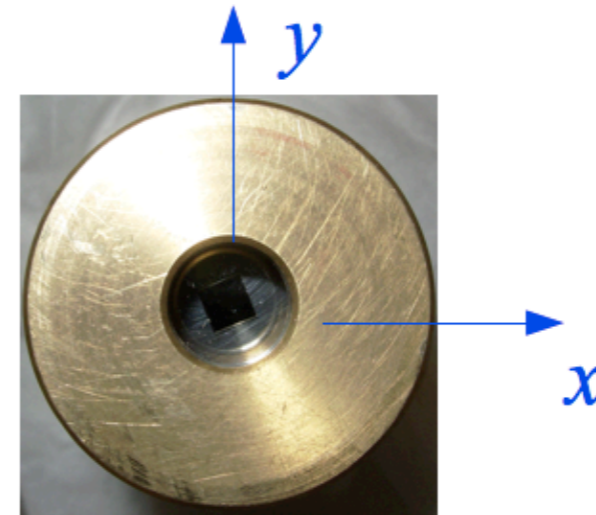
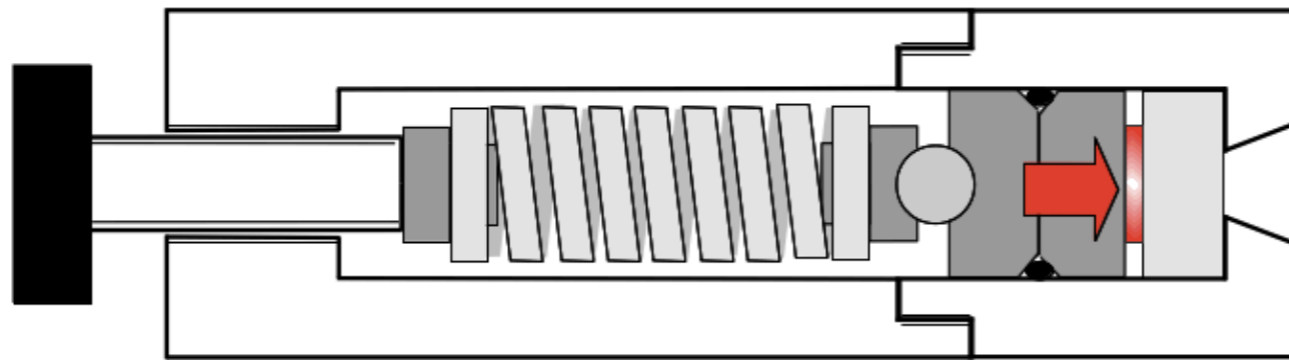
36 el/ μm

	Graphyte	Diamond
density/g/cm ³	2.01÷2.23	3.51
hardness/Mohs	1÷2	10
resistivity/ $\Omega\cdot\text{m}$	$\sim (2\div 3000)\times 10^{-6}$	$\leq 10^{18}$

