

2359-14

**Joint ICTP-IAEA Workshop on Physics of Radiation Effect and its Simulation
for Non-Metallic Condensed Matter**

13 - 24 August 2012

Ion beam lithography - II

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Ion beam lithography - II

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CNISM Consortium

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ICTP-IAEA Workshop, Trieste, 13-24 August 2012



Outline

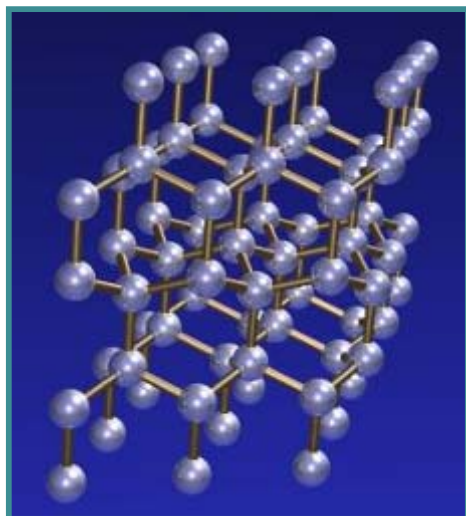
- **Diamond**
 - **Synthesis**
 - **Properties**
 - **Applications**
- **IBL in diamond**
 - **MeV ion lithography in diamond**
 - **keV ion beam lithography in diamond**
- **Activities at the University of Torino**
 - **Electrical features**
 - **Optical features**
 - **Microfluidics**

Outline

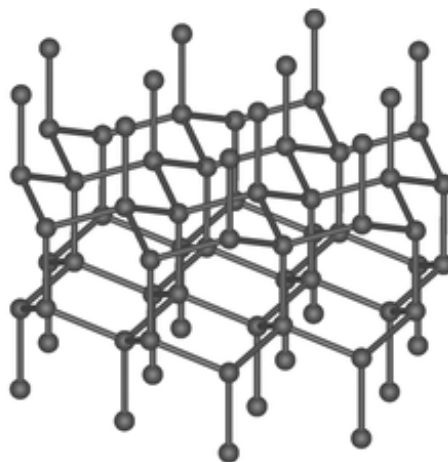
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Diamond

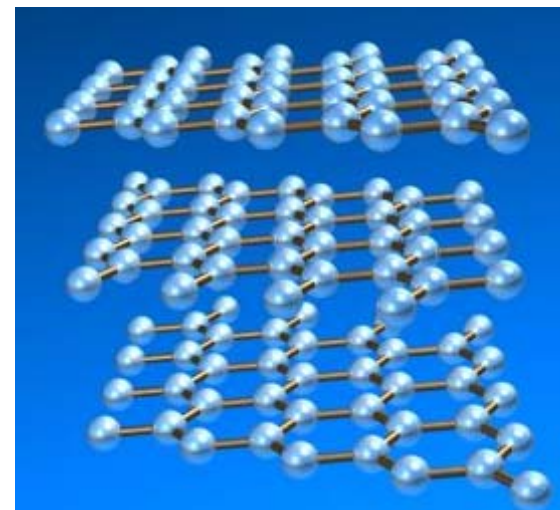
αδάμας (*indestructible*)



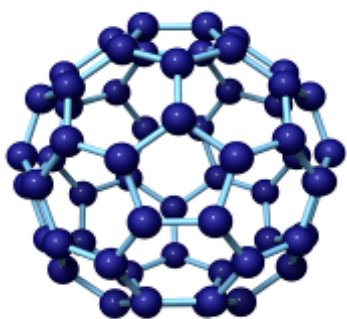
diamond



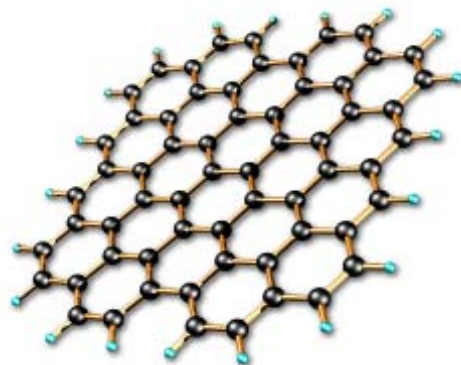
lonsdaleite



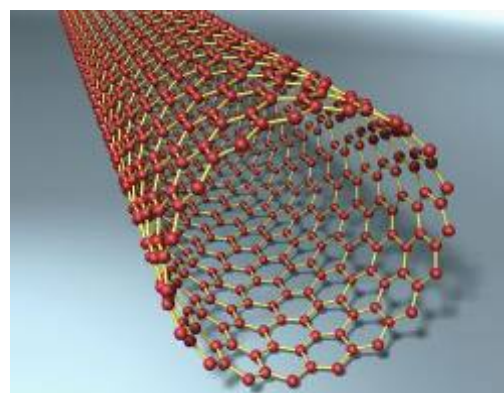
graphite



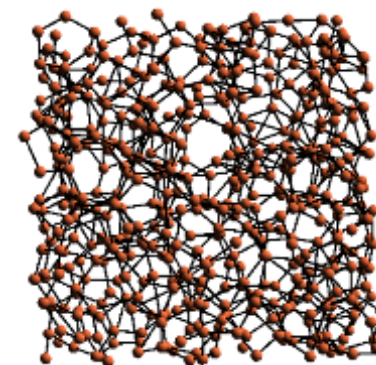
fullerene



graphene



nanotube



amorphous carbon 4

Diamond

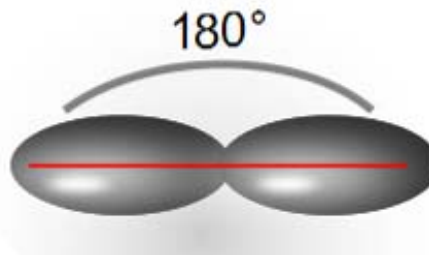
Carbon

Periodic table showing the position of Carbon (C) in group 14, period 2. The table is color-coded: yellow for metals, cyan for transition metals, and pink for non-metals. Carbon is highlighted in a blue box.

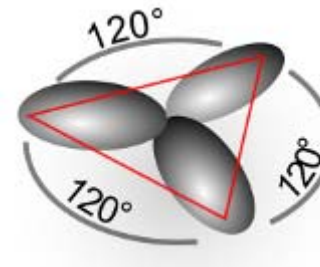
6
C
 Carbon
 12.0107



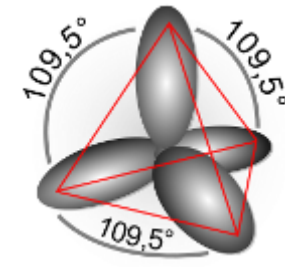
Three types of hybrid orbitals



sp^1



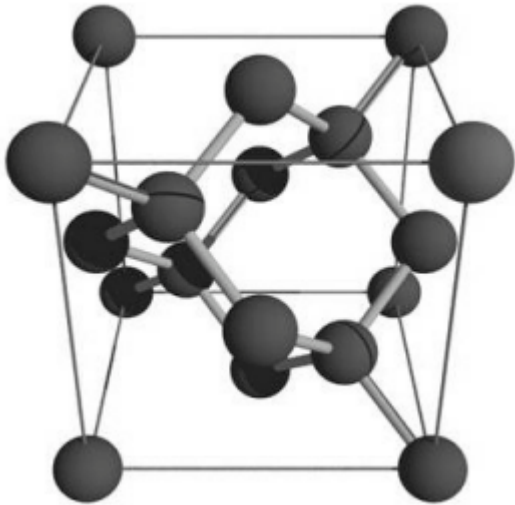
sp^2



sp^3

Diamond

Crystal structure

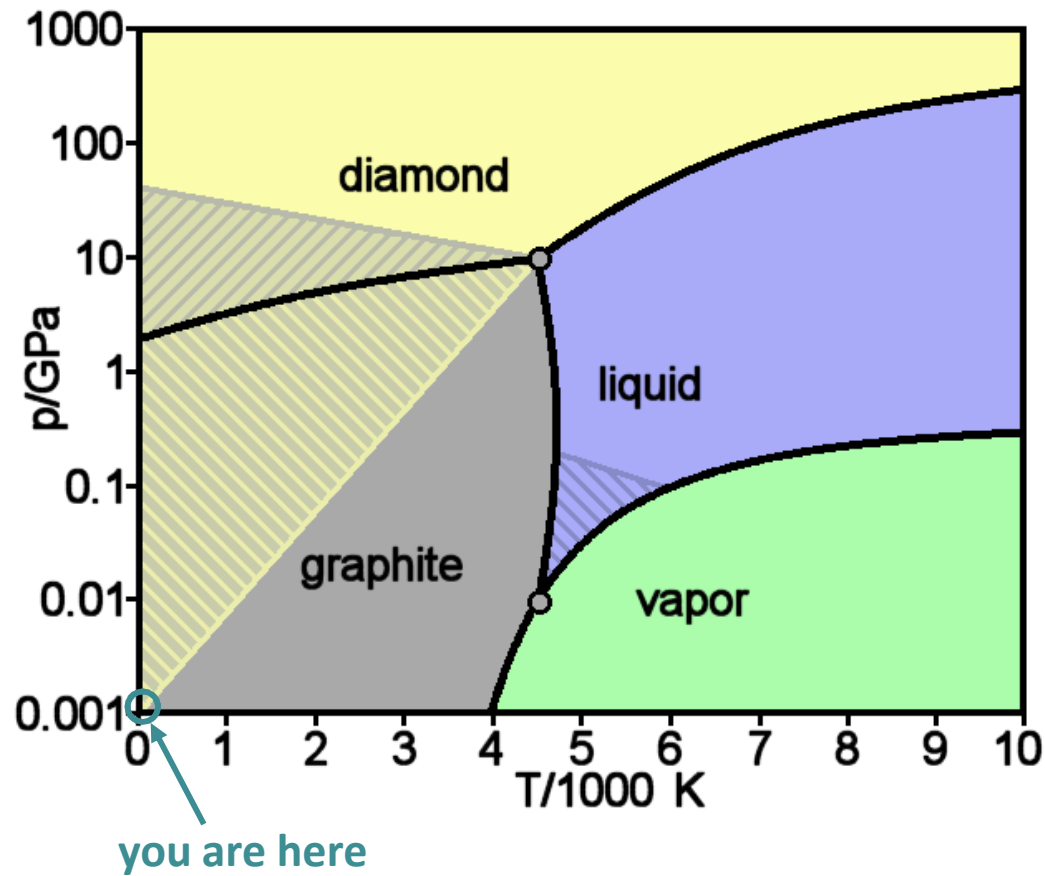


- Lattice: face-centered cubic
- Base: $\{ (0, 0, 0); (\frac{1}{4}a, \frac{1}{4}a, \frac{1}{4}a) \}$
- Crystal: diamond

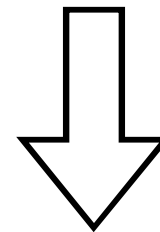
- Bond length: 1.54 Å
- Cell parameter: 3.57 Å
- Atomic density: $1.77 \cdot 10^{23}$ atoms cm^{-3}

Diamond

Phase diagram of Carbon



Room pressure and temperature:
diamond is **meta-stable**



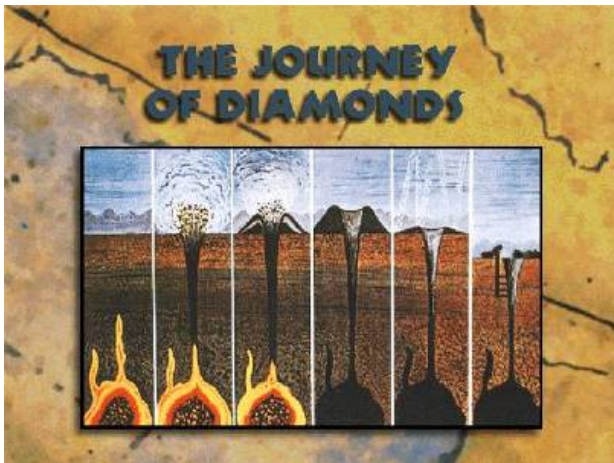
Natural diamond forms at **high pressure and temperature**

Diamond – Synthesis

Natural diamond

At the lithosphere (140-190 km below the Earth surface):

- pressure: 4.5 – 6 Gpa
- temperature: 900 – 1300 °C



Transport to Earth surface: volcanic eruption from deep regions.

Diamond: included in rocks.

- Primary sources: volcanoes
- Secondary sources: sites where diamonds are eroded from the rocks (kimberlite, lampronite)



Kimberley Mine, the largest human hole on Earth

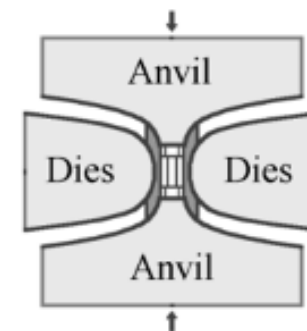
Diamond – Synthesis

High pressure high temperature synthesis



HPHT growth system
© Kobelco

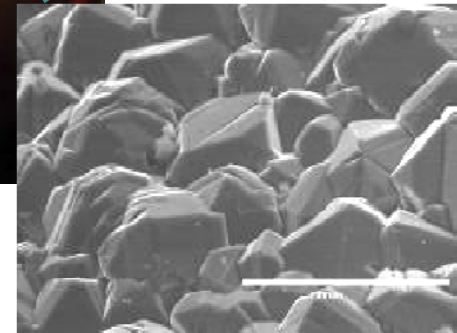
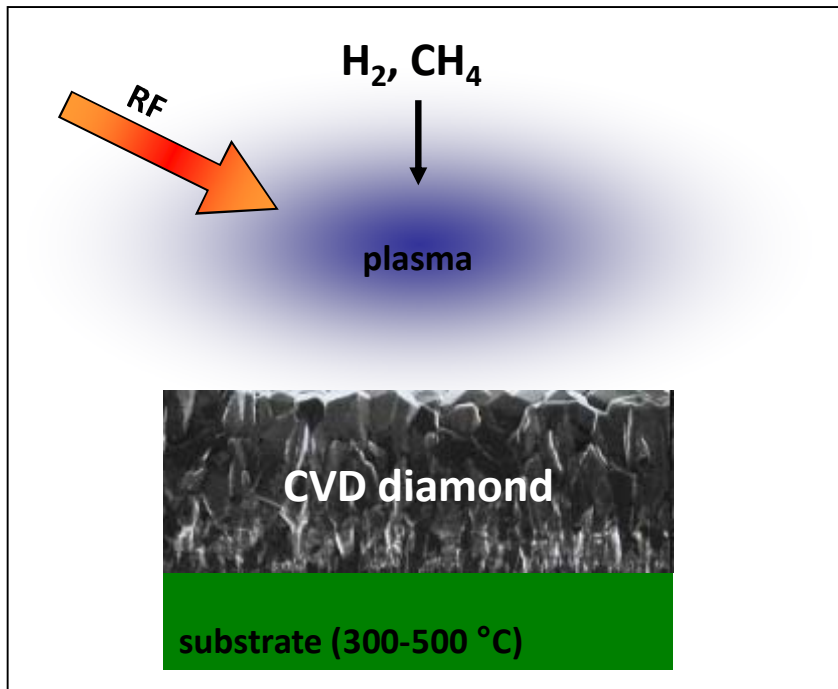
1954: General Electric obtains systematic and commercially viable synthesis of diamond by HPHT.



