Laser Processing and Characterisation of 3D Diamond Detectors

ADAMAS
GSI meeting
3rd Dec 2015

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3D Diamond Group / RD42

Outline

- Laser setup for fabricating graphitic electrodes
  - How electrodes are fabricated
  - Issues with the setup
  - Planned upgrades

- Sample processed in Manchester

- Resistivity measurements

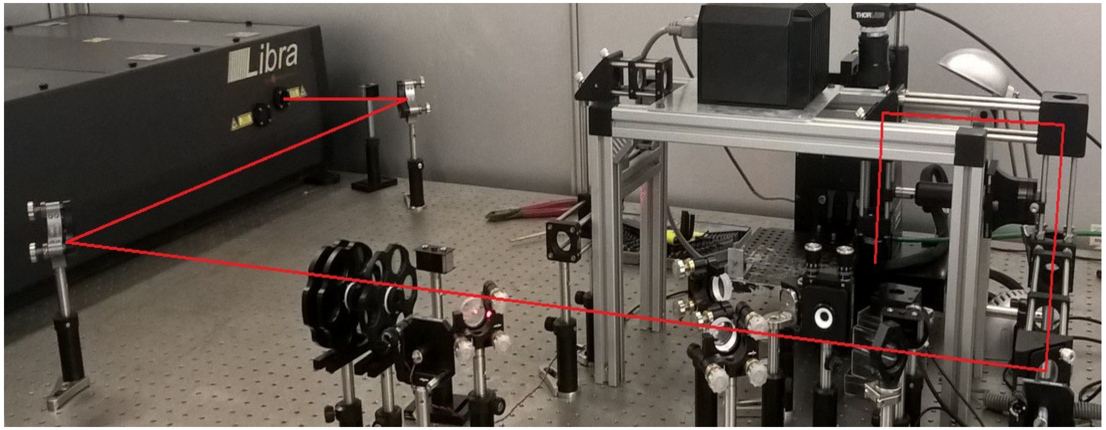
- Cross polarised microscope
  - Images of single and polycrystalline samples
  - Relative stress measurements

- Raman spectroscopy
  - Effect of fabrication parameters on diamond to graphite ratio

Laser - Setup

University of Manchester,
Laser Processing Research Center

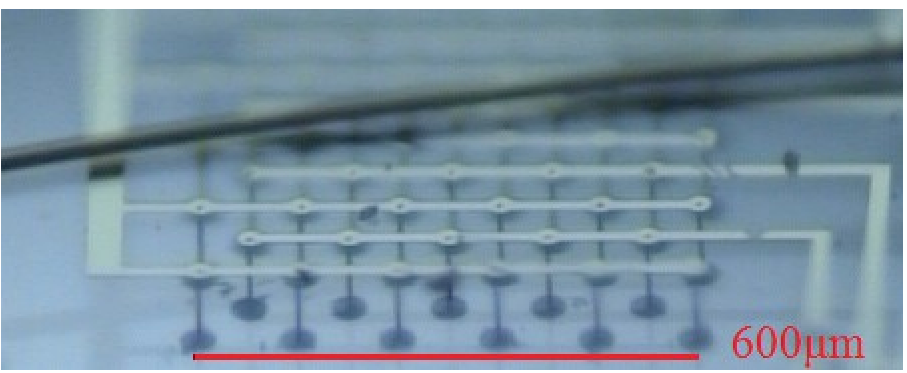
- Wavelength = 800 nm
- Repetition rate = 1 kHz
- Pulse duration = 100 fs
- Spot size = 4 μm
- Pulse Energy ~ 1 μJ