Diamond		
e-h/ μm	e-h/500 μm	e-h/500 μm (gathered in poly)
36	1800	≈ 900

Si-Diamond bonding 1

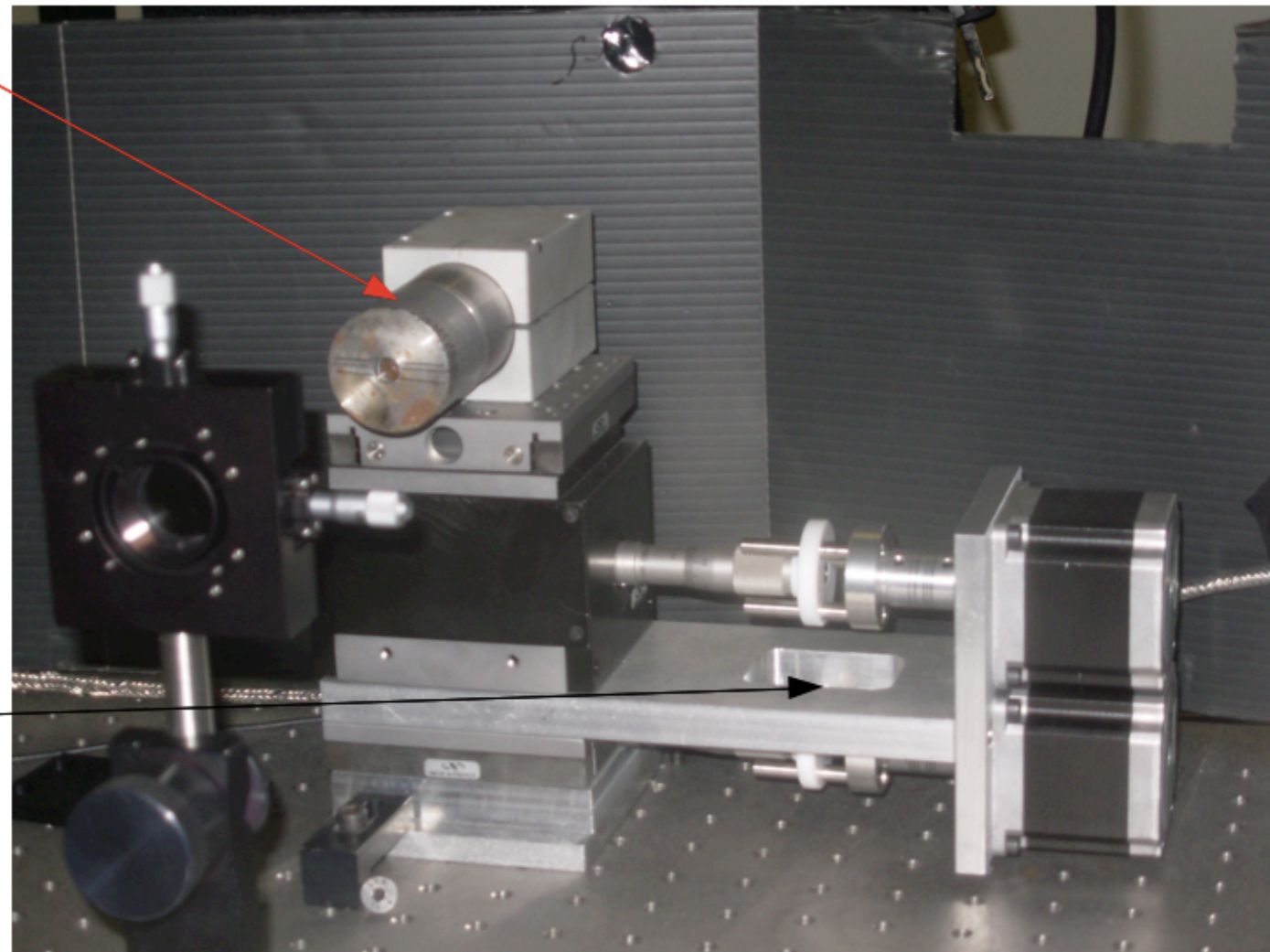
800 atm uniaxial stress



Sample holder

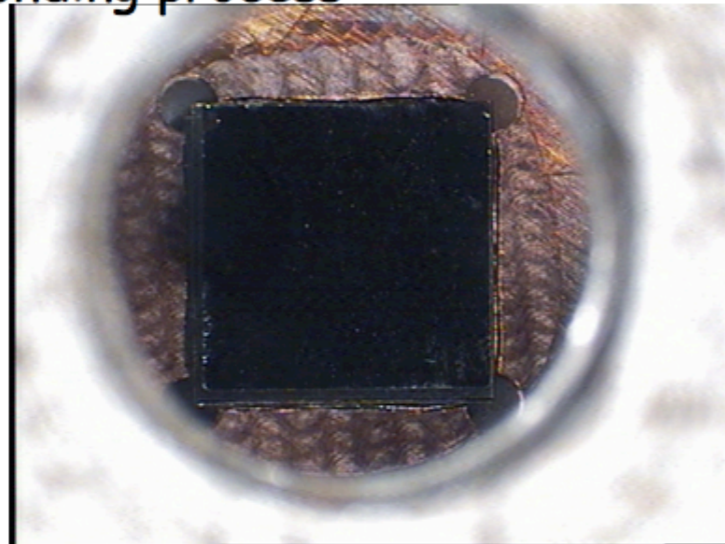
20 ps 355 nm laser pulses,
At 1 -0.5 J/cm²

Samples scanned by
the laser beam via xy
interfaced linear
stages



Si-Diamond bonding 2

Diamond-on-Silicon **seen through the quartz viewport, before the bonding process**



Clean and adherent surfaces, defects (white dots) of the order of the diamond roughness