- growth from a diamond seed
- graphite with catalytic elements (Ni, Fe, ...)
- single-crystals: good structural properties, impurities

Diamond – Synthesis

Chemical Vapour Deposition



- deposition of C on a “cold” substrate from a plasma
- selective etching of non-sp³ C by H in the plasma
- heteroepitaxial growth of polycrystalline samples
- homoepitaxial growth of single-crystals with high purity

Diamond – Synthesis

Classifications

Structure: single-crystal
 polycrystalline
 nanocrystalline

Impurities: type I: N in aggregates

la: [N] = 100-1000 ppm

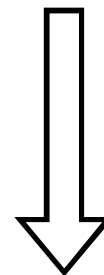
lb: [N] = 10-100 ppm

 type II: substitutional B

Ila: [N] < 1 ppm

IIb: [N] < 1 ppm, B doping

Applications: thermal/mechanical grade
 optical grade
 electronic grade
 detector grade



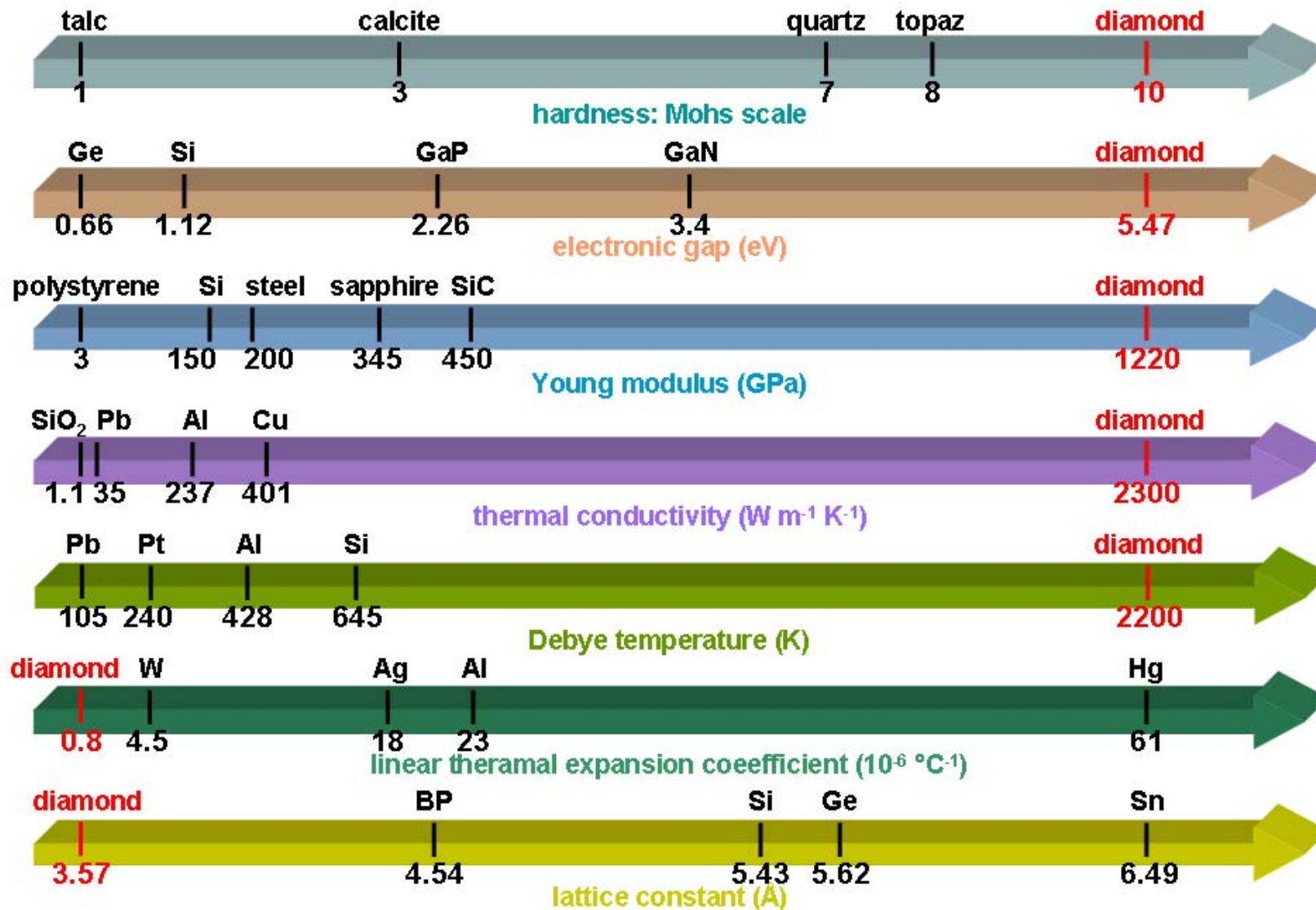
crystal quality

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 - Electrical features
 - Optical features
 - Microfluidics

Diamond – Properties

Extreme physical properties



Diamond – Properties

Other properties

- high carriers mobility
- high breakdown field
- radiation hardness
- wide band-gap → broad transparency, low leakage currents
- chemical inertness
- bio-compatibility
- tissue-equivalence
- surface functionalization → negative electron affinity, 2D hole gas
- efficient luminescent centers
- ...

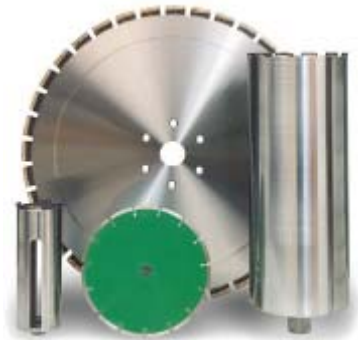
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Diamond – Applications

Mechanical properties

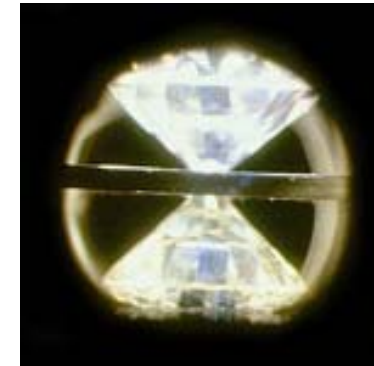
Cutting tools



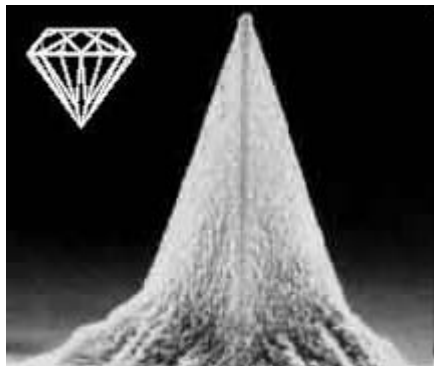
Tweeters



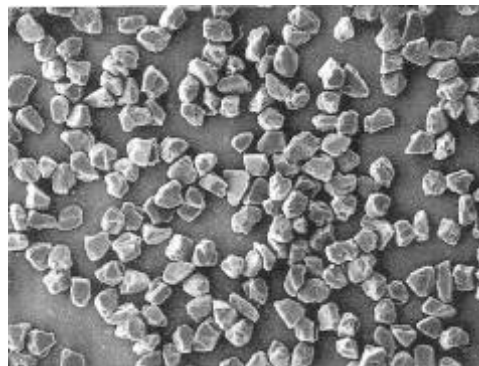
Anvil cells



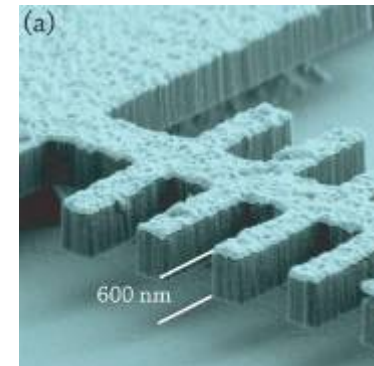
Scanning probe tips



Abrasive powders



MEMS



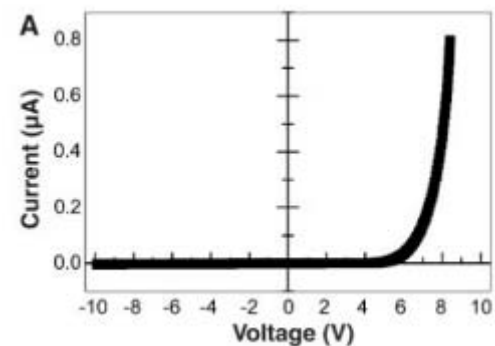
Diamond – Applications

Electronic properties

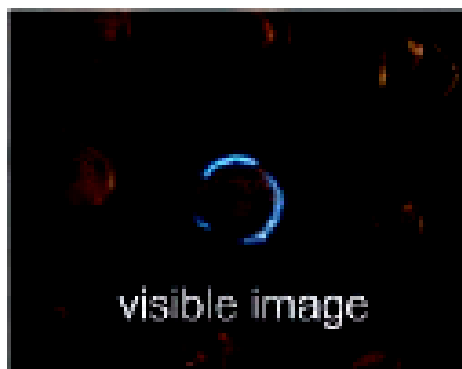
Particle detectors



Power diodes



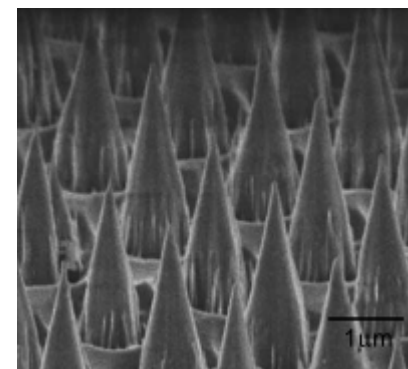
UV diodes



Dosimeters



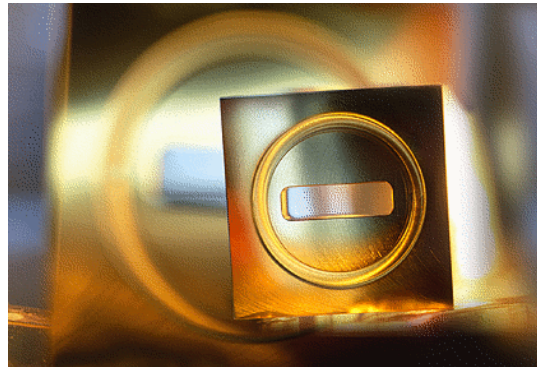
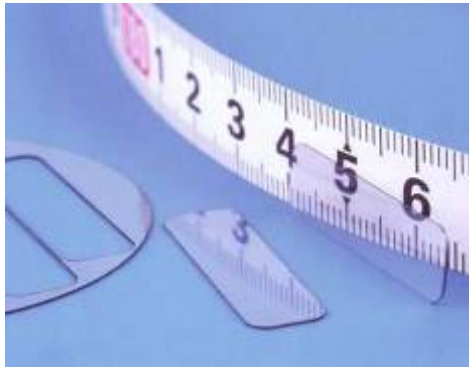
Field emitters



Diamond – Applications

Optical properties

High-power laser windows



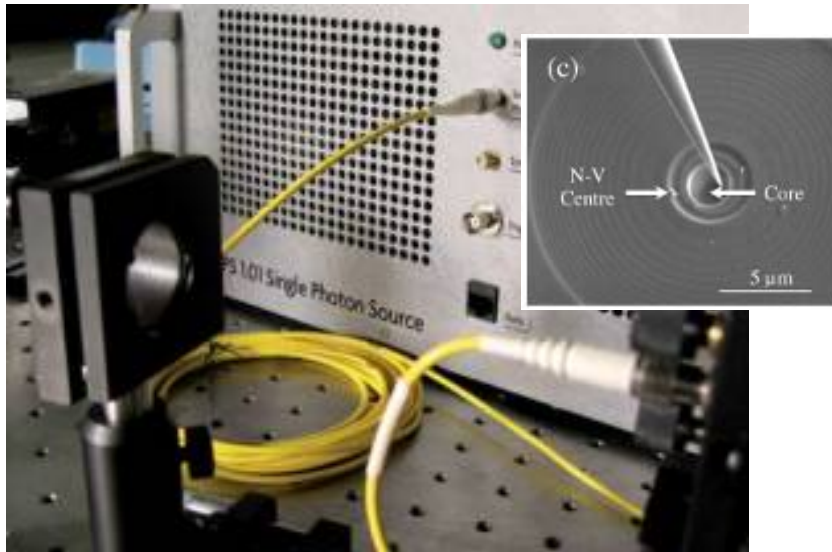
Heat sinks



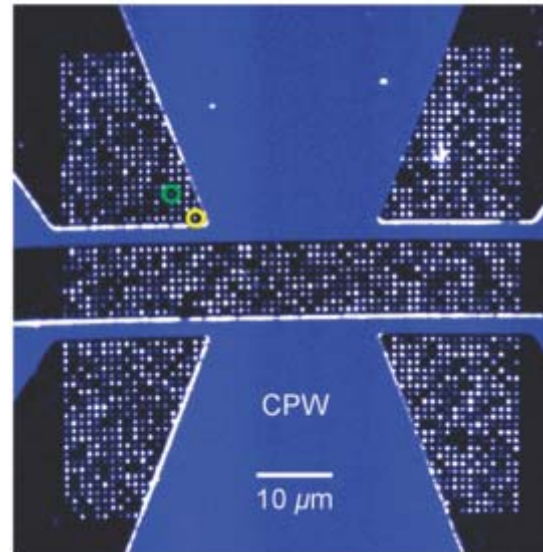
Diamond – Applications

Optical properties

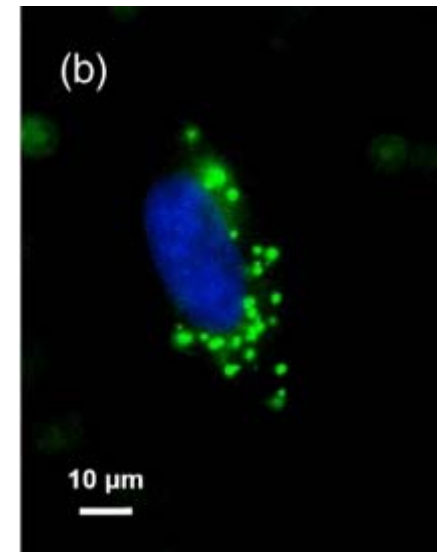
Single photon sources



Quantum computing



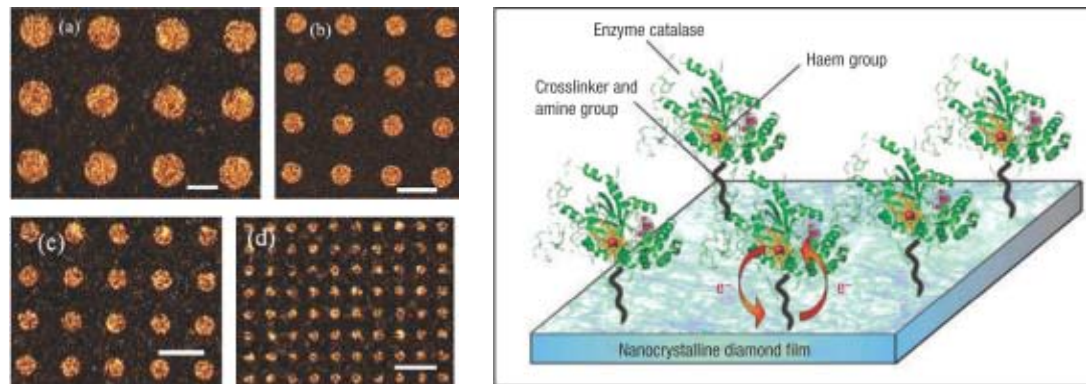
Luminescent markers



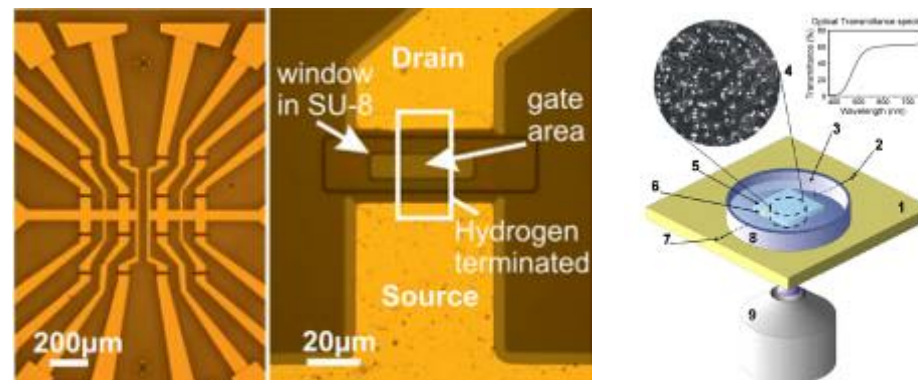
Diamond – Applications

Bio-chemical properties

Molecular bio-sensors



Cellular bio-sensors



Diamond – Applications

Companies in diamond synthesis



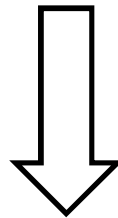
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MeV ion implantation in diamond