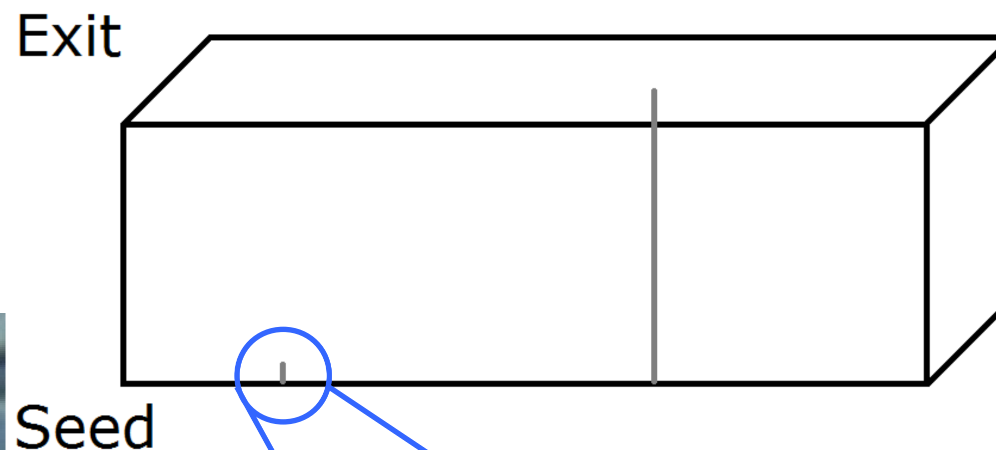


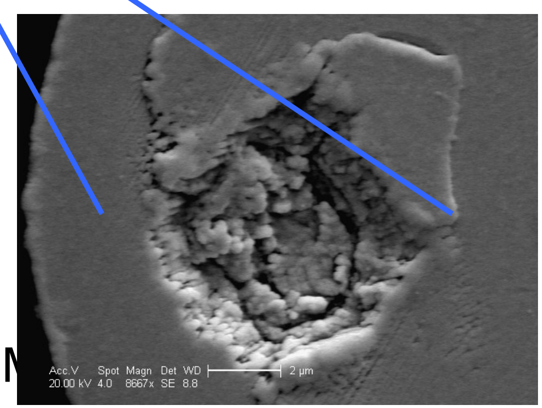
Laser - Fabrication

- Graphitic electrode formation starts from bottom

- Pulp formed on the seed side





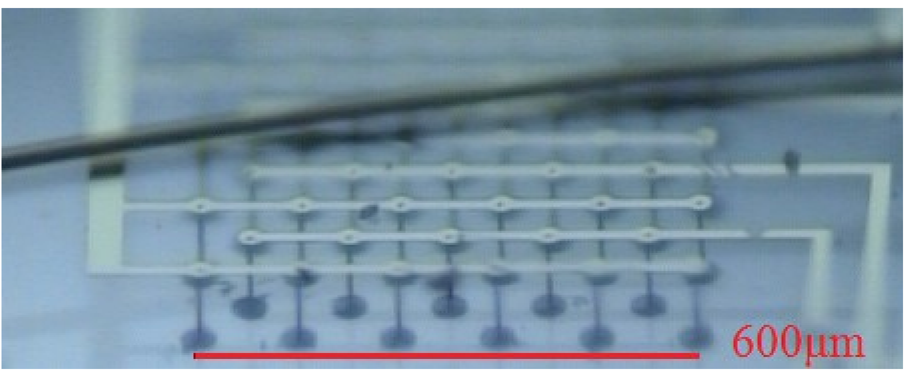


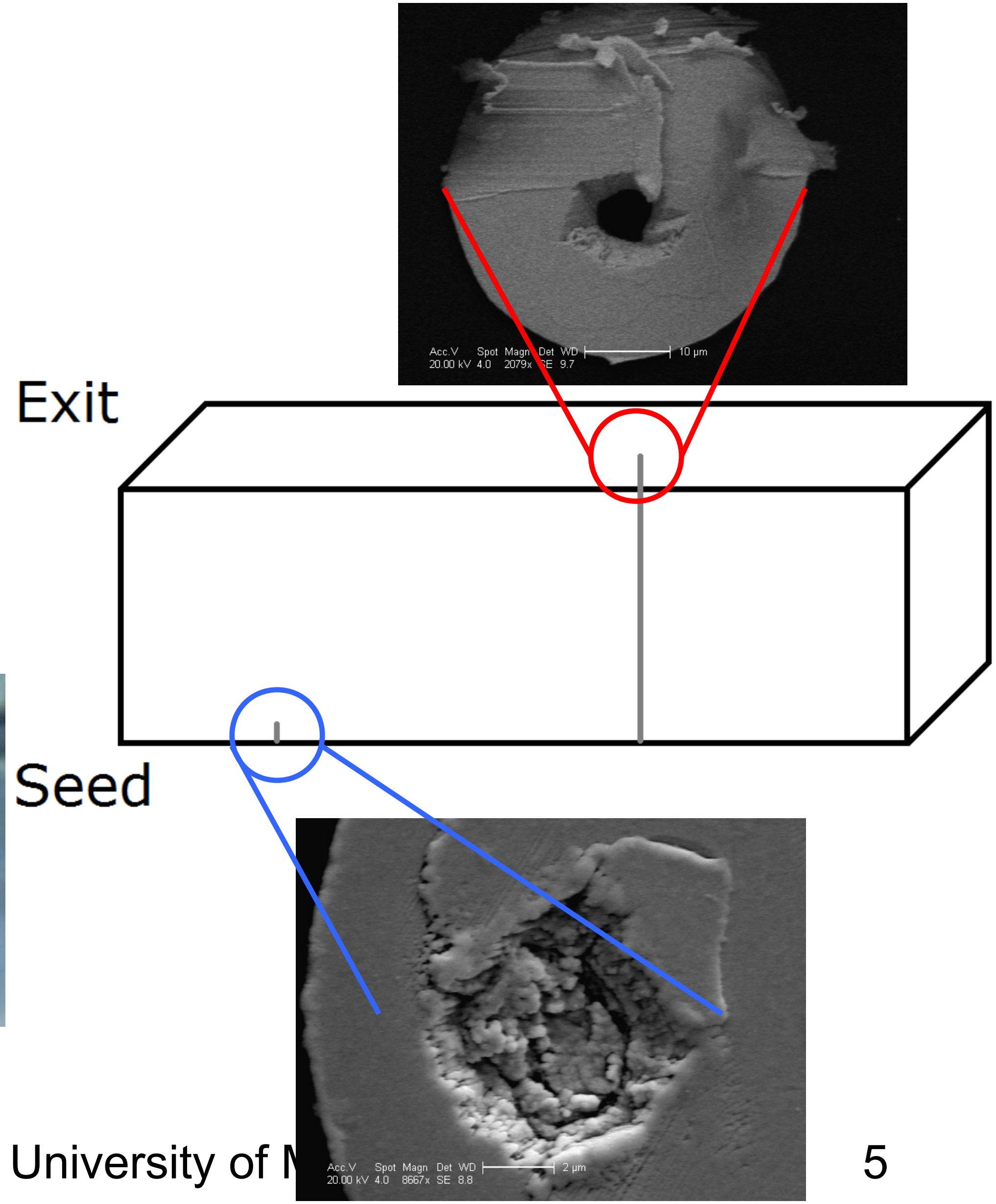
Laser - Fabrication

• Graphitic electrode formation starts from bottom

• Pulp formed on the seed side

• Craters formed on the exit side due to lower density of column material

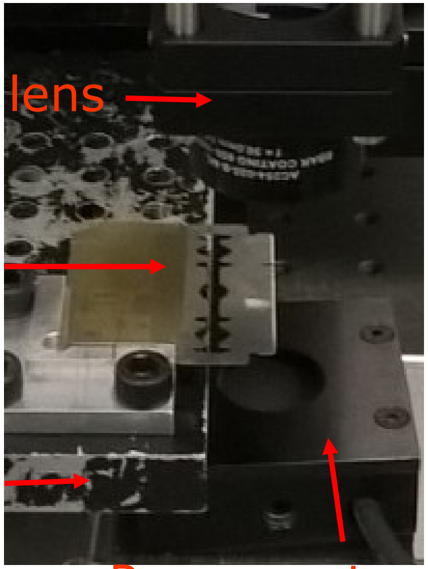




Laser - Beam Width

Measure beam width using knife-edge technique

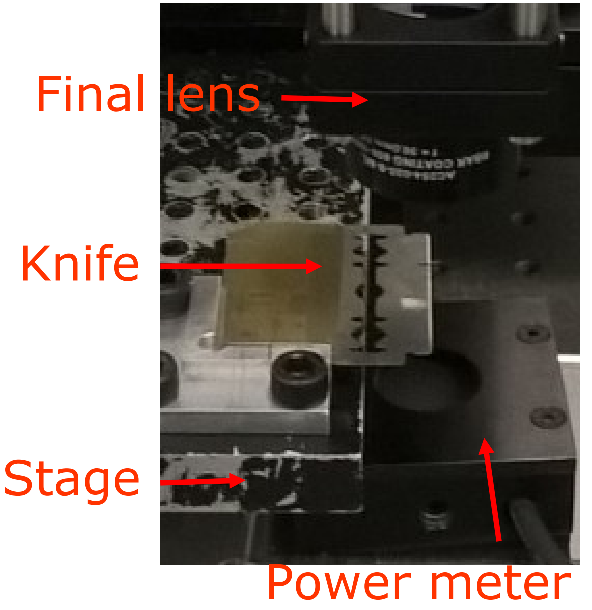
- Attach knife to stage and move across beam -> drop in power
- Record power -> build up error function profile
- Repeat at different z positions
    - Estimate focal point position
    - Estimate focal spot size - related to X_{90}-X_{10}

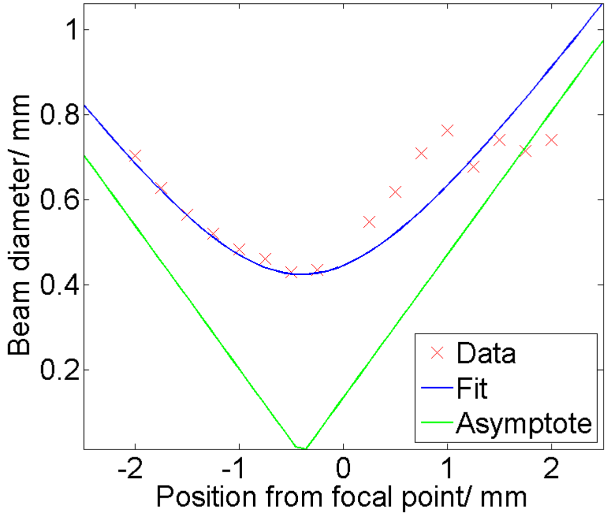


Laser - Beam Width

Measure beam width using knife-edge technique

- Attach knife to stage and move across beam -> drop in power
- Record power -> build up error function profile
- Repeat at different z positions
  - Estimate focal point position
  - Estimate focal spot size - related to X_{90}-X_{10}
- Focal spot size is very large
  - ~ 400 μm (4 μm in glass!)
- Asymmetry in the measurements
- No sign of converging close to focal point