SOD 23, 5 x5 mm²

Matrix of laser pulses for uniform illumination

rows of 16 pulses per mm,

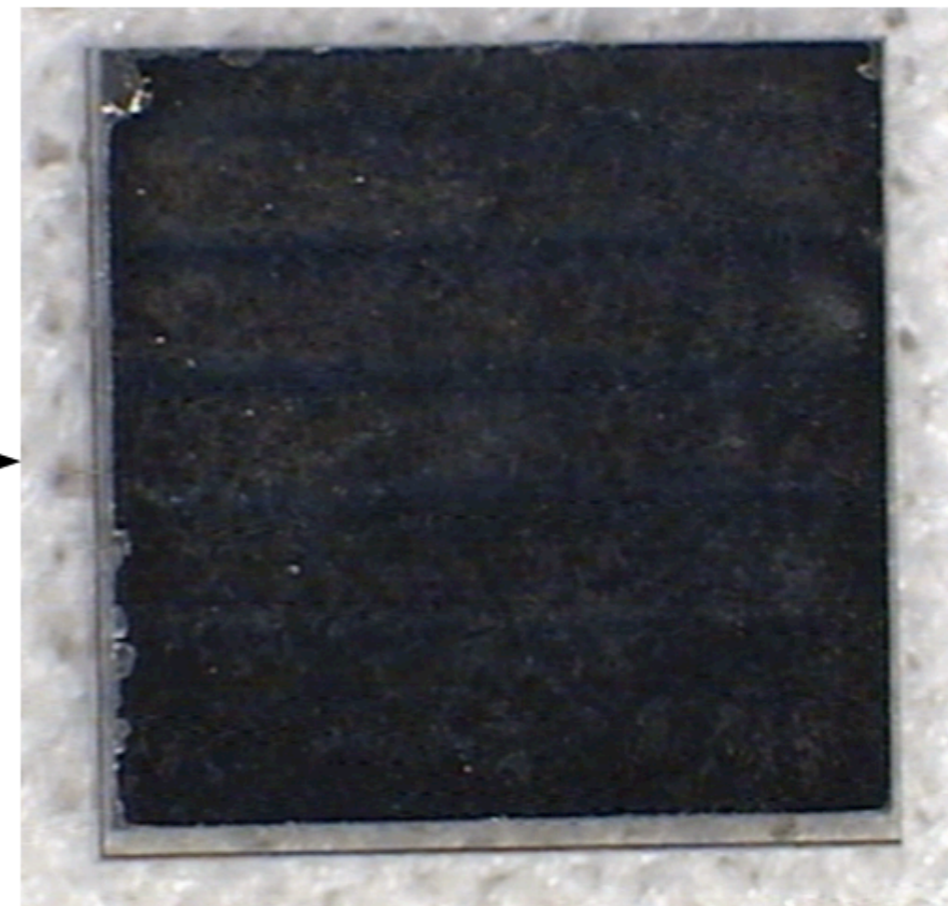
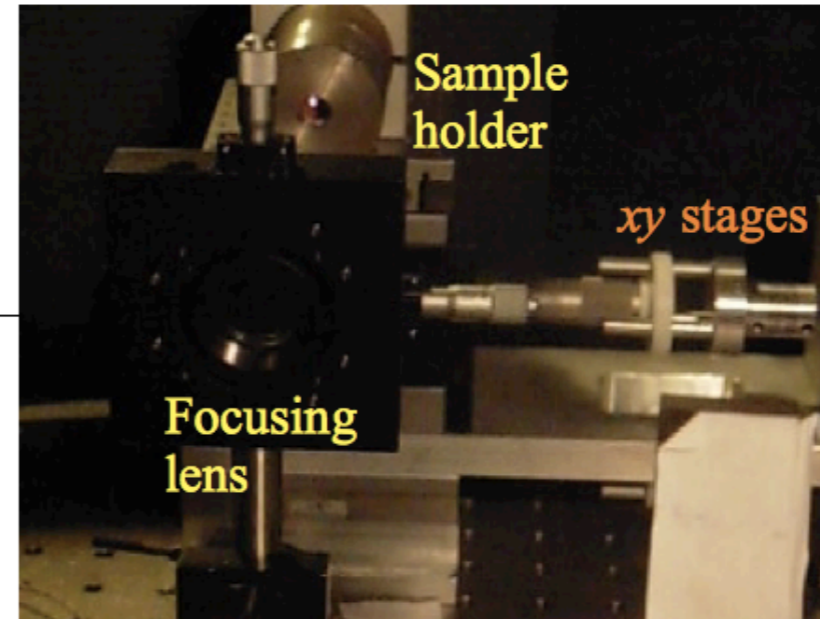
distance between rows 0.8 mm,