Diamond: a *hard* material for micro-fabrication:

- Mechanical hardness
- Chemical inertness
- Optical transparency



Ion beam lithography

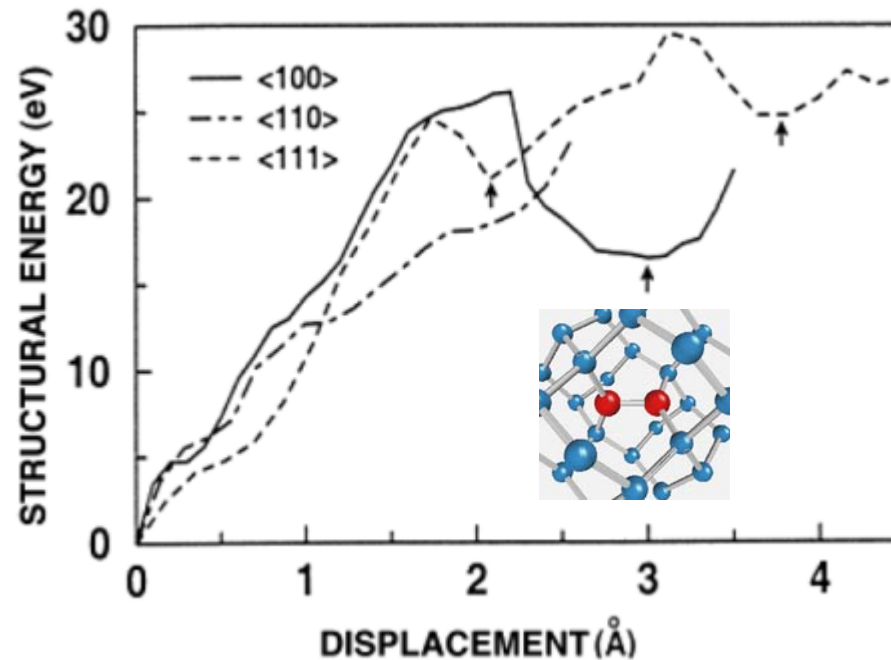
MeV ion implantation in diamond

Electronic energy loss →

No effects (thermal spikes, coulomb explosions) reported so far

Nuclear energy loss →

Significant structural effects on a **meta-stable** material



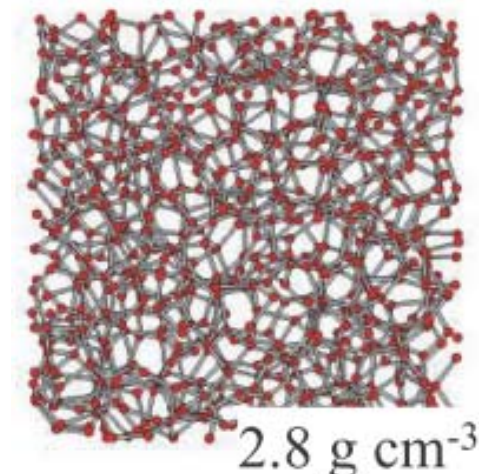
Atom displacement → Formation of an **sp²**-bonded split interstitial

MeV ion implantation in diamond

A crude linear approximation: $\rho_{\text{vac}} = (\text{linear damage density})_{\text{SRIM}} \times (\text{fluence})$

$$[\#_{\text{vac}} \text{ cm}^{-3}] \quad [\#_{\text{vac}} \#_{\text{ion}}^{-1} \text{ cm}^{-1}] \quad [\#_{\text{ion}} \text{ cm}^{-2}]$$

- Non-linear effects (defect-defect interaction, self-annealing, ...) are ignored.
- At high implantation fluences the defect density is not realistic (over-estimated density of point-defects)
- More advanced approaches: Atomistic simulations



MeV ion implantation in diamond

A crude linear approximation: $\rho_{\text{vac}} = (\text{linear damage density})_{\text{SRIM}} \times (\text{fluence})$

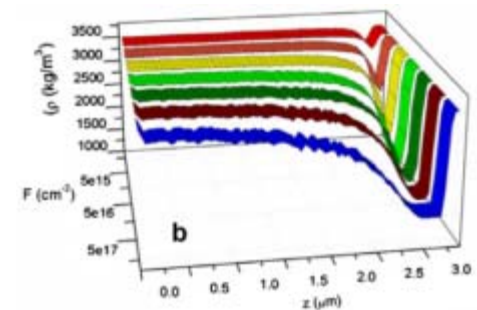
$$[\#_{\text{vac}} \text{ cm}^{-3}] \quad [\#_{\text{vac}} \#_{\text{ion}}^{-1} \text{ cm}^{-1}] \quad [\#_{\text{ion}} \text{ cm}^{-2}]$$

- Non-linear effects (defect-defect interaction, self-annealing, ...) are ignored.
- At high implantation fluences the defect density is not realistic (over-estimated density of point-defects)
- More advanced approaches: Semi-analytical / empirical models

Crystal-TRIM (04/1D - last revision May 2004)
developed by Matthias Posselt ^{1) 2)}



Radiation defects and their annealing behaviour in ion-implanted diamonds
Johan F. Prins ^{*,}, Trevor E. Derry [†]

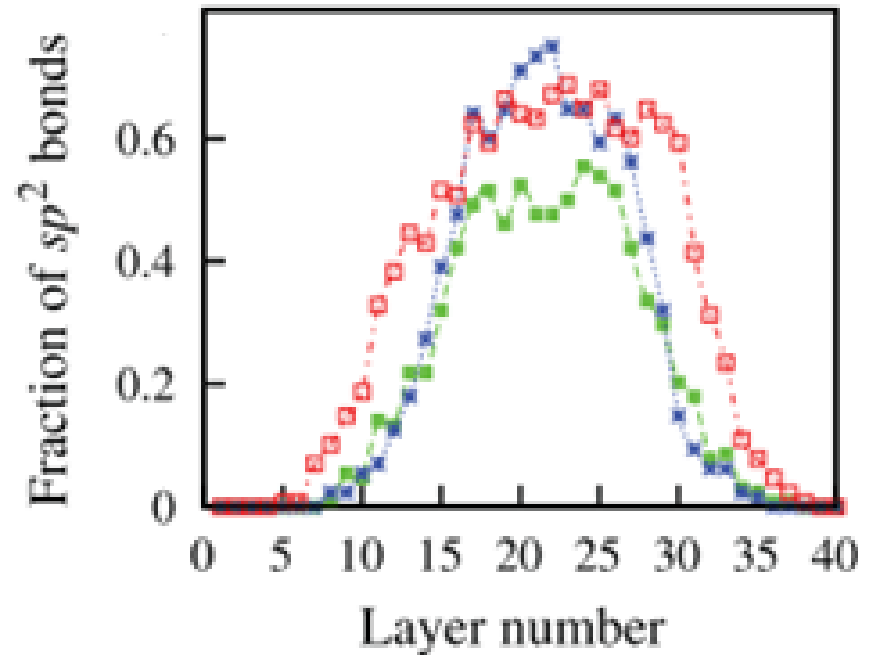
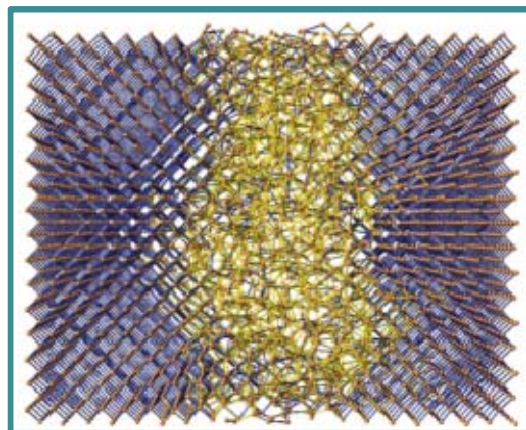
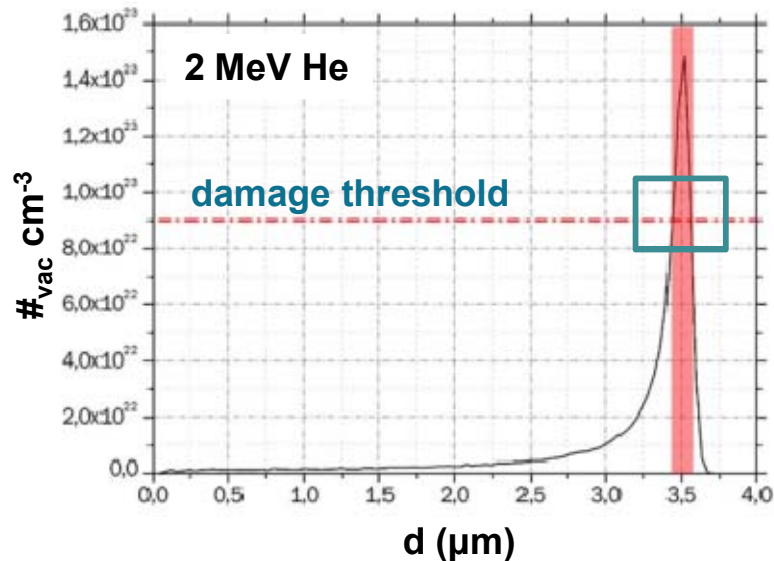


@ : Institute of Ion Beam Physics and Materials Research (Dresden),

Department of Physics – University of Pretoria, Solid State Physics Group – University of Torino

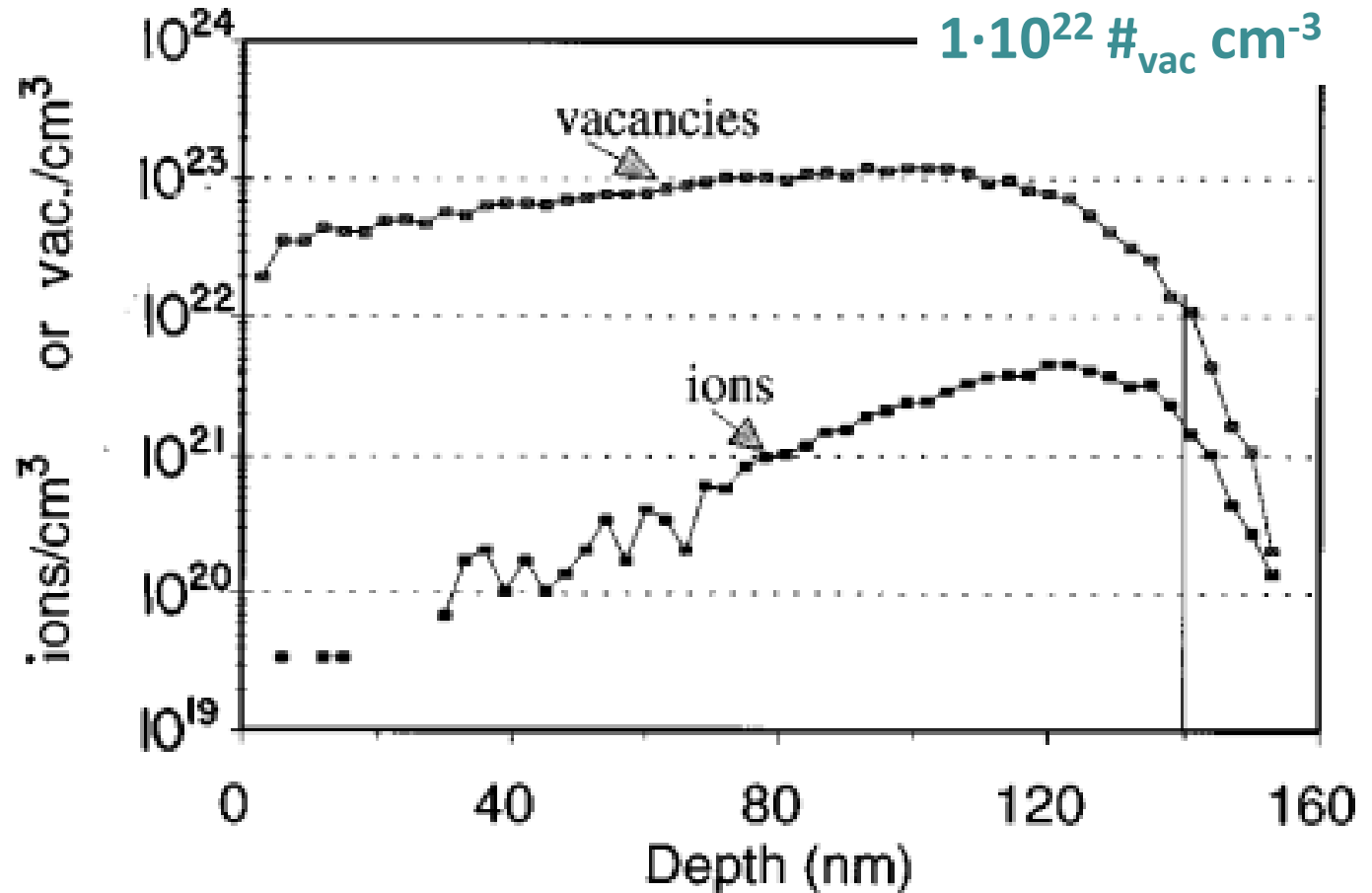
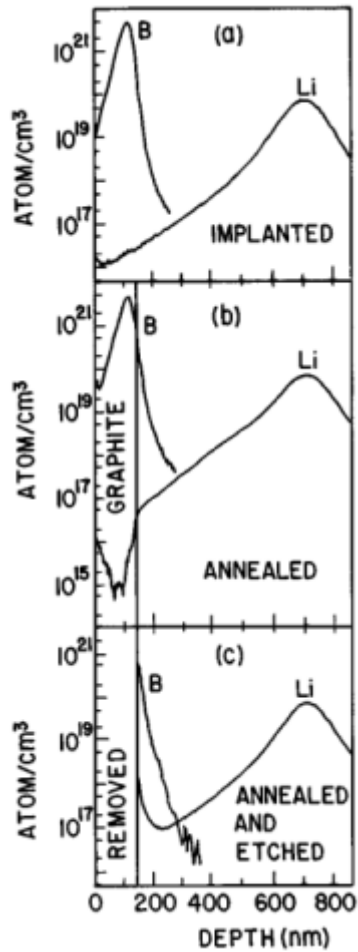
MeV ion implantation in diamond