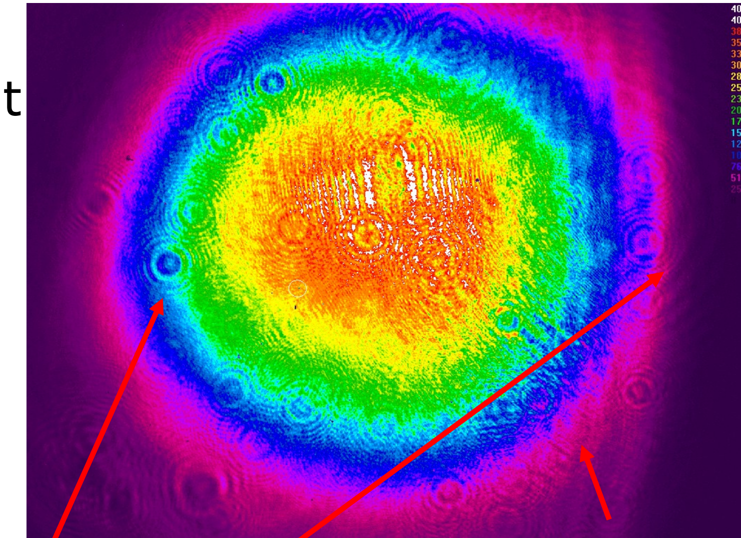


Laser - Beam Width II

Need to accurately measure near the focal point
- Use a beam profiler

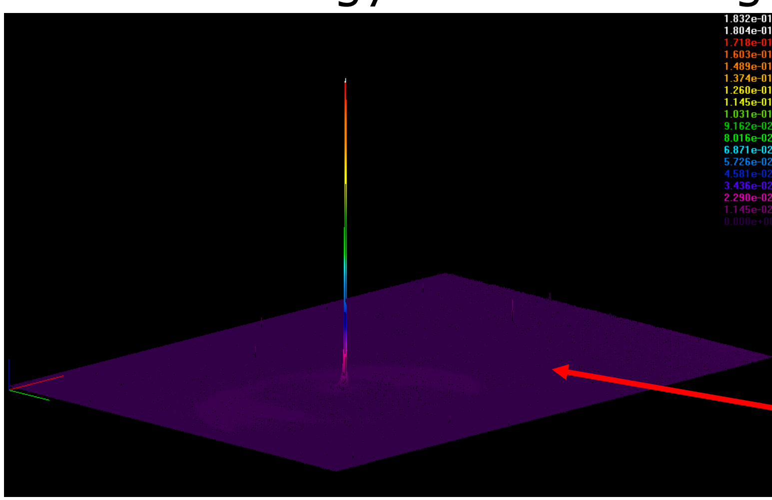
Beam profiler shows shape at the beam
- 6 mm before all optics

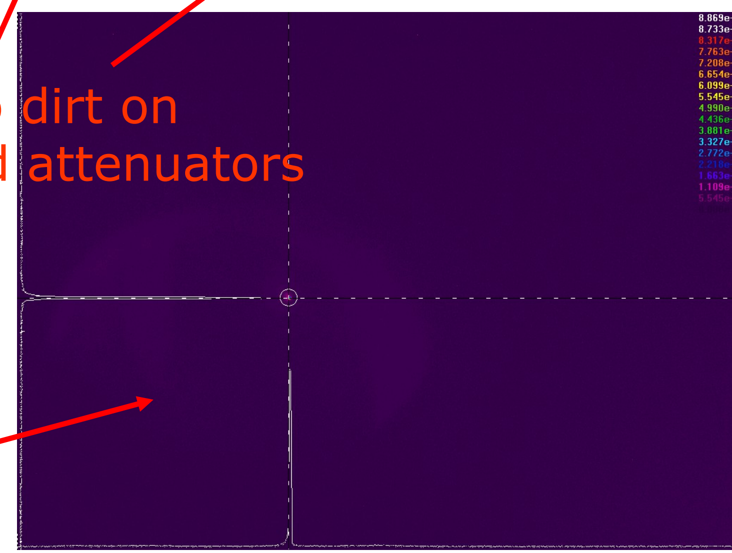
- 16 µm at focal point
    - 90% width of Gaussian
    - Size of spot in diamond smaller due to energy threshold for graphite



Before optics

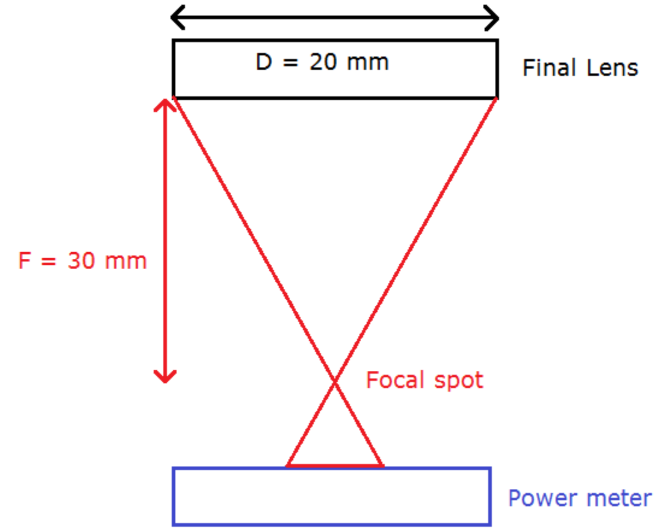
Spots due to dirt on 10+ year old attenuators





After optics

Laser - Issues



Spent most of 2014 and 2015 in repair
- Laser was moved to a new location

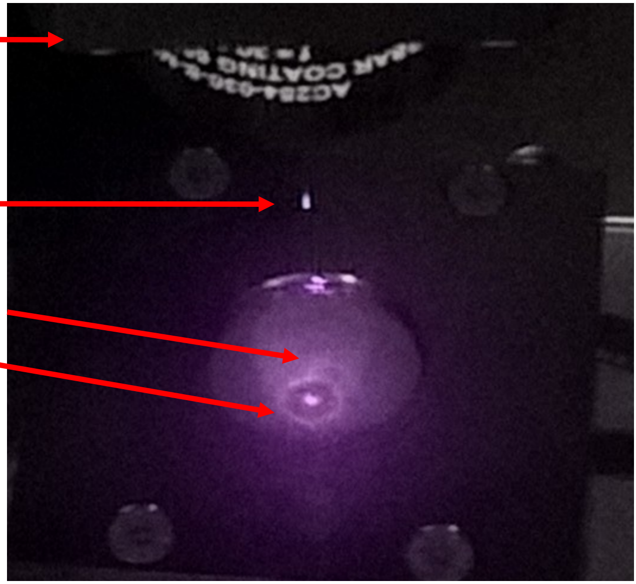
- Faulty power supply diagnosed

- Fixed as of Oct 2015

Final lens

Focal spot

Secondary rings

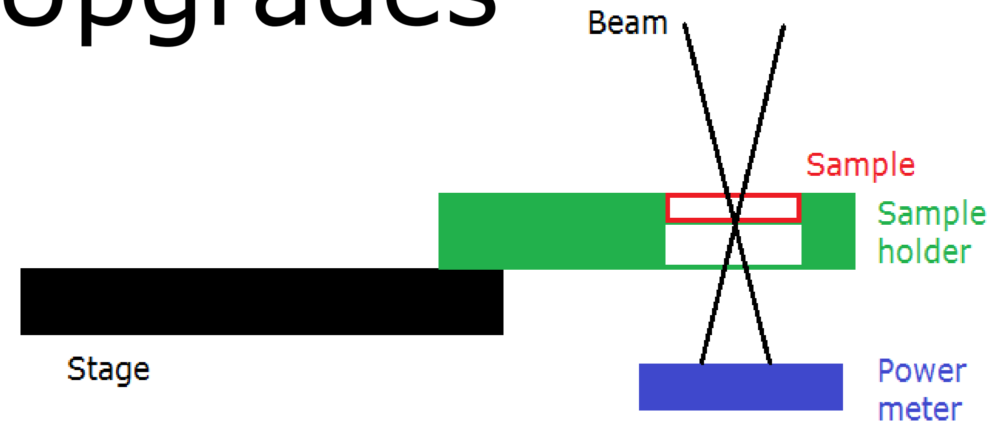


Power meter

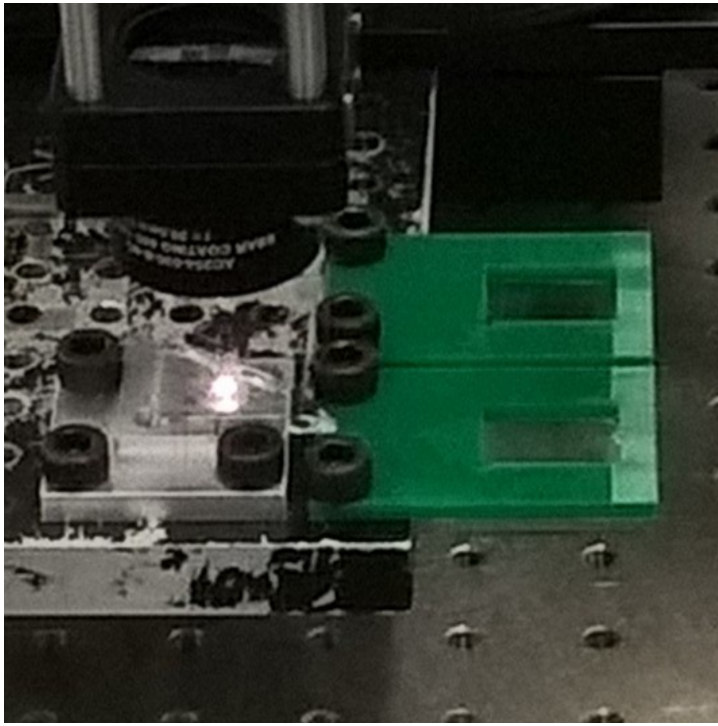
New optics installed
- Spot size reduced from 6 μm -> 4 μm