spot diameter 0.9 mm

3.5 mJ/pulse, 0.5 J/cm²

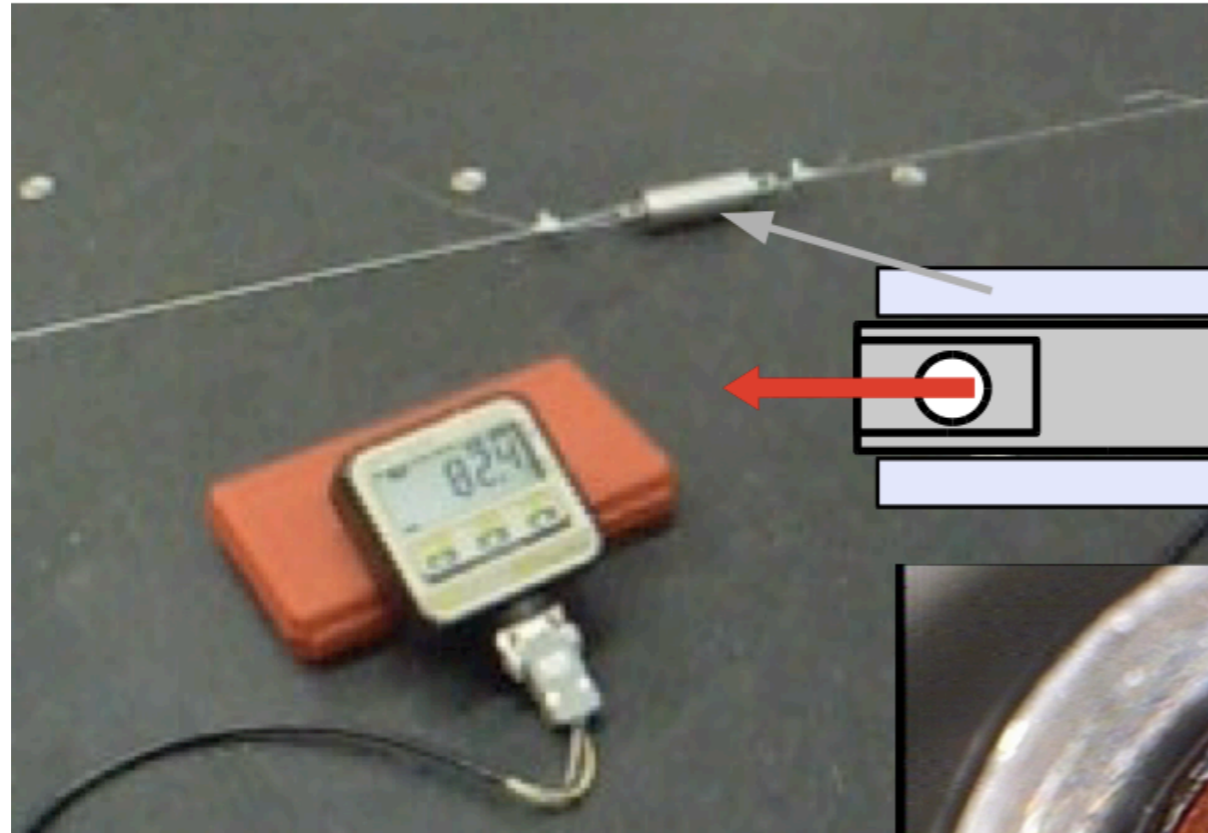
Calculated min value for bonding 0.22 J/cm²