High fluence implantation → Formation of an amorphous carbon layer where the damage density exceeds a **threshold value**



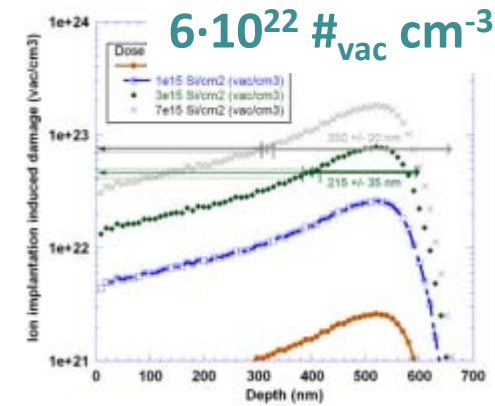
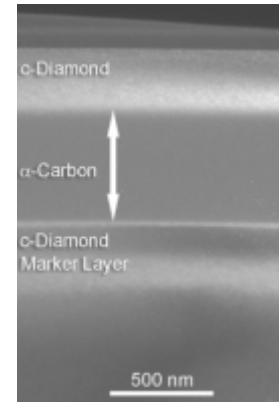
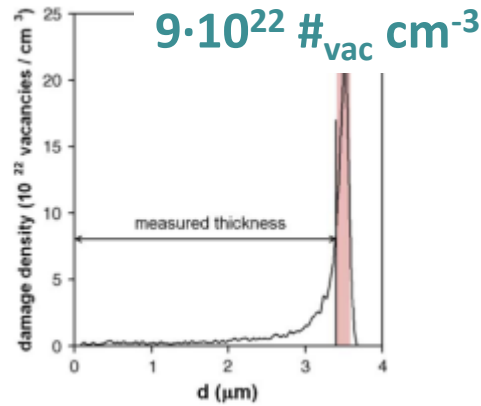
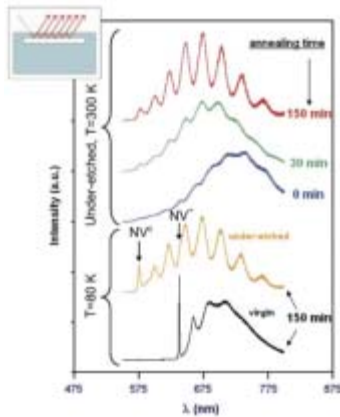
MeV ion implantation in diamond

Graphitization threshold



MeV ion implantation in diamond

Graphitization threshold

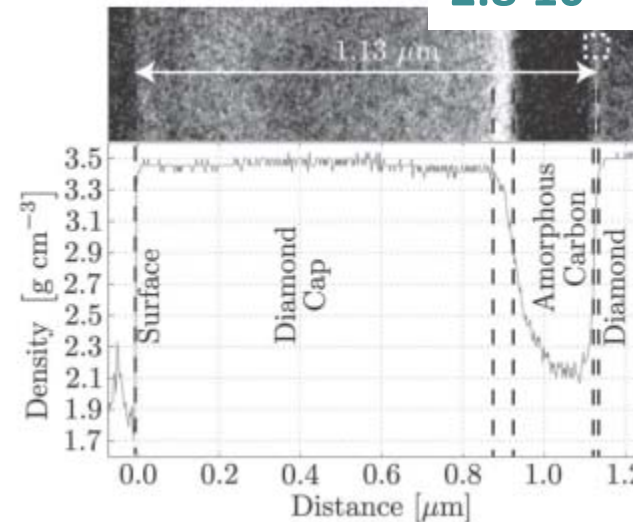
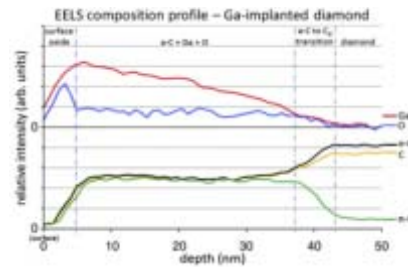
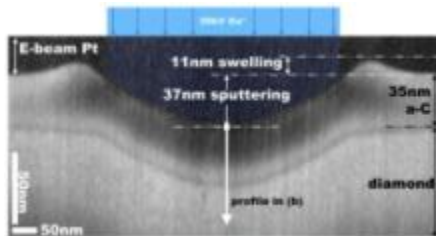


@ : School of Physics – University of Melbourne

@ : Uni. of Florida & Australian National Uni.

$2 \cdot 10^{22} \#_{\text{vac}} \text{cm}^{-3}$

$2.8 \cdot 10^{22} \#_{\text{vac}} \text{cm}^{-3}$

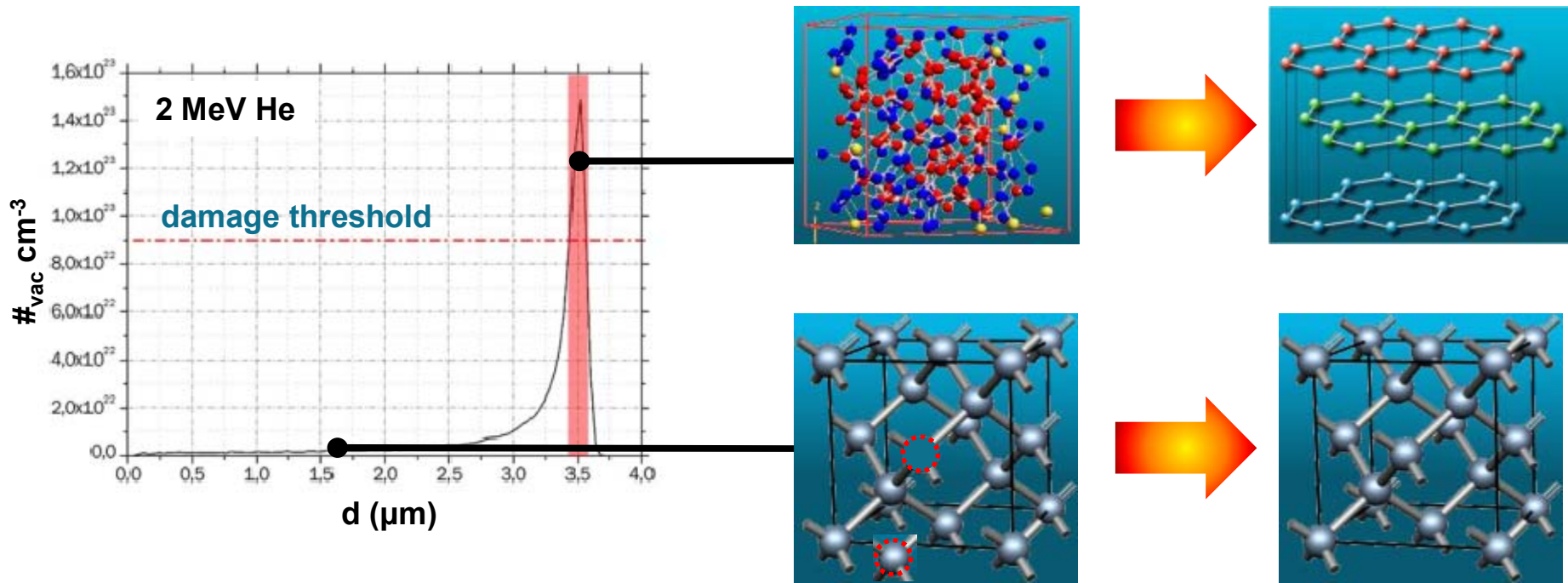


@ : University of New South Wales

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MeV ion implantation in diamond

Thermal annealing

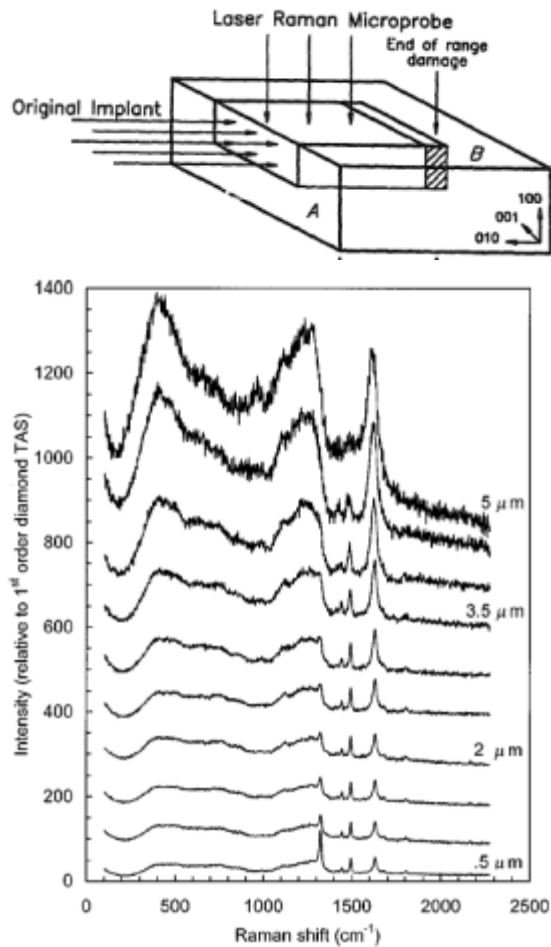


- **Above** threshold: amorphous carbon → polycrystalline graphite
- **Below** threshold: diamond with Frenkel defects → diamond

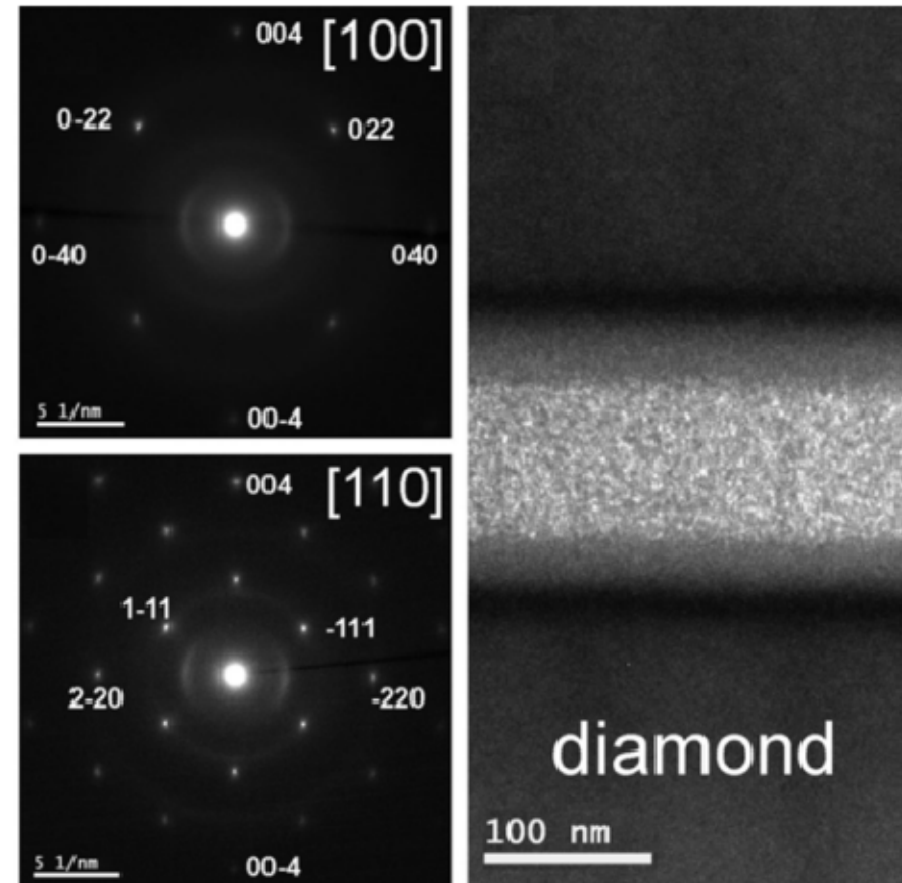
MeV ion implantation in diamond

Experimental evidences

Cross-sectional μ -Raman



Cross-sectional TEM



IBL in Diamond: State of the Art

The diamond lift-off Technique

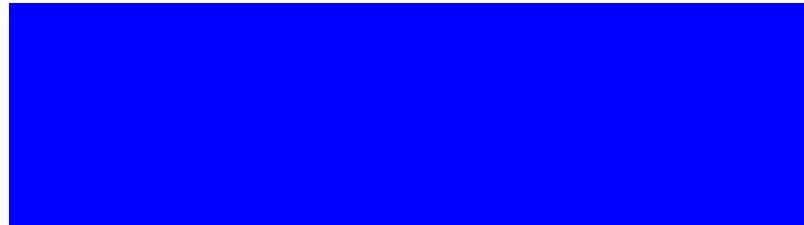
Single-crystal diamond plate liftoff achieved by ion implantation and subsequent annealing

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Appl. Phys. Lett. **61** (26), 28 December 1992 3124



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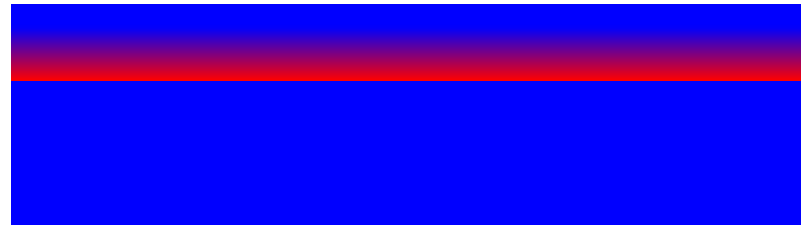
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- MeV ion implantation

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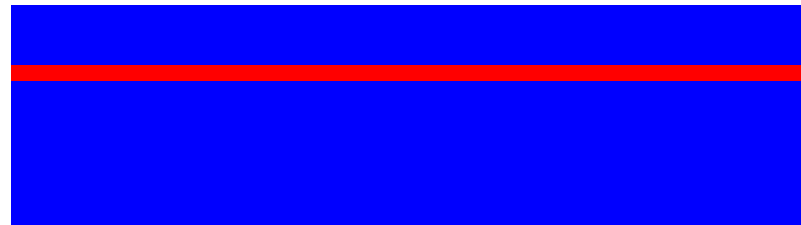
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- MeV ion implantation
- Thermal annealing

IBL in Diamond: State of the Art

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- MeV ion implantation
- Thermal annealing
- Selective graphite etching

IBL in Diamond: State of the Art

The diamond lift-off Technique

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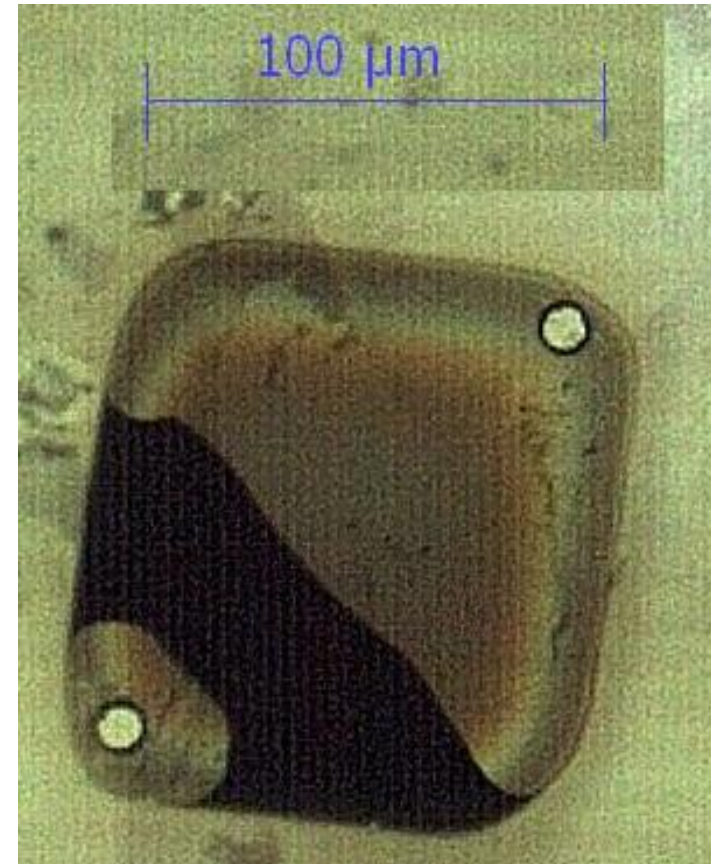
- MeV ion implantation
- Thermal annealing
- Selective graphite etching
- Lift-out