- Secondary rings due to airy disc

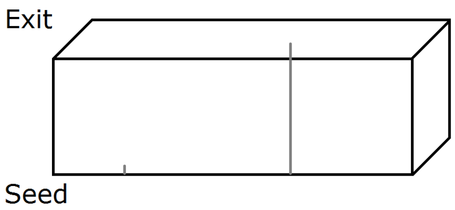
Laser - Upgrades

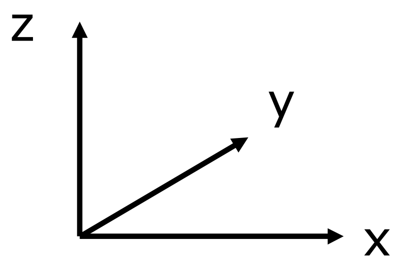


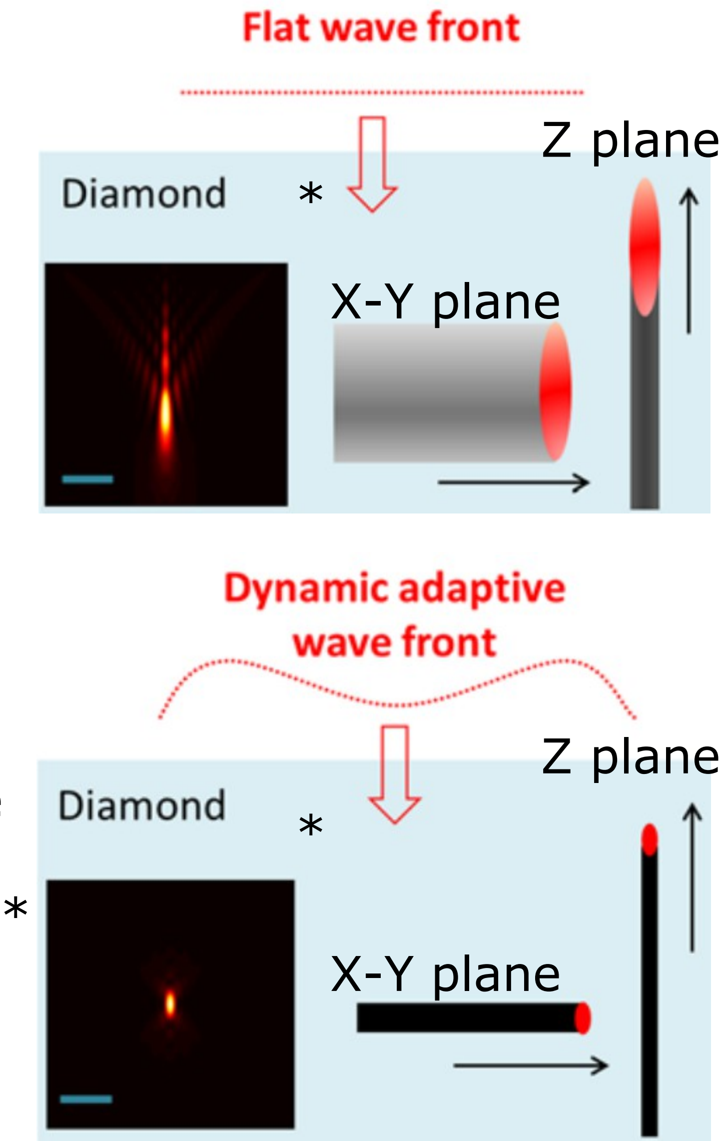
- New power meter
  - Measure lower power
  - Useful for knife edge measurements
- In-situ camera to track progress
- Record beam power during long-term drilling
  - Measure of beam stability
  - Identify malformed structures -> re-drill



Laser - Upgrades II





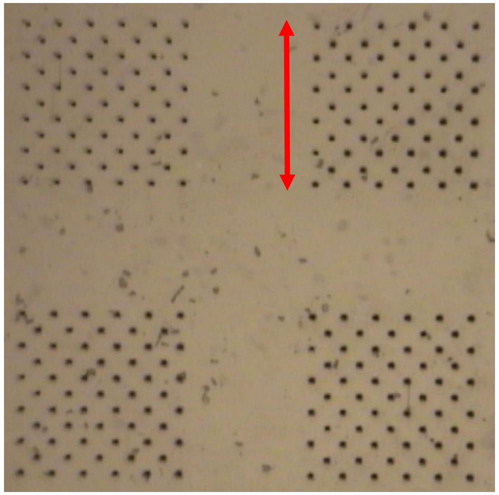


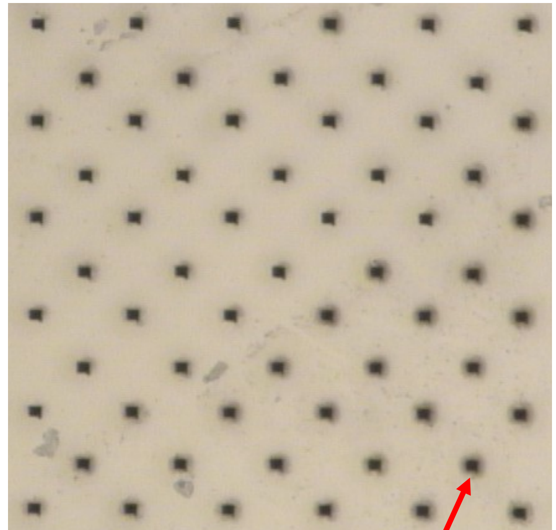
Spatial light modulator

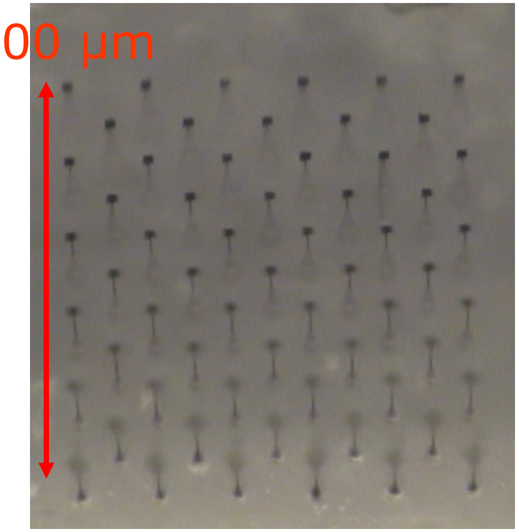
- Dynamic adaptive wavefront of beam with z
- Higher aspect ratio of graphitic electrodes
- Lower resistivity of electrodes
- Multiple passes to ensure well formed structure
- Established process by the University of Oxford*

*APPLIED PHYSICS LETTERS 105, 231105 (2014)

Laser - Upgrades III



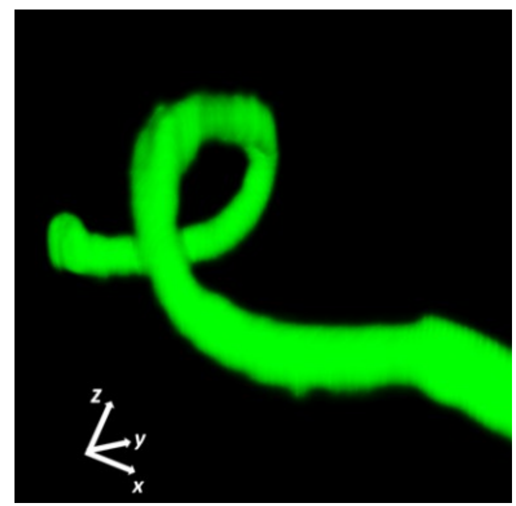




Received sample from Oxford made using the spatial light modulator

- Good optics -> diameter of O(1 μm)