Expected interface thickness: 50 nm

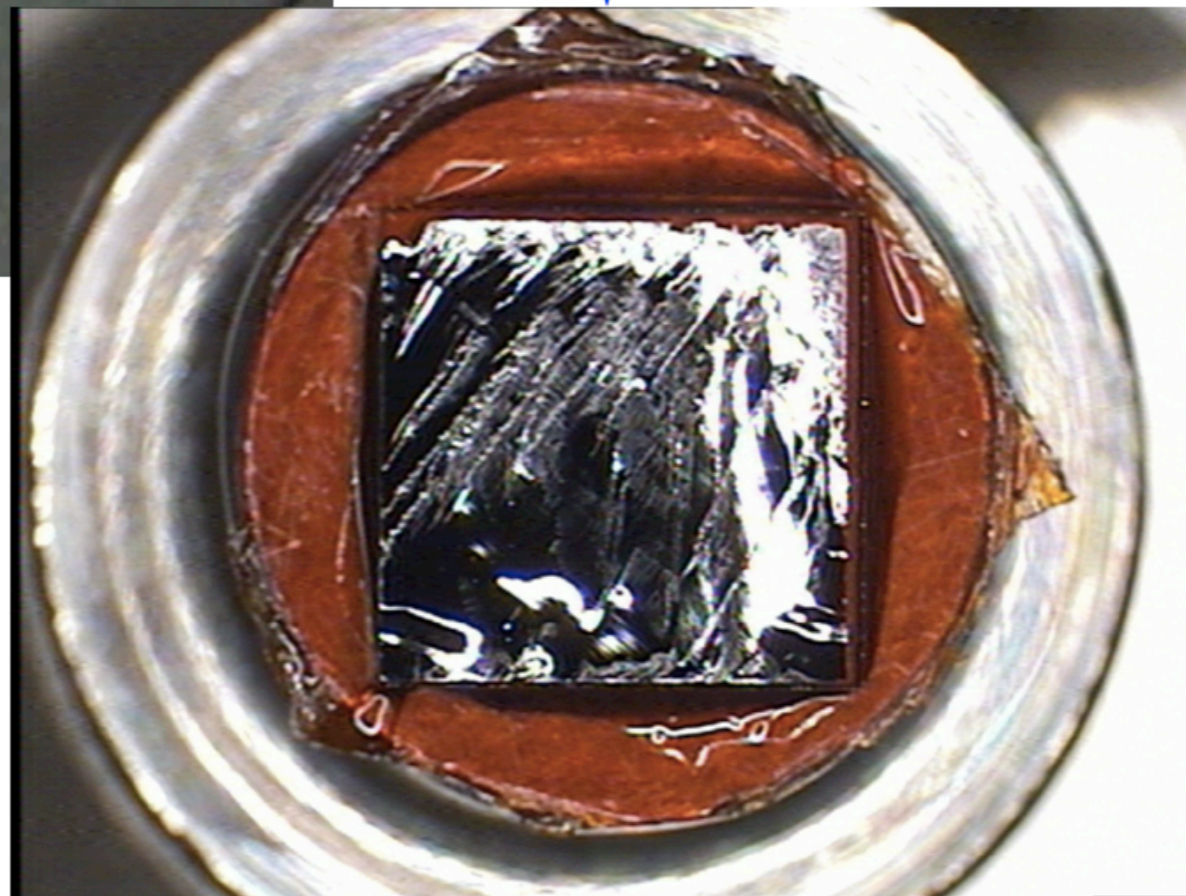
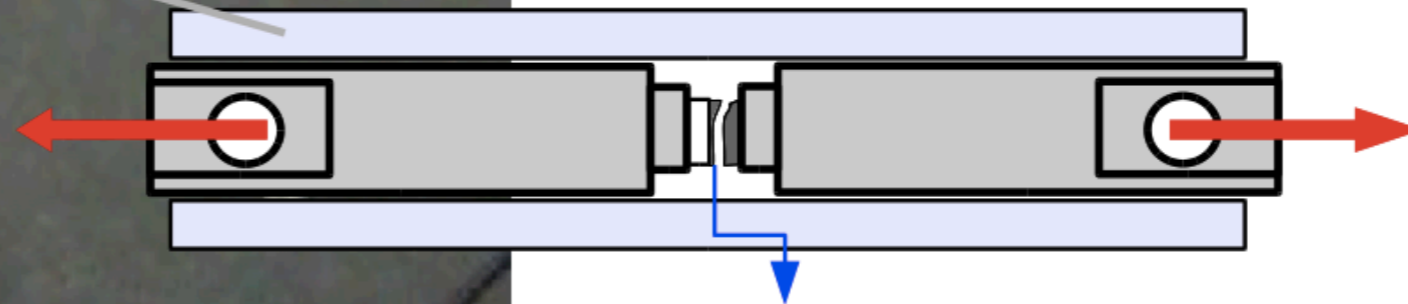