IBL in Diamond: State of the Art

Selective graphite etching



- Wet chemical etching
i.e.: 1:1:1 H_2SO_4 : HNO_3 : HClO_4 boiling acid
- Annealing in oxygen atmosphere
 $T = 550 - 580$ °C in air
- Annealing in ozone atmosphere
 $T = 500 - 550$ °C in air under UV illumination
- Electrochemical etching
 H_3BO_3 , non-contact Pt electrodes, $V \cong 200$ V



IBL in Diamond: State of the Art

Lift-off + laser micro-cutting

Fabrication of single-crystal diamond microcomponents

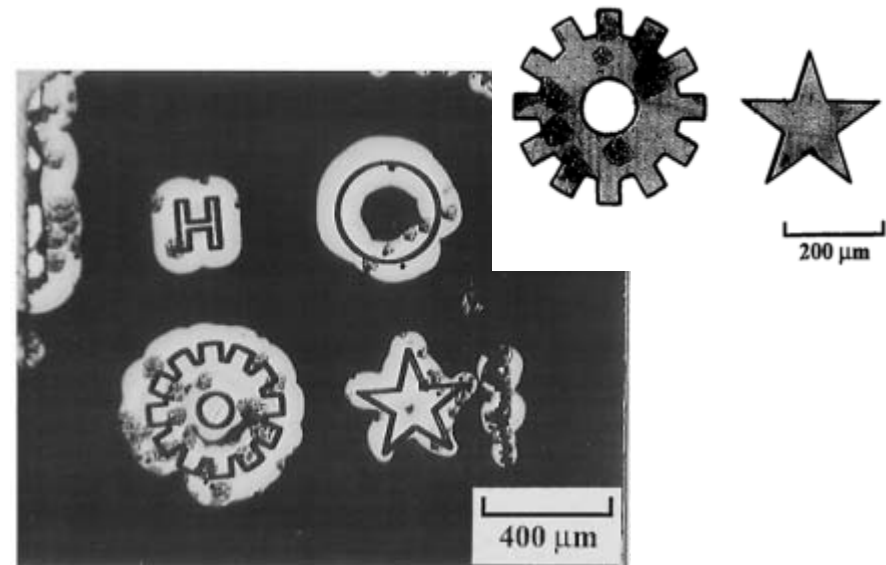
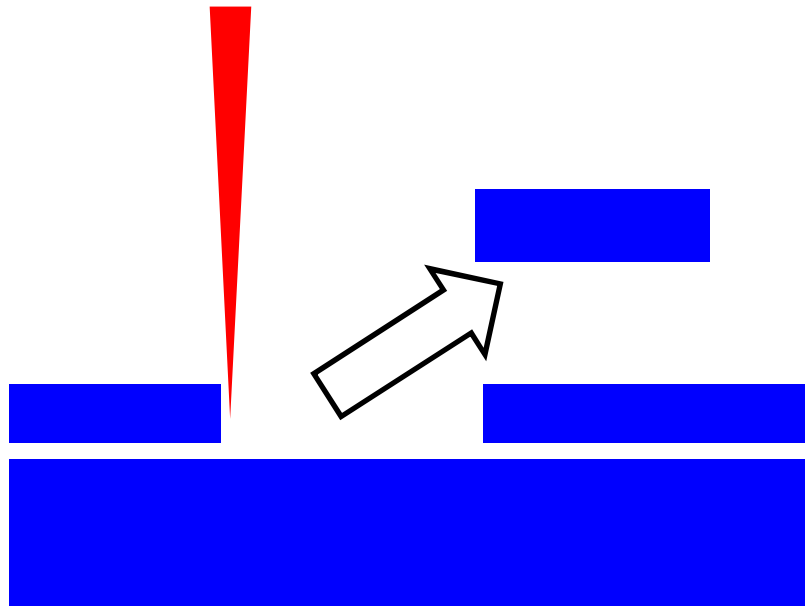
John D. Hunn, S. P. Withrow, C. W. White, R. E. Clausing, and L. Heatherly
Oak Ridge National Laboratory, Bldg 5500 MS-6376, Oak Ridge, Tennessee 37831-6376

C. Paul Christensen
Potomac Photonics, Lanham, Maryland 20705

(Received 26 August 1994; accepted for publication 7 October 1994)

We have combined a technique for the lift-off of thin diamond films from a bulk diamond with a technique for engraving diamond with a focused excimer laser to produce free-standing single-crystal diamond microstructures. One microcomponent that has been produced is a 12 tooth gear $\sim 400 \mu\text{m}$ in diameter and $\sim 13 \mu\text{m}$ thick. Other microstructures have also been demonstrated, showing the versatility of this method. This process should be applicable to producing diamond microcomponents down to spatial dimensions (width and thickness) of a few micrometers. © 1994 American Institute of Physics.

3072 Appl. Phys. Lett. 65 (24), 12 December 1994



IBL in Diamond: State of the Art

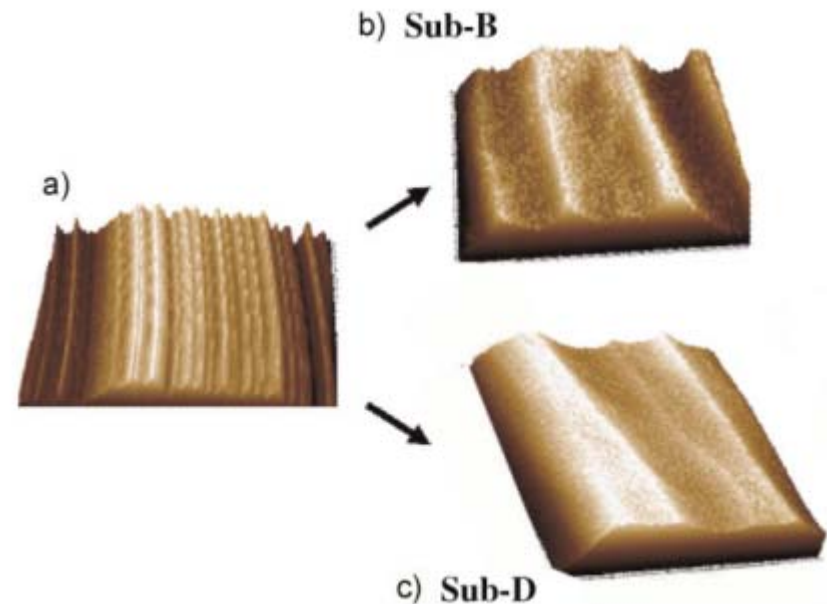
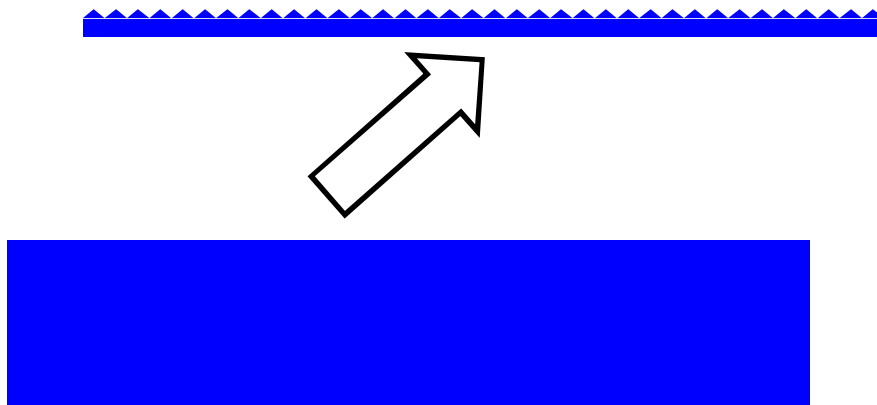
Surface smoothening

Phys. Status Solidi A 206, No. 9, 1955–1959 (2009) / DOI 10.1002/pssa.200982232



High surface smoothening of
diamond HPHT (100) substrates

C. Mer-Calfati^{1,1}, N. Habka^{1,2}, A. Ben-Younes¹, M.-A. Pinault², J. Barjon², and P. Bergonzo¹



IBL in Diamond: State of the Art

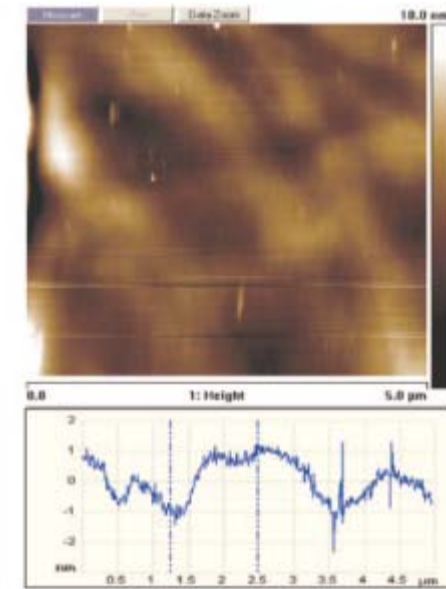
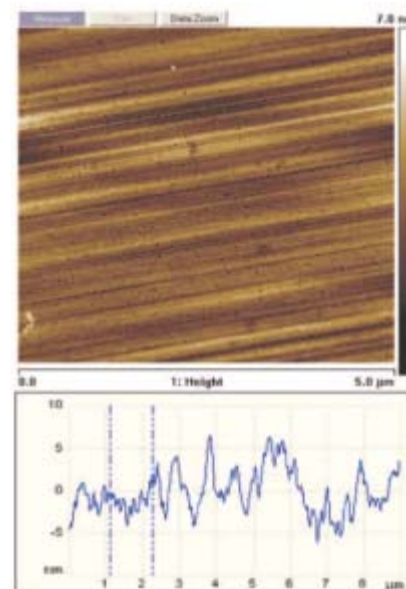
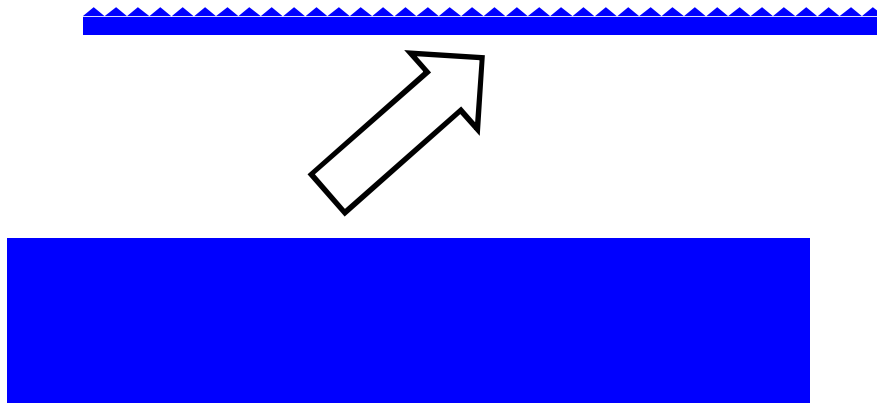
Surface smoothing

Phys. Status Solidi A 208, No. 9, 2057–2061 (2011) / DOI 10.1002/pssa.201100038



Ultra-smooth single crystal diamond surfaces resulting from implantation and lift-off processes

T. N. Tran Thi^{**1}, B. Fernandez¹, D. Eon¹, E. Gheeraert¹, J. Härtwig², T. Lafford², A. Perrat-Mabilon², C. Peaucelle², P. Olivero⁴, and E. Bustarret^{**1}



IBL in Diamond: State of the Art

Lift-off + CVD growth

Diamond & Related Materials 20 (2011) 616–619



Contents lists available at ScienceDirect

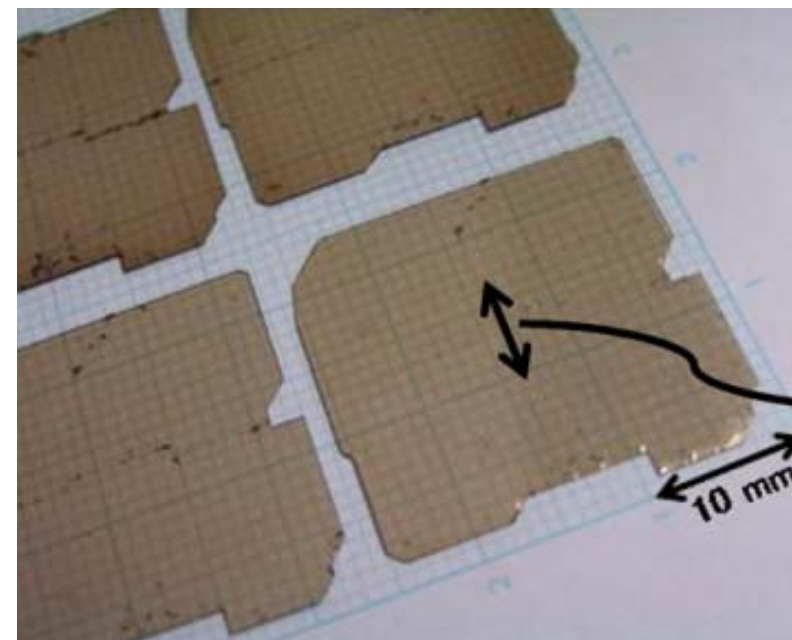
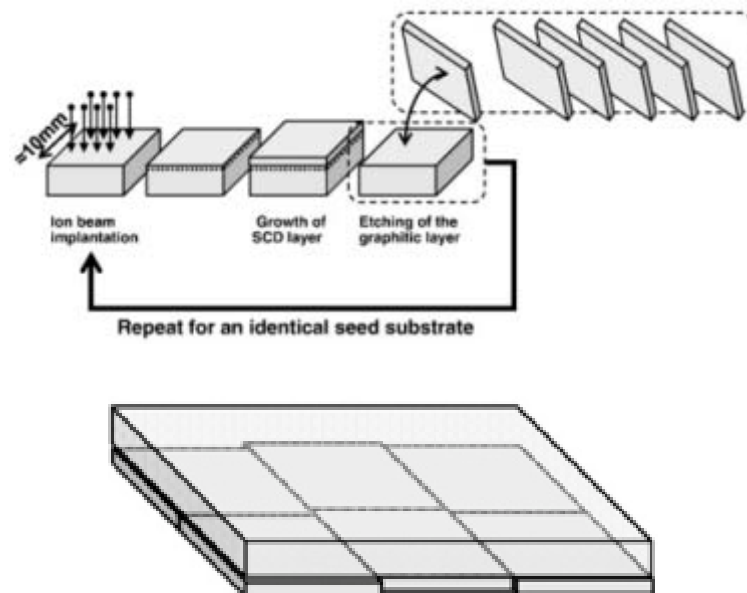
Diamond & Related Materials

journal homepage: www.elsevier.com/locate/diamond



Developments of elemental technologies to produce inch-size single-crystal diamond wafers[☆]