- Can fabricate 3D wires
  - Form new graphitic structures in diamond
  - Even more radiation hard



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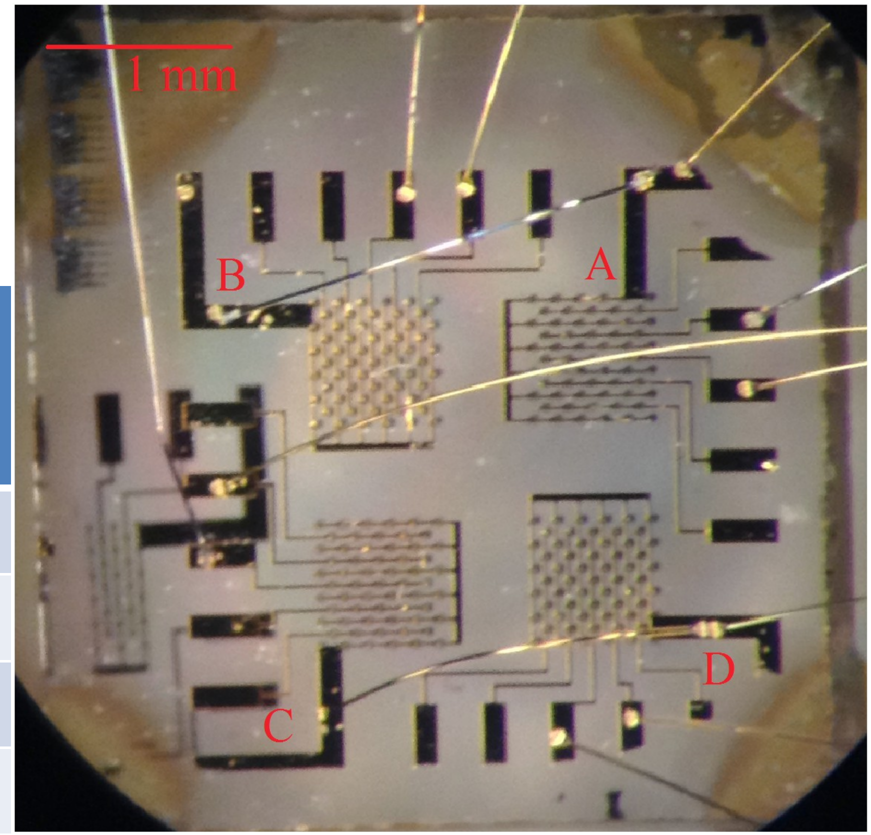
Sample

- Processed in Manchester
  - Laser drilling, metallisation, wire bonding

- Fabricate arrays of electrodes with different fluences
  - Diameter increases with fluence

Array	Fluence/ Jcm^{-2}	Column diameter/ μm
A	2.0	7.6 ± 0.8
B	3.0	10.2 ± 1.2
C	3.5	12.4 ± 0.9
D	4.8	18.0 ± 1.3

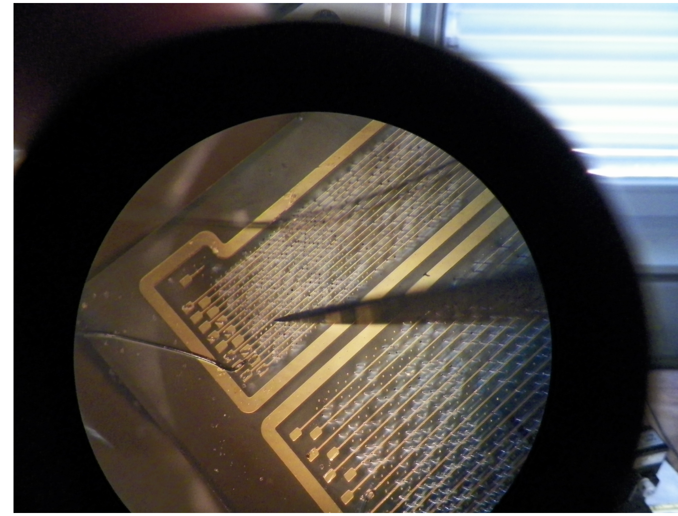


Resistivity - Results

Resistivity constant with fluence
- O(few Ωcm)

- Only 1 measurement for array A
    - Difficulty contacting electrodes

- c.f. Oxford sample
    - Diameter = 1.1 - 1.3 μm
    - Resistivity = 0.2 Ωcm



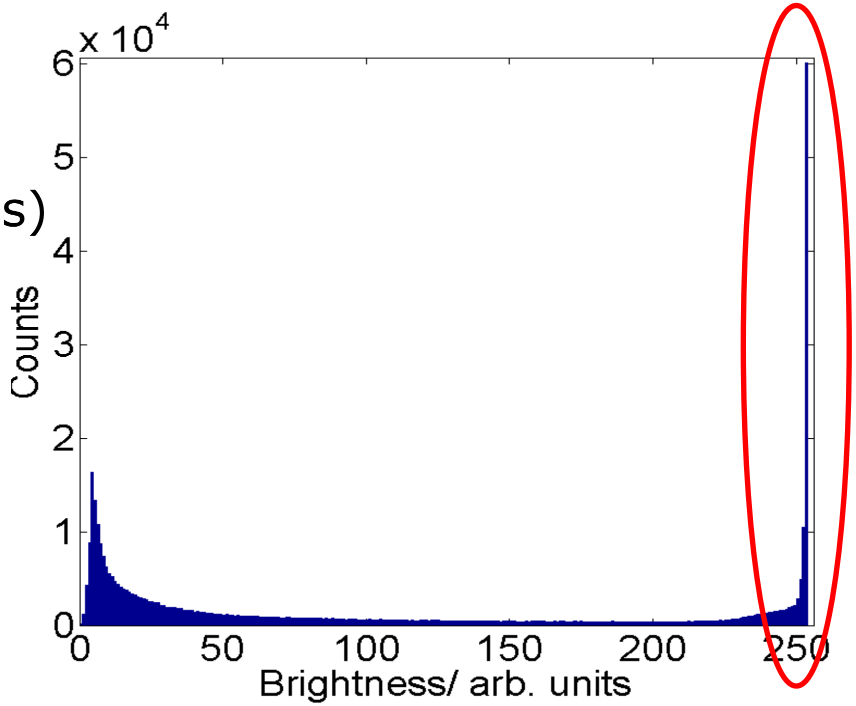
Array	Fluence/ Jcm^{-2}	Column diameter/ μm	Resistivity/ Ωcm
A	2.0	7.6 ± 0.8	0.75
B	3.0	10.2 ± 1.2	2.47 ± 0.86
C	3.5	12.4 ± 0.9	2.58 ± 0.89
D	4.8	18.0 ± 1.3	2.63 ± 0.71

Cross Polariser - Setup

• Use two polarisers, crossed by 90º

• Shouldn't see anything, except diamond has weak birefringence

• Any signs of dislocations (grain boundaries) stress due to presence of graphite can be seen