Si-Diamond bonding 3

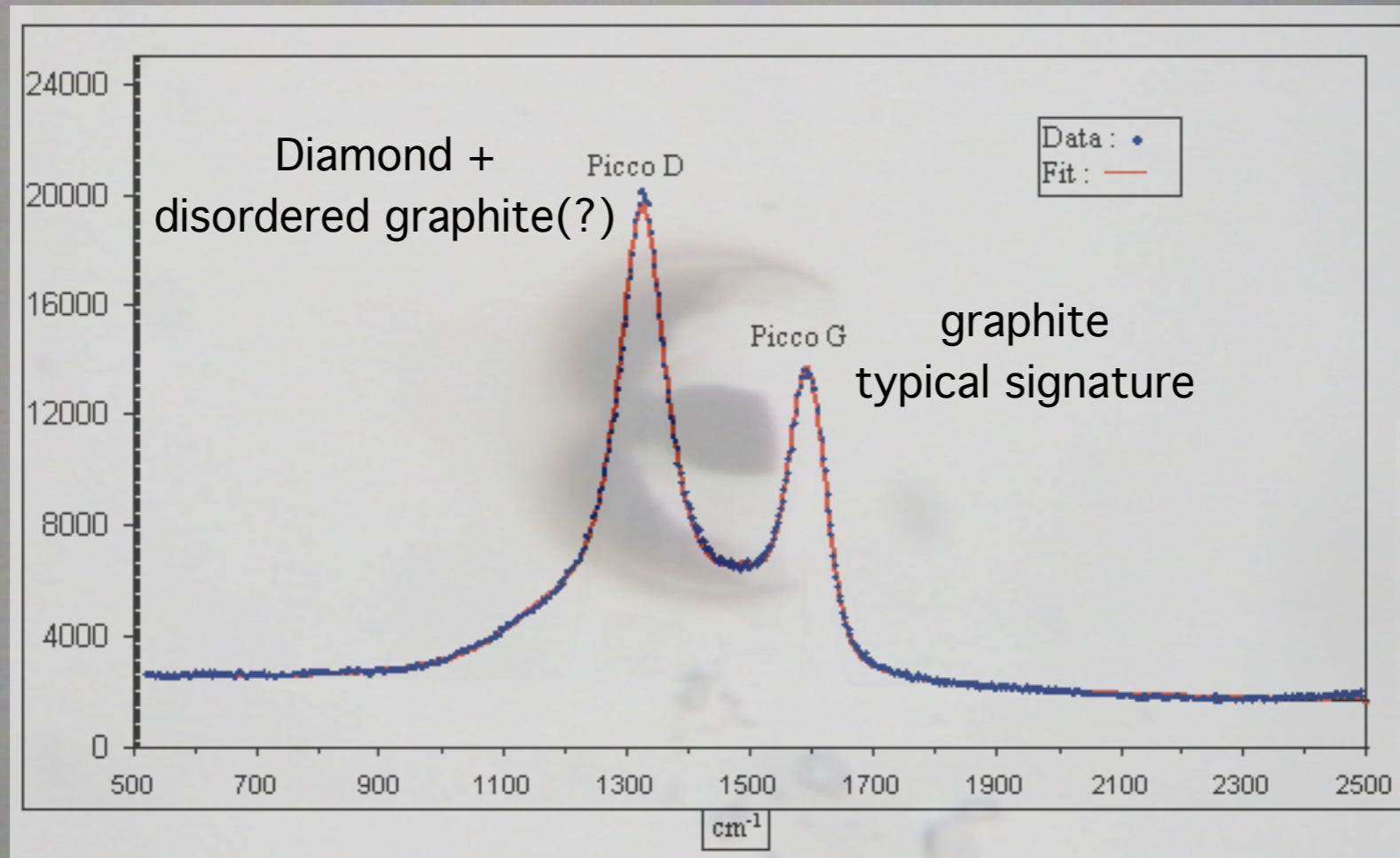
SOD 23 mechanical strength



After more than 82 kgp the silicon cracks internally and the binding interface remain intact



Raman Spectroscopy of Graphitic Columns



Geis et al. [1] : graphitization of diamond by pulses of 193 nm radiation from an ArF laser followed by chemical graphite removal resulting in modified 40-60 nm layers. Contacts to the layers with tungsten probes, silver paint, or electron beam evaporated Al are ohmic in character over a current range exceeding four orders of magnitude.