Hideaki Yamada^{*}, Akiyoshi Chayahara, Yoshiaki Mokuno, Nobuteru Tsubouchi, Shin-ichi Shikata, Naoji Fujimori¹



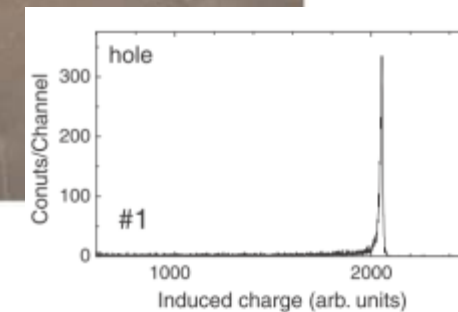
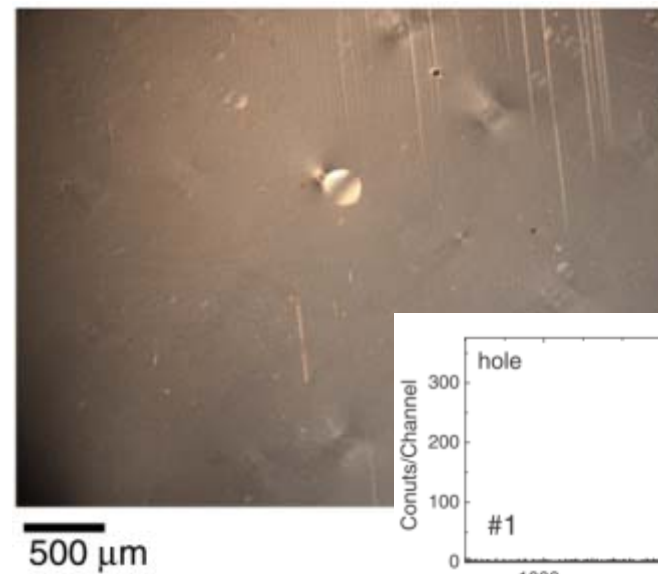
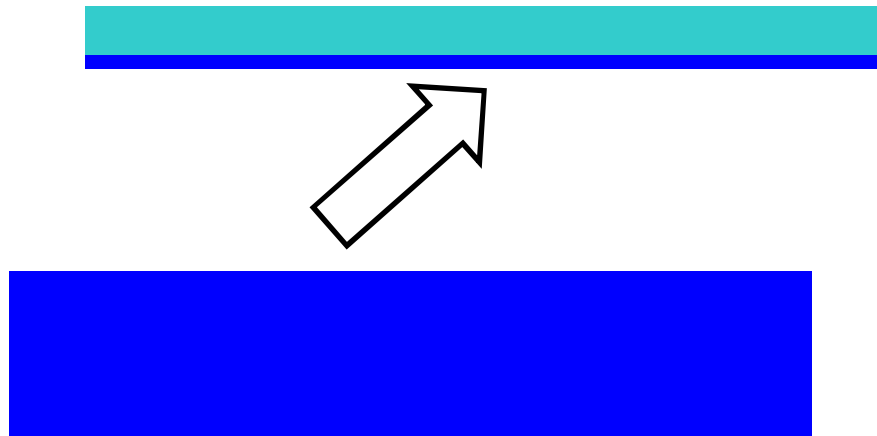
IBL in Diamond: State of the Art

Lift-off + CVD growth

Diamond & Related Materials 24 (2012) 74–77



Characterization of a sandwich-type large CVD single crystal diamond particle detector fabricated using a lift-off method[☆]



IBL in Diamond: State of the Art

Lift-off + CVD growth

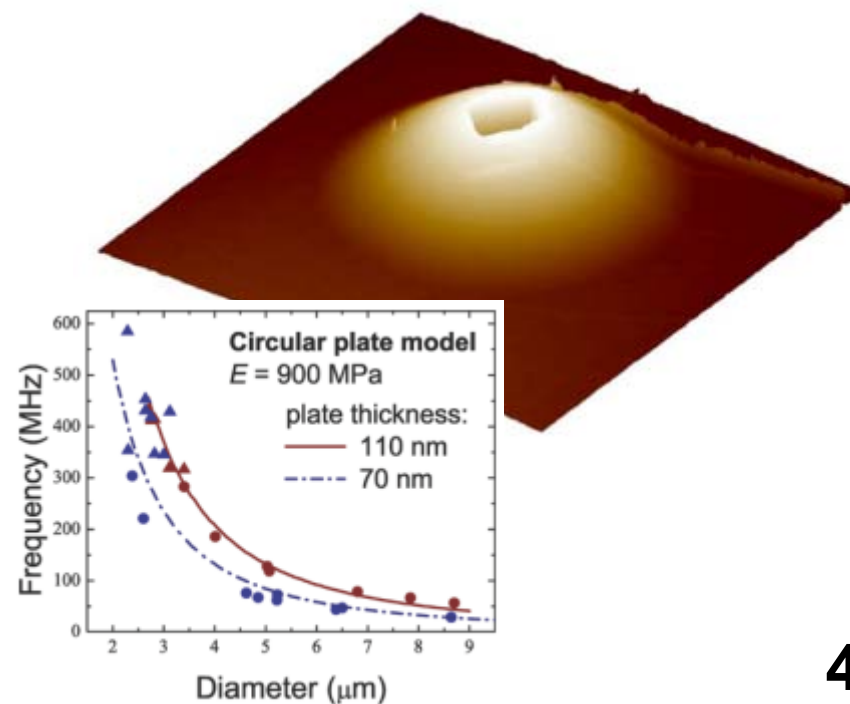
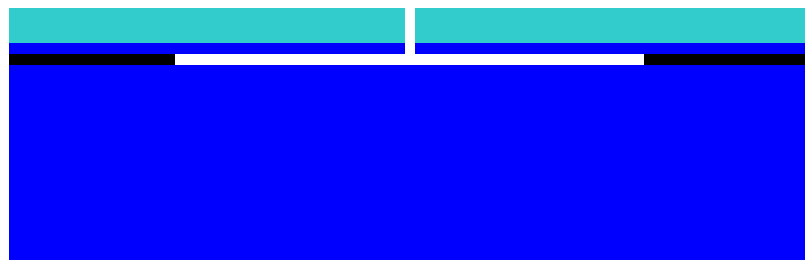
NANO LETTERS

LETTER

pubs.acs.org/NanoLett

Ultrathin Single Crystal Diamond Nanomechanical Dome Resonators

Maxim K. Zalalutdinov,^{*,†} Matthew P. Ray,[†] Douglas M. Photiadis,[†] Jeremy T. Robinson,[†] Jeffrey W. Baldwin,[†] James E. Butler,[§] Tatyana I. Feygelson,[§] Bradford B. Pate,[†] and Brian H. Houston[†]



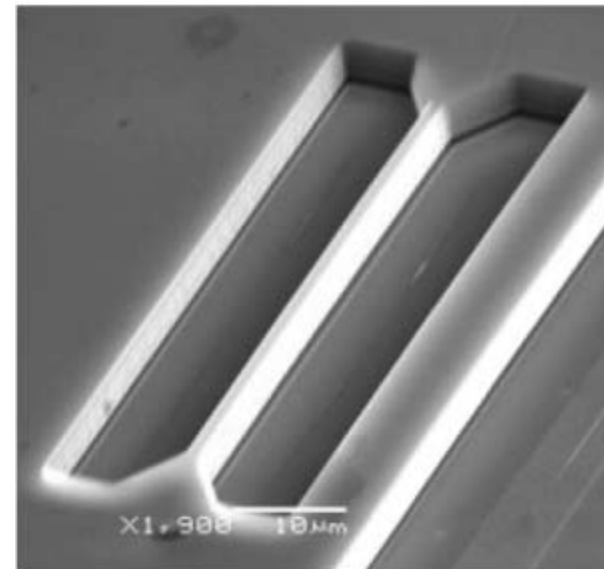
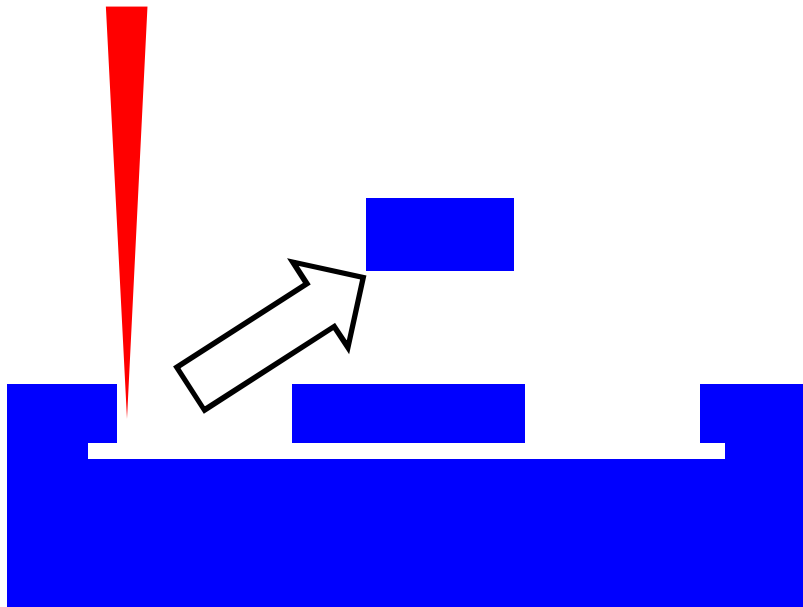
IBL in Diamond: State of the Art

Lift-off + Focused Ion Beam (FIB) milling

Ion-Beam-Assisted Lift-Off Technique for Three-Dimensional Micromachining of Freestanding Single-Crystal Diamond**

By Paolo Olivero, Sergey Rubanov, Patrick Reichart,
Brant C. Gibson, Shane T. Huntington, James Rabeau,
Andrew D. Greentree, Joseph Salzman, David Moore,
David N. Jamieson, and Steven Prawer*

Adv. Mater. **2005**, 17, 2427–2430



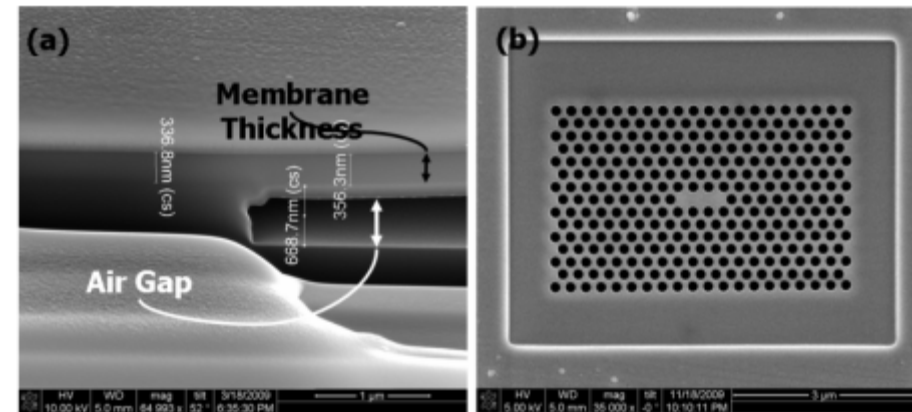
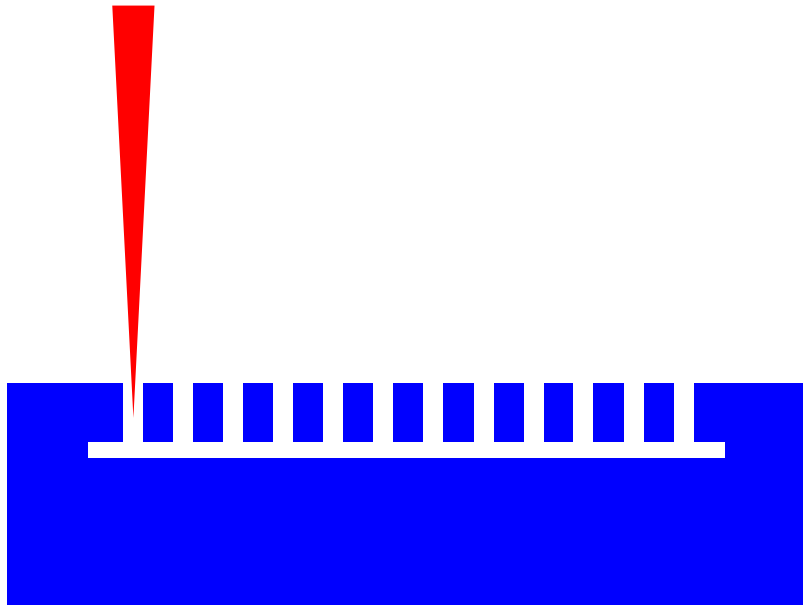
IBL in Diamond: State of the Art

Lift-off + Focused Ion Beam (FIB) milling



Triangular nanobeam photonic cavities in
single-crystal diamond

Igal Bayn^{1,3}, Boris Meyler¹, Joseph Salzman¹ and Rafi Kalish²



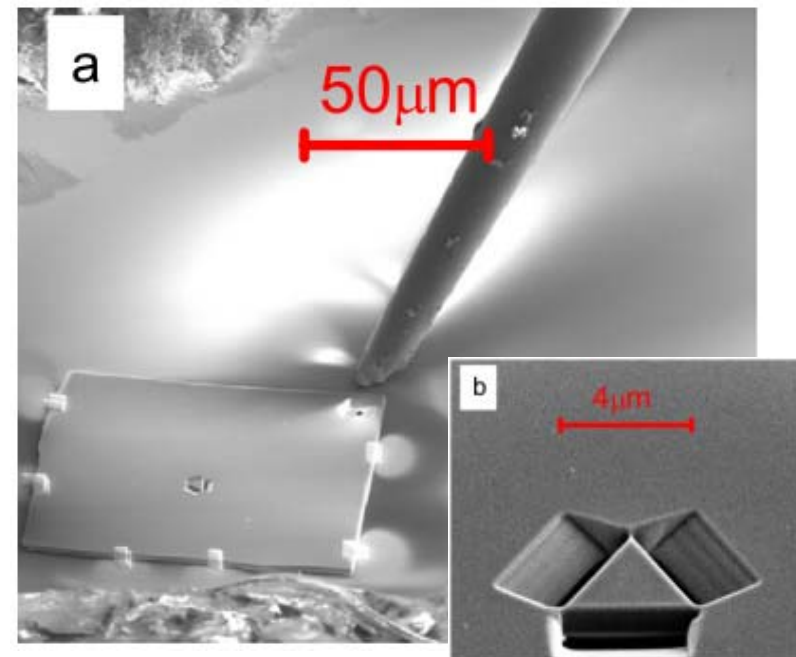
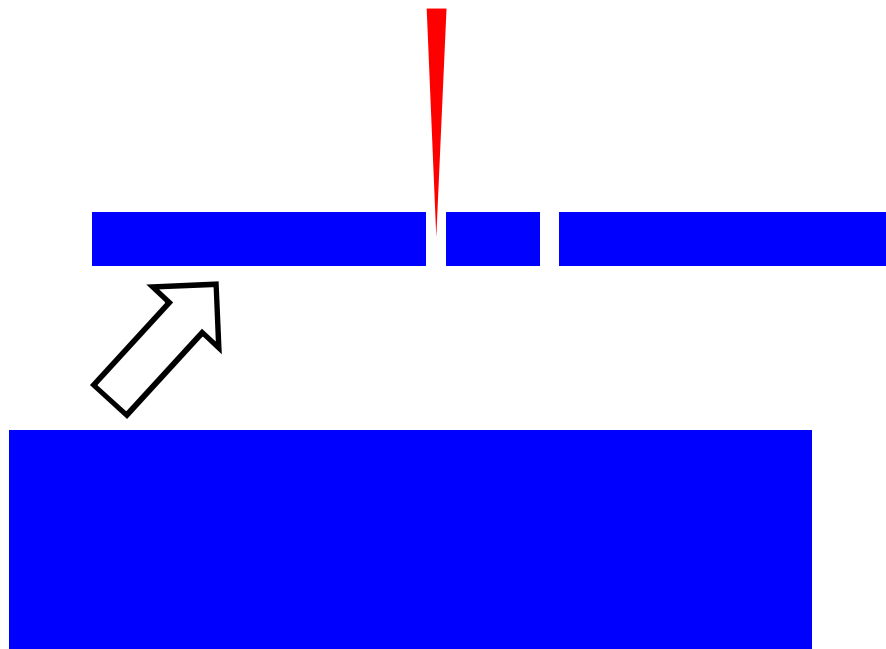
IBL in Diamond: State of the Art

Lift-off + Focused Ion Beam (FIB) milling



Optical properties of single crystal diamond microfilms fabricated by ion implantation and lift-off processing

Brian R. Patton ^a, Philip R. Dolan ^a, Fabio Grazioso ^a, Matthew B. Wincott ^a, Jason M. Smith ^{a,*},
Matthew L. Markham ^b, Daniel J. Twitchen ^b, Yanfeng Zhang ^c, Erdan Gu ^c, Martin D. Dawson ^c,
Barbara A. Fairchild ^d, Andrew D. Greentree ^d, Steven Prawer ^d



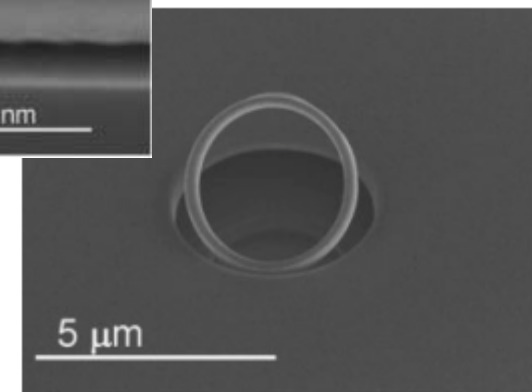
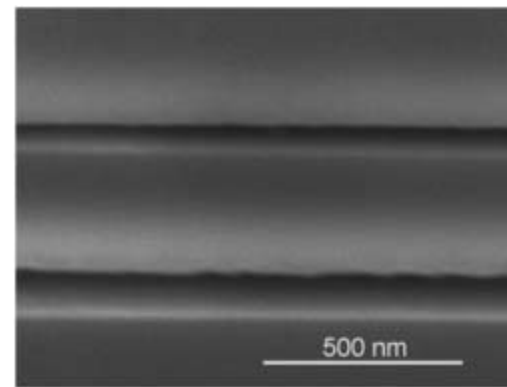
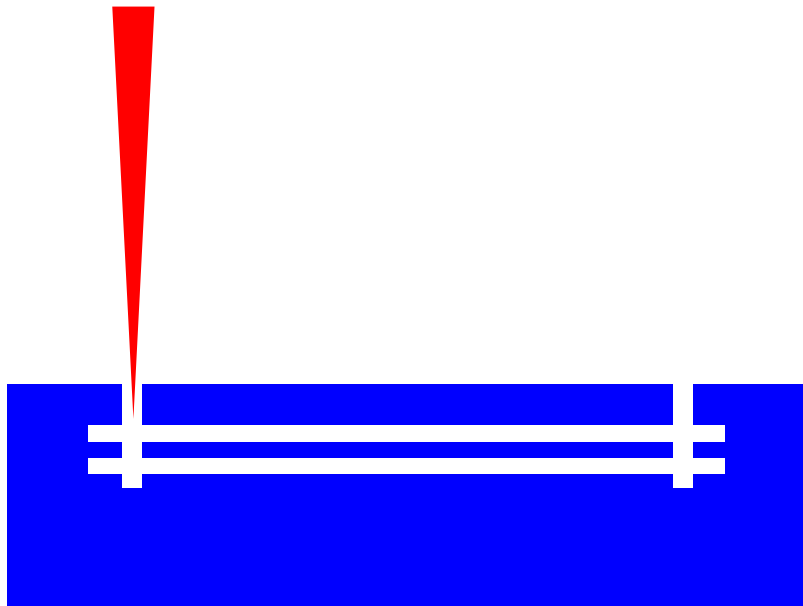
IBL in Diamond: State of the Art

Lift-off (double implantation) + FIB

Fabrication of Ultrathin Single-Crystal Diamond Membranes**

By *Barbara A. Fairchild,* Paolo Olivero, Sergey Rubanov, Andrew D. Greentree, Felix Waldermann, Robert A. Taylor, Ian Walmsley, Jason M. Smith, Shane Huntington, Brant C. Gibson, David N. Jamieson, and Steven Prawer*

Adv. Mater. **2008**, *20*, 4793–4798



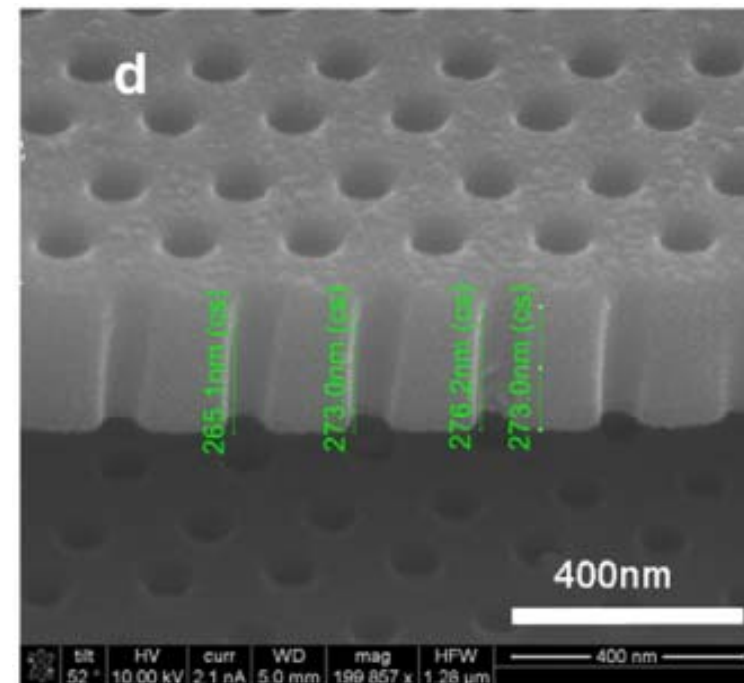
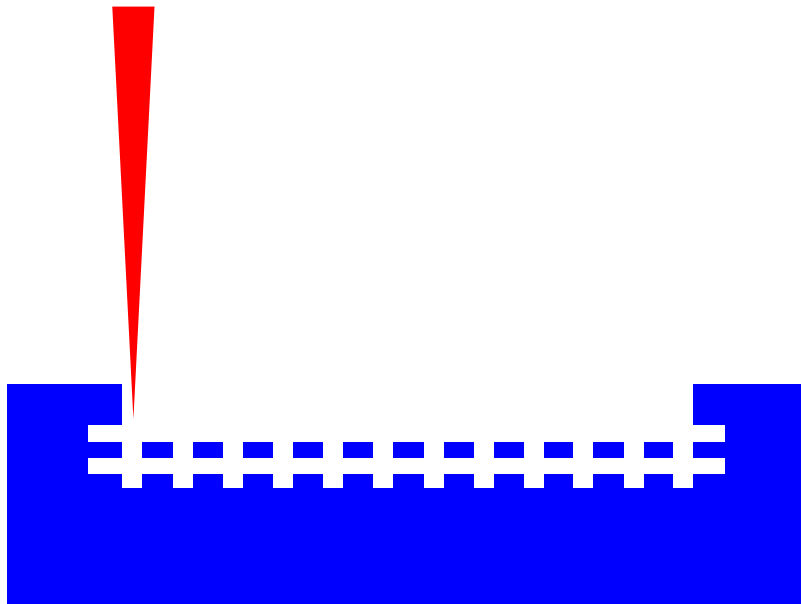
IBL in Diamond: State of the Art

Lift-off (double implantation) + FIB



Processing of photonic crystal nanocavity for quantum information in diamond

Igal Bayn ^{a,*}, Boris Meyler ^a, Alex Lahav ^a, Joseph Salzman ^a, Rafi Kalish ^b, Barbara A. Fairchild ^c, Steven Prawer ^c, Michael Barth ^d, Oliver Benson ^d, Thomas Wolf ^e, Petr Siyushev ^e, Fedor Jelezko ^e, Jorg Wrachtrup ^e



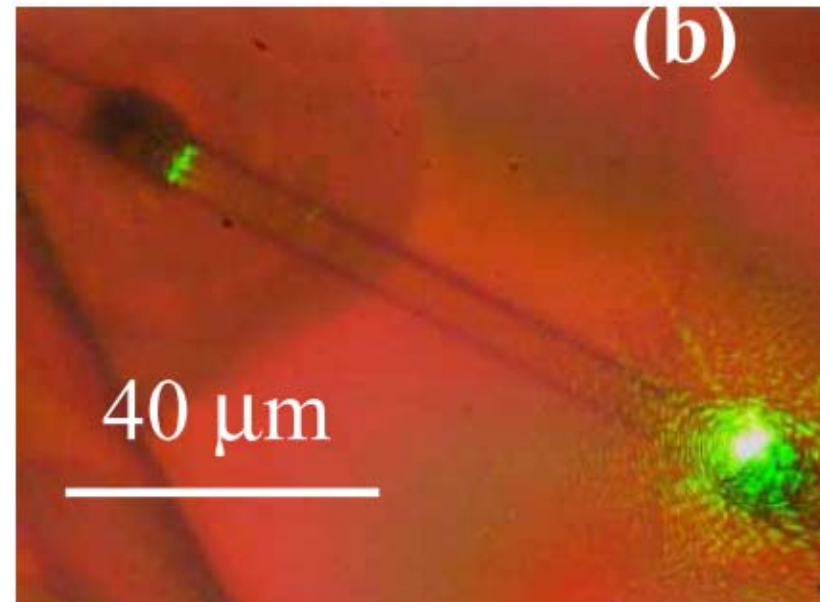
IBL in Diamond: State of the Art

Lift-off + Reactive Ion Etching (RIE)

Diamond waveguides fabricated by reactive ion etching

Mark P. Hiscocks¹, Kumaravelu Ganesan², Brant C. Gibson³, Shane T. Huntington²,
François Ladouceur¹, and Steven Prawer³

24 November 2008 / Vol. 16, No. 24 / OPTICS EXPRESS 19512



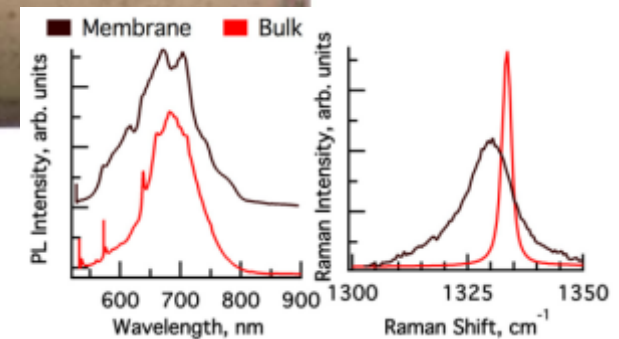
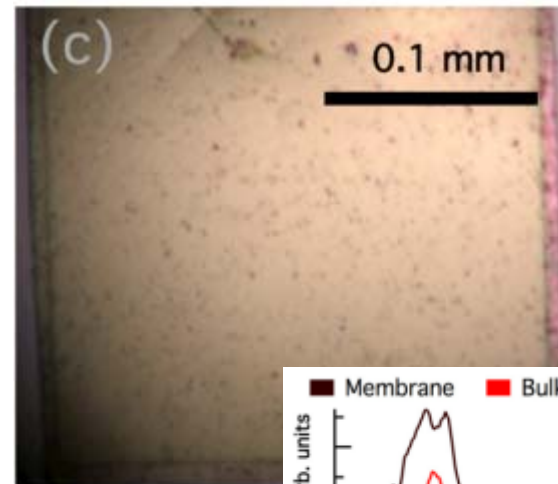
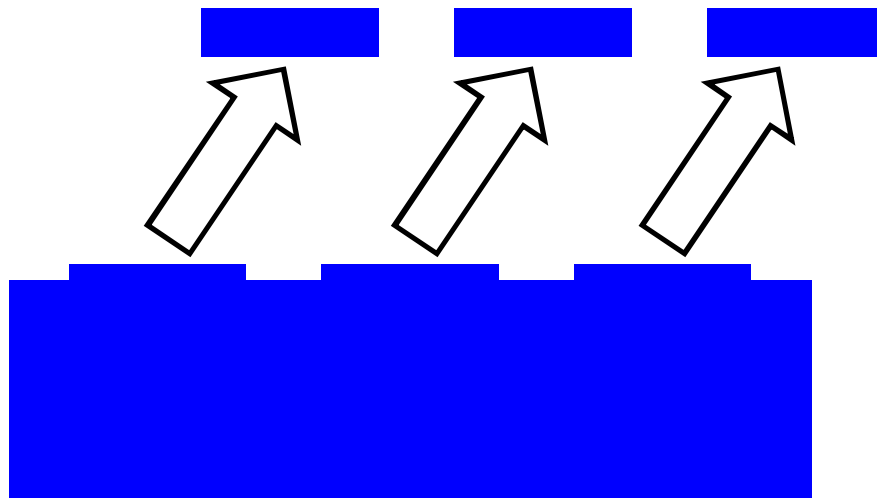
IBL in Diamond: State of the Art

Lift-off + Reactive Ion Etching (RIE)

APPLIED PHYSICS LETTERS 99, 081913 (2011)

Fabrication of thin, luminescent, single-crystal diamond membranes

Andrew P. Magyar,¹ Jonathan C. Lee,¹ Andi M. Limarga,¹ Igor Aharonovich,¹ Fabian Rol,¹ David R. Clarke,¹ Mengbing Huang,² and Evelyn L. Hu^{1,a)}



IBL in Diamond: State of the Art

Lift-off + CVD growth + RIE

Fabrication of suspended single crystal diamond devices by electrochemical etch

C. F. Wang^{a1}

Department of Physics, University of California, Santa Barbara, California 93106

E. L. Hu

Department of Electrical and Computer Engineering, University of California, Santa Barbara, California 93106 and Materials Department, University of California, Santa Barbara, California 93106

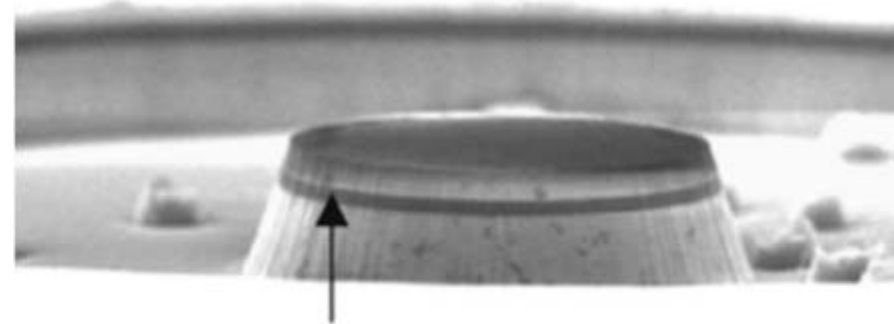
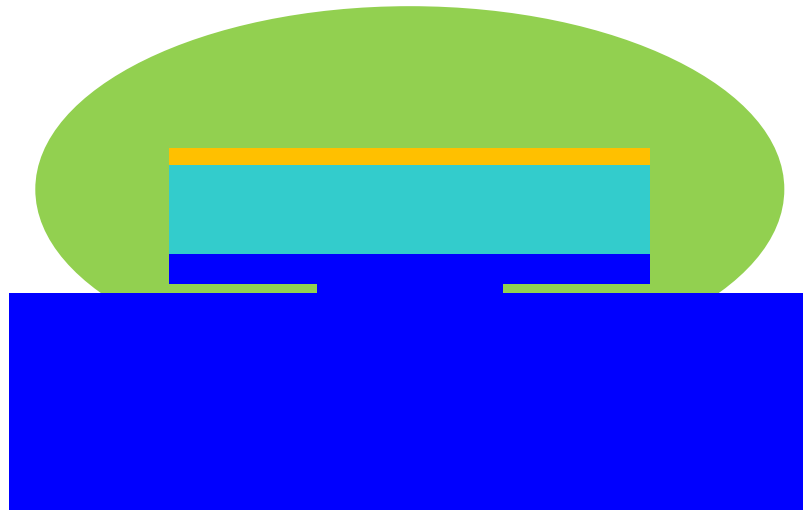
J. Yang

Gas/Surface Dynamics Section, Naval Research Laboratory, Washington, DC 20375 and NOVA research, Inc., Alexandria, Virginia 22308

J. E. Butler

Gas/Surface Dynamics Section, Naval Research Laboratory, Washington, DC 20375

730 J. Vac. Sci. Technol. B 25(3), May/June 2007



Ion Implanted Layer

$2 \mu\text{m}$

IBL in Diamond: State of the Art

Lift-off + CVD growth + RIE

Materials Views

www.MaterialsViews.com

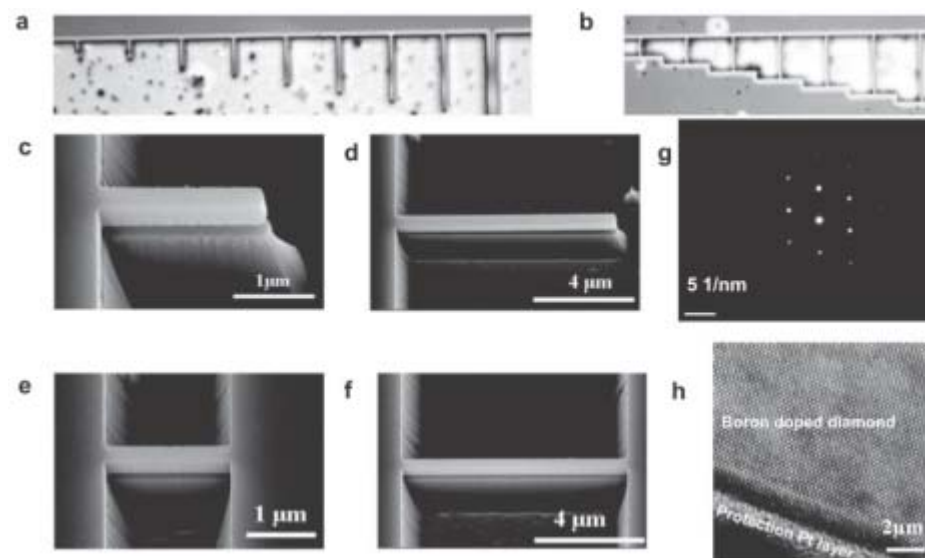
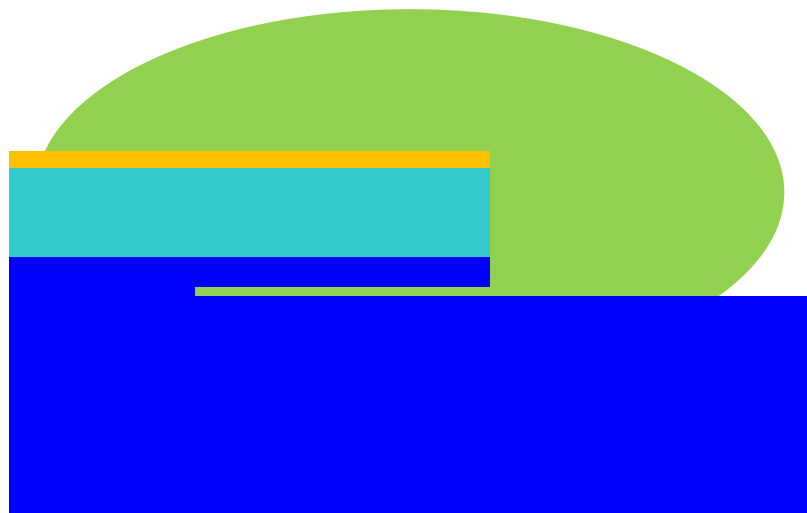
ADVANCED MATERIALS

www.advmat.de

Suspended Single-Crystal Diamond Nanowires for High-Performance Nanoelectromechanical Switches

By Meiyong Liao,* Shunichi Hishita, Eiichiro Watanabe, Satoshi Koizumi, and Yasuo Koide

Adv. Mater. 2010, 22, 5393–5397



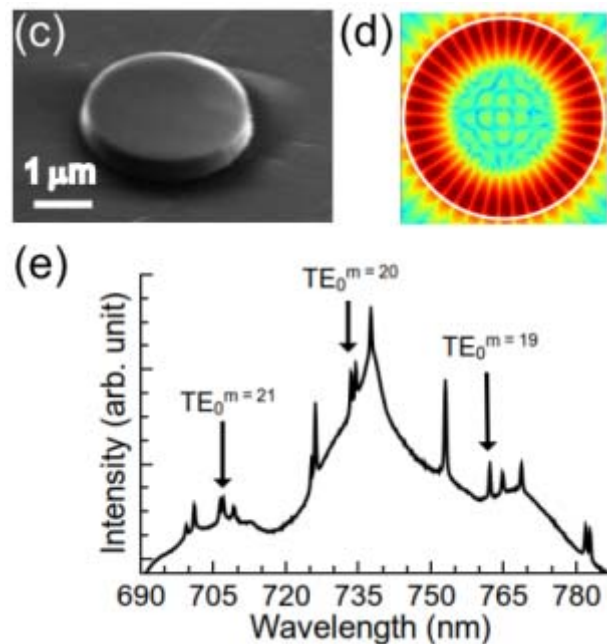
IBL in Diamond: State of the Art

Lift-off + CVD growth + RIE

Coupling of silicon-vacancy centers to a single crystal diamond cavity

Jonathan C. Lee,* Igor Aharonovich, Andrew P. Magyar, Fabian Rol,
and Evelyn L. Hu

9 April 2012 / Vol. 20, No. 8 / OPTICS EXPRESS 8891



IBL in Diamond: State of the Art

Lift-off + CVD growth + RIE

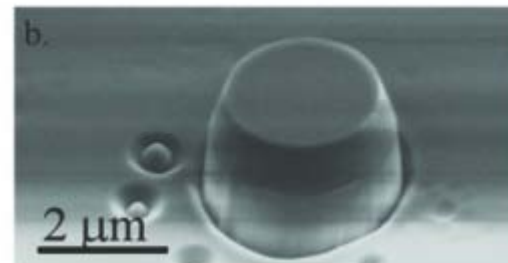
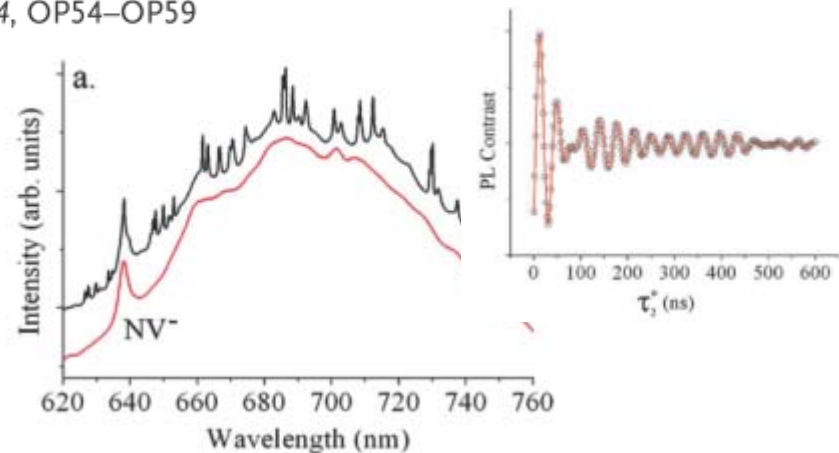
ADVANCED
OPTICAL
MATERIALS
www.advopticalmat.de

Materials
Views
www.MaterialsViews.com

Homoepitaxial Growth of Single Crystal Diamond Membranes for Quantum Information Processing

Igor Aharonovich,* Jonathan C. Lee, Andrew P. Magyar, Bob B. Buckley, Christopher G. Yale, David D. Awschalom, and Evelyn L. Hu

Adv. Mater. 2012, 24, OP54–OP59



Outline

- Diamond
 - Synthesis
 - Properties
 - Applications
- **IBL in diamond**
 - MeV ion lithography in diamond
 - **keV ion beam lithography in diamond**
- Activities at the University of Torino
 - Electrical features
 - Optical features
 - Microfluidics

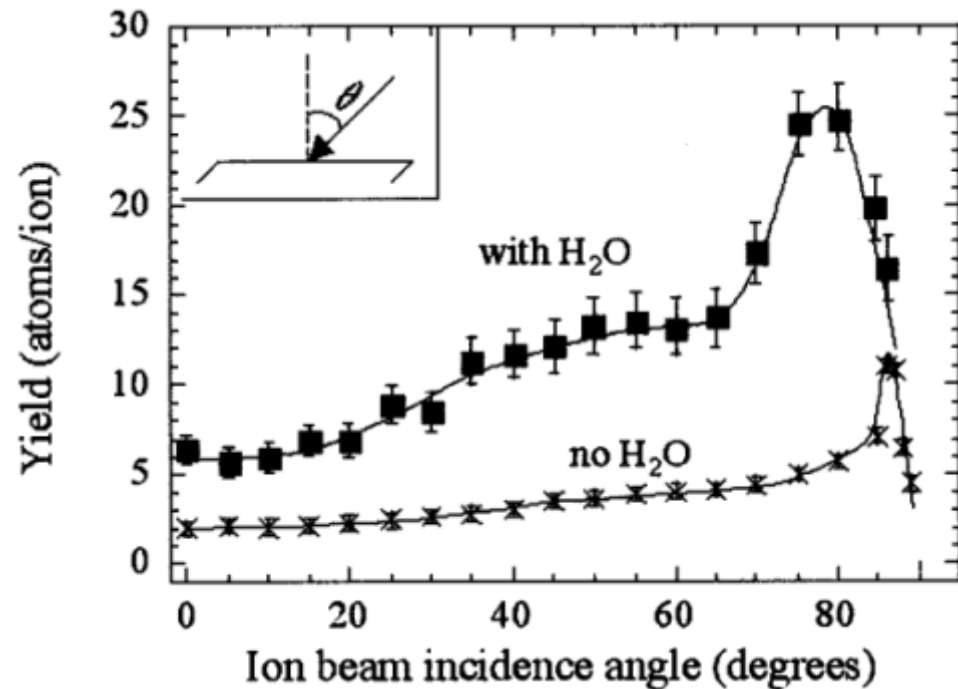
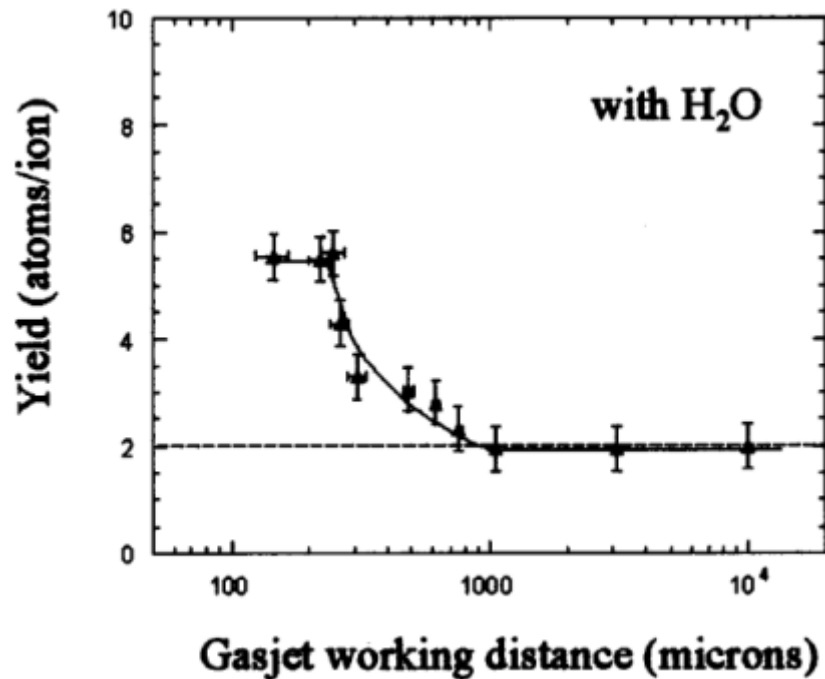
FIB in Diamond: State of the Art

Gas-assisted milling

Focused ion beam milling of diamond: Effects of H₂O on yield, surface morphology and microstructure

D. P. Adams,^{a)} M. J. Vasile, T. M. Mayer, and V. C. Hodges

2334 J. Vac. Sci. Technol. B 21(6), Nov/Dec 2003



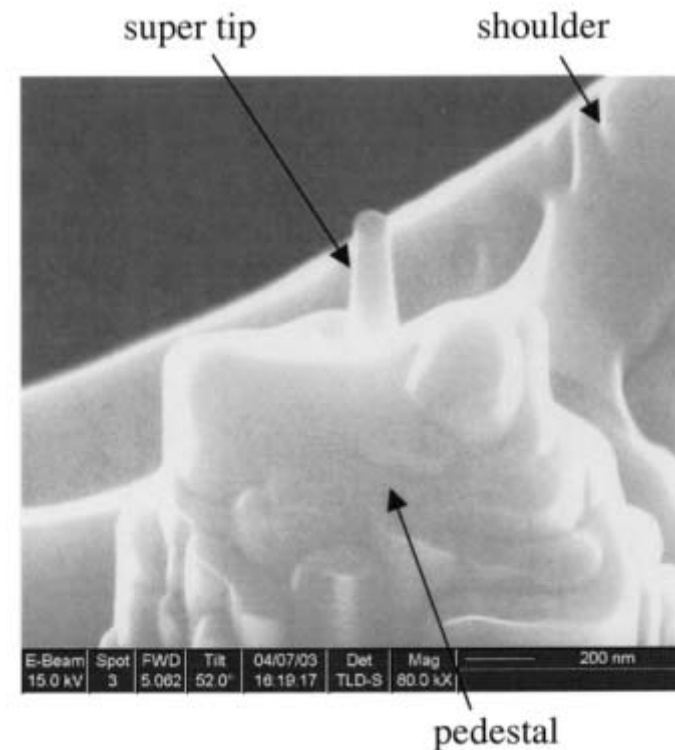