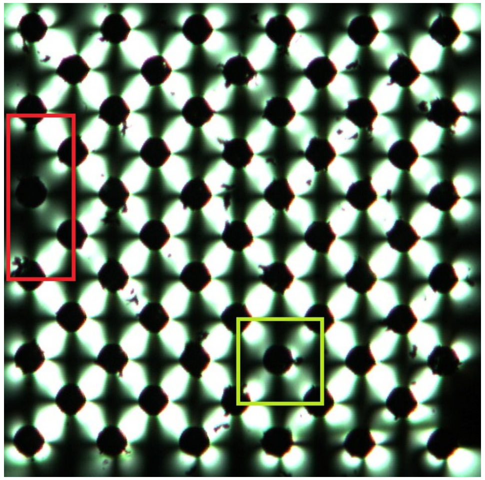


Diamond bulk under stress from electrodes
• Bright areas -> high stress

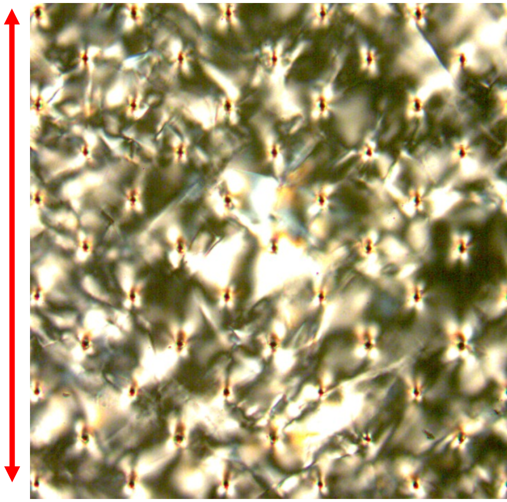
• Can estimate relative stress between arrays of electrodes
    • Plot histogram of brightness

Cross Polariser - Results

600 µm



600 µm

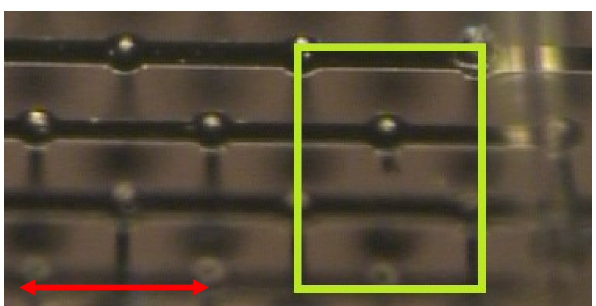


Single crystal (E6, 0.5 mm thickness):
high stress around electrodes

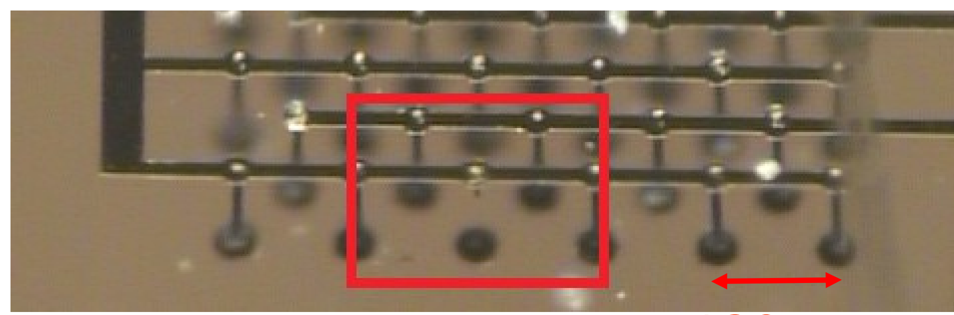
Poly-crystal (E6, 0.5 mm thickness):
high stress due to grain boundaries in diamond bulk

Useful diagnostic for electrode yield

- Malformed electrodes have low surrounding stress

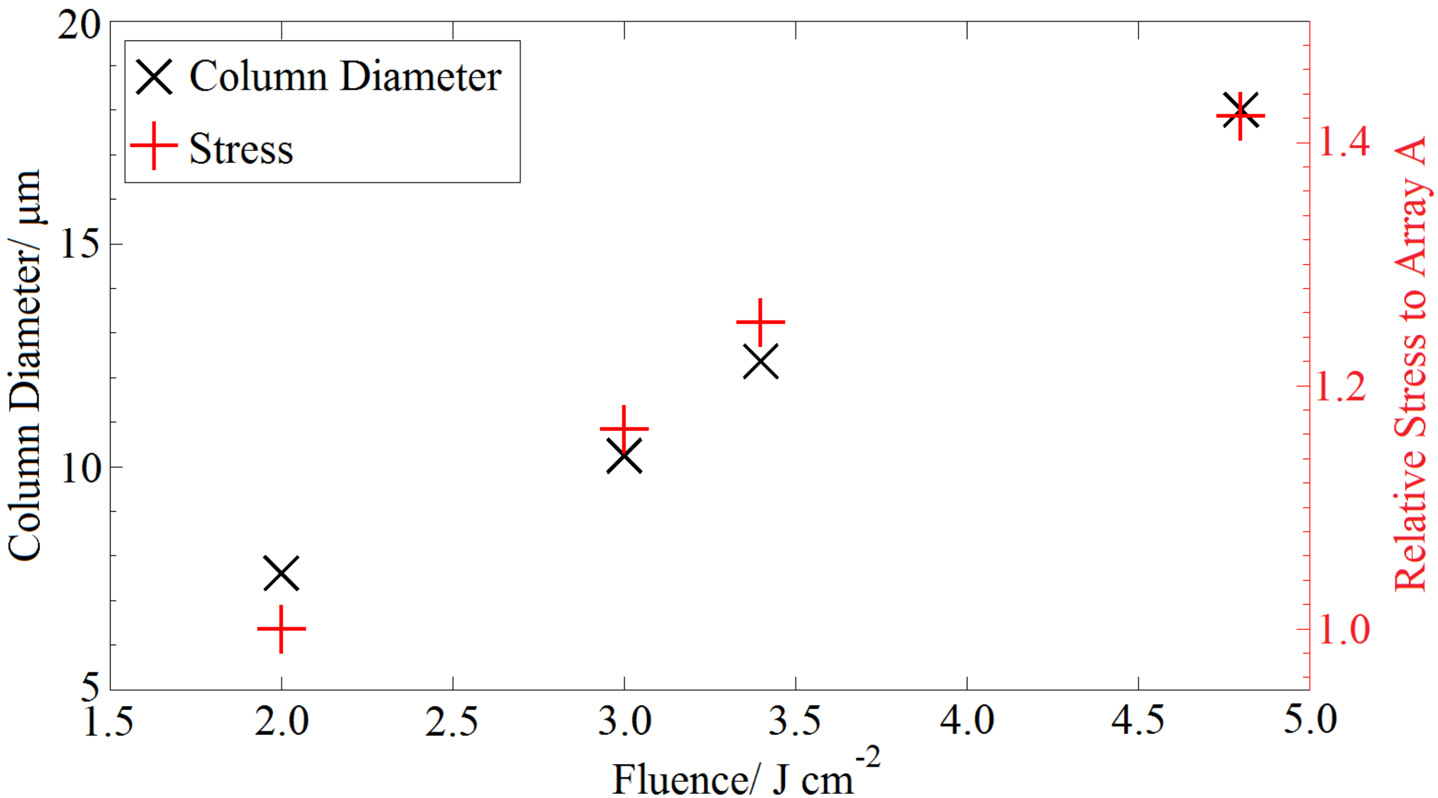


120 µm



120 µm

Cross Polariser - Results II



Use entire array for calculating stress
- All images are the same size

Stress increases with fluence
- Higher fluence -> higher diameter so higher stress surrounding electrode

Raman - Setup

University of Manchester,
Laser Processing Research Center

- Photons from laser incident on surface

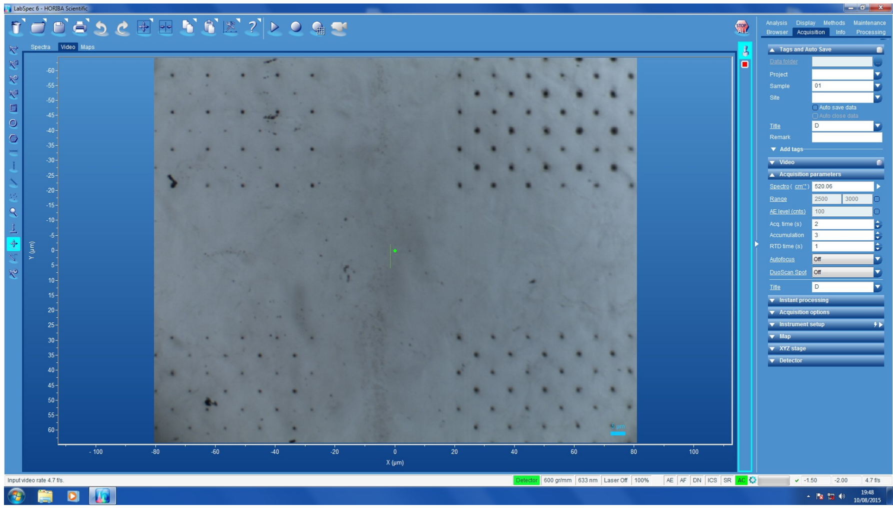
- Photons scattered by molecules within sample -> energy loss from vibrational levels

- Spectrometer analyses scattered photons -> spectrum characteristic of molecule

- HORIBA LabRam spectrometer

- 488, 633 nm wavelength sources

- Spot size = 5 μm



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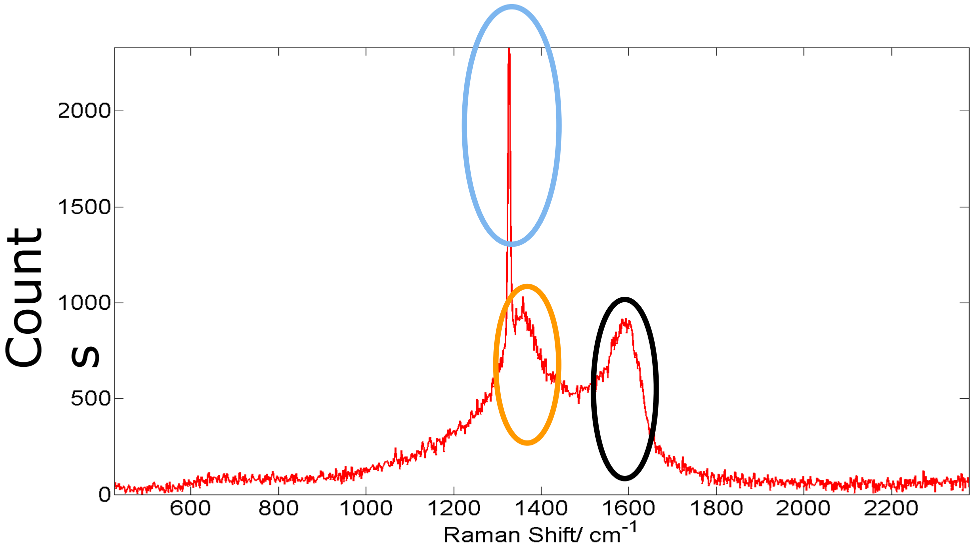
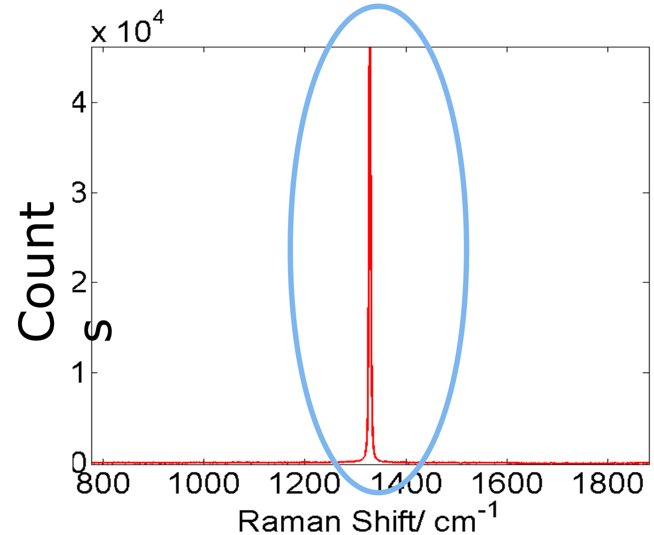
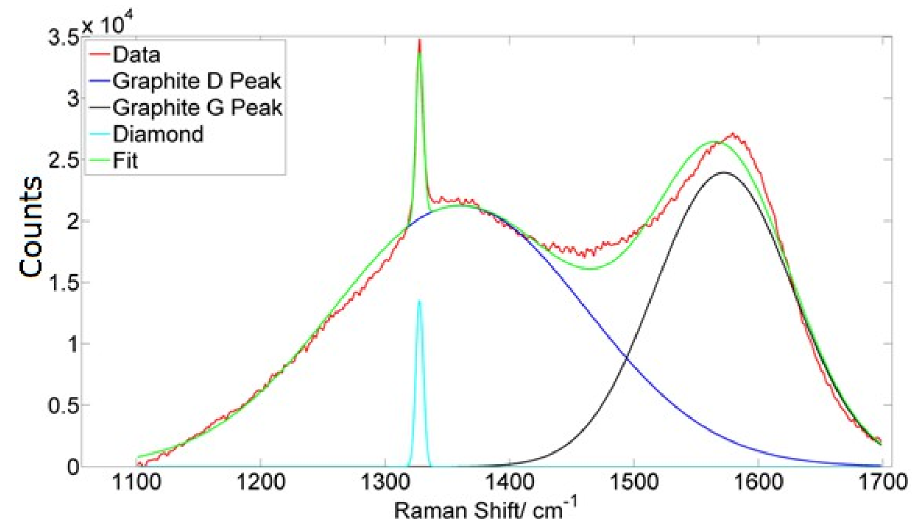
Raman - Results





See peaks at ~(1355 ± 50) cm^{-1} and (1580 ± 30) cm^{-1}

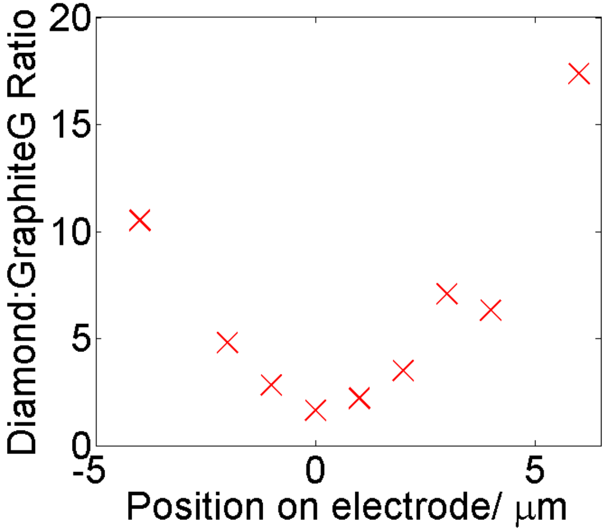
- D band graphite = 1350 cm^{-1}
- G band graphite = 1580 cm^{-1}
- Diamond peak = 1330 cm^{-1}



Apply fits to data

- Extract peak counts and peak width

Raman - Results II



Measure ratio of diamond to graphite G peak
- Estimate of material composition of electrodes

- Ratio reaches minimum at centre of electrode

Raman - Results II

Measure ratio of diamond to graphite G peak
• Estimate of material composition
of electrodes

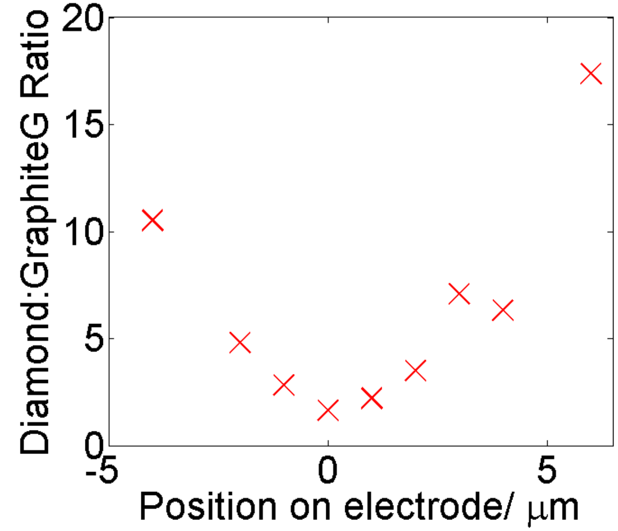
• Ratio reaches minimum at centre of electrode

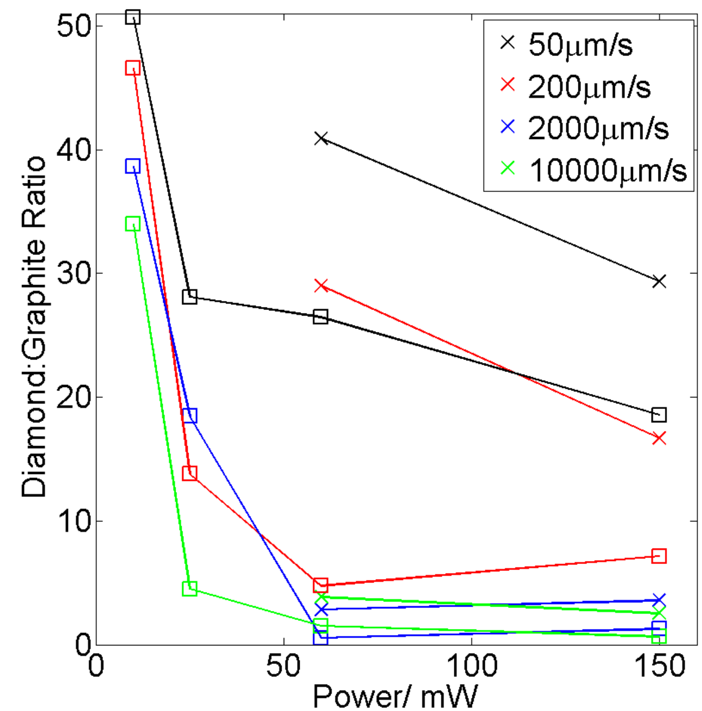
• Ratio is a diagnostic for deducing best fabrication parameters
(beam power, movement speed)

• More diamond on exit side (x)
vs seed side (□)

• High power (150mW), fast speeds
(10 mm/s) -> best results

• Results are for a poly sample from Göttingen





Summary

- Laser working again
  - Some work needed to correct for airy disc

- 3 useful diagnostic techniques available in Manchester
  - Resistivity - measure of electrical properties
  - Cross polariser - measure of stress in diamond
  - Raman - measure of electrode material content

- Implement improvements to laser setup in 2016
  - Record beam power
  - Install spatial light modulator

Backup - Knife edge

- Spot size of Gaussian beam varies with z:

$$w(z) = w_0\sqrt{\left(1 + \frac{z^2}{z_R^2}\right)} \qquad z_R = \frac{\pi w_0^2}{\lambda}$$

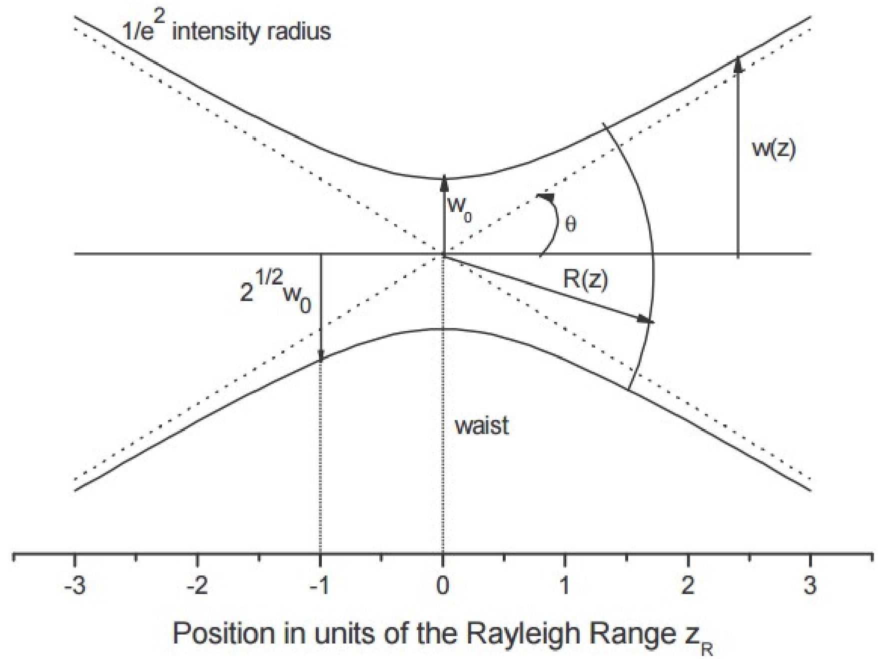
- For a beam propagating in z-direction:

$$I(x,y) = I_0 e^{-2x^2/w_x^2} e^{-2y^2/w_y^2}$$

$$P_{TOT} = \int_{-\infty}^{\infty}\int_{-\infty}^{\infty} I(x,y)\,dxdy = \frac{\pi}{2} I_0 w_x w_y$$

- It can be shown that the power at X is [1]:

$$P(X) = \frac{P_{TOT}}{2}\left[1 - erf\left(\frac{\sqrt{2}X}{w_x}\right)\right]$$



- The distance between 10% and 90% beam power relates to the spot size:

$$X_{10} - X_{90} = 1.28 w_x$$

[1]: http://massey.dur.ac.uk/resources/grad_skills/KnifeEdge.pdf

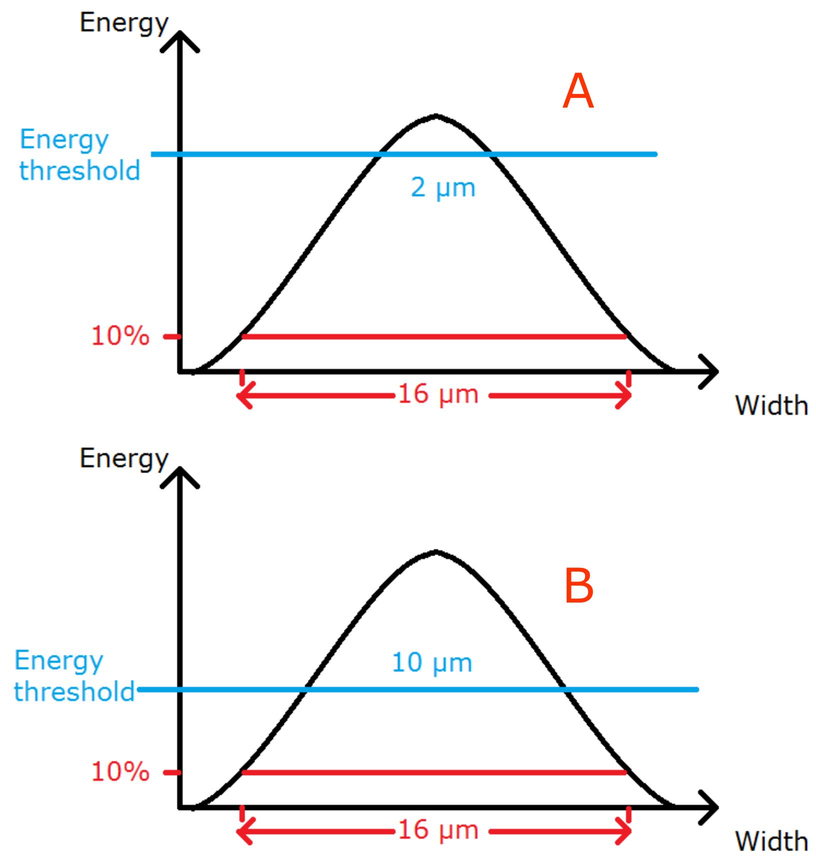
Backup - Graphitization

- Energy threshold for graphitization

- Below threshold -> no graphitization

- Above threshold -> spot size depends on how far above threshold peak energy is

- Peak energy of A < peak energy of B
    - Energy above threshold is lower -> smaller electrode diameter



Backup - Polariser