Kalish [2] high dose low energy B implantation followed by graphitization and chemical etching. This procedure leaves, after the graphite removal, only the tail of the B distribution hence creating a shallow (10 nm thick) heavily doped p-type layer on the sample surface. The p+ doping of diamond by this high dose surface implantation / annealing / etching technique can be used to realize excellent ohmic contacts to p-type diamond.

Tachibana and Glass states[3] that damaging diamond results in mechanically fragile and very noisy contacts.

According to this widespread opinion the standard ohmic contacts on diamond detectors are usually made, on oxygenated diamond surfaces, via the following methods:

(1) sputtering carbide forming metals as Ti, coating with Au Pt and low temperature annealing[3,4]

(non carbide forming metals result in Schottky barriers)

(2) sputtering Al[5] (simply evaporating Al results in surface trap states which strongly affect the electrical performances)

(3) Growing a diamond-like layer on the diamond surface and coating with gold [Diamond Detectors Ltd.]

[1] M. W. Geis, et al. Crystallographic, and optical properties of ArF laser modified diamond surfaces, Appl. Phys. Lett. 55 (22), 27 November 1989

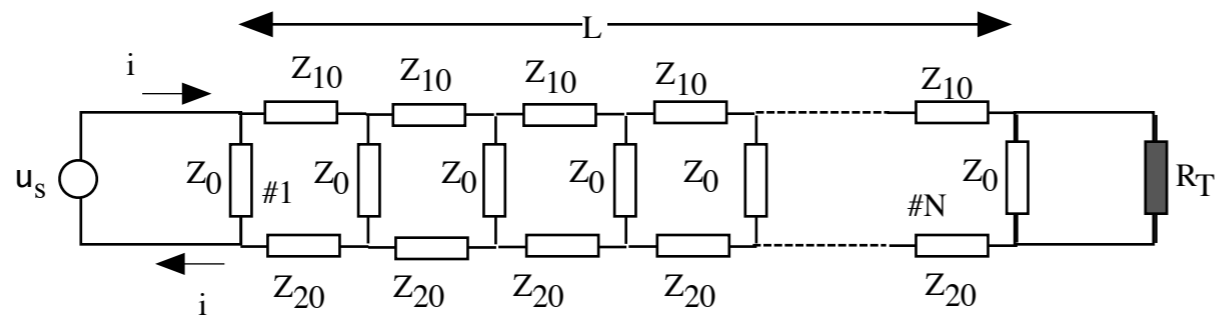
[2] R. Kalish, Doping of diamond, Carbon 37 (1999) 781 –785

[3] Takeshi Tachibana, Jeffrey T. Glass, Electric contacts to diamond, in “Diamond: Electronic Properties and Applications (Electronic Materials: Science & Technology) ,” Lawrence S. Pan, Don R. Kania, Editors, Springer; 1 edition (December 31, 1994)

[4] T. Tachibana, B. E. Williams, and J. T. Glass, Correlation of the electrical properties of metal contacts on diamond films with the chemical nature of the metal-diamond interface. II. Titanium contacts: A carbide-forming metal, Phys. Rev. B 45, 11975–11981 (1992)

[5] T. Tachibana, B. E. Williams, and J. T. Glass, Correlation of the electrical properties of metal contacts on diamond films with the chemical nature of the metal-diamond interface. I. Gold contacts: A non-carbide-forming metal, Phys. Rev. B 45, 11968–11974 (1992)

[6] W. Deferme , A. Mackova , K. Haenen , and M. Nesládek, Surface states and photo-induced charge transfer on oxygen-terminated chemical vapor deposition diamond, J. Appl. Phys. 109, 063701 (2011); doi:10.1063/1.3556748



$$Z_{10} = \frac{Z_1}{N} = \frac{Z_1 L}{L N} = \frac{Z_1}{L} \Delta x \quad Z_{20} = \frac{Z_2}{N} = \frac{Z_2 L}{L N} = \frac{Z_2}{L} \Delta x$$

$$Z_0 = Z N = Z L \frac{N}{L} = Z \frac{L}{\Delta x}$$

$$Z_{linea} = \frac{1}{j\omega C} + \frac{R}{3}$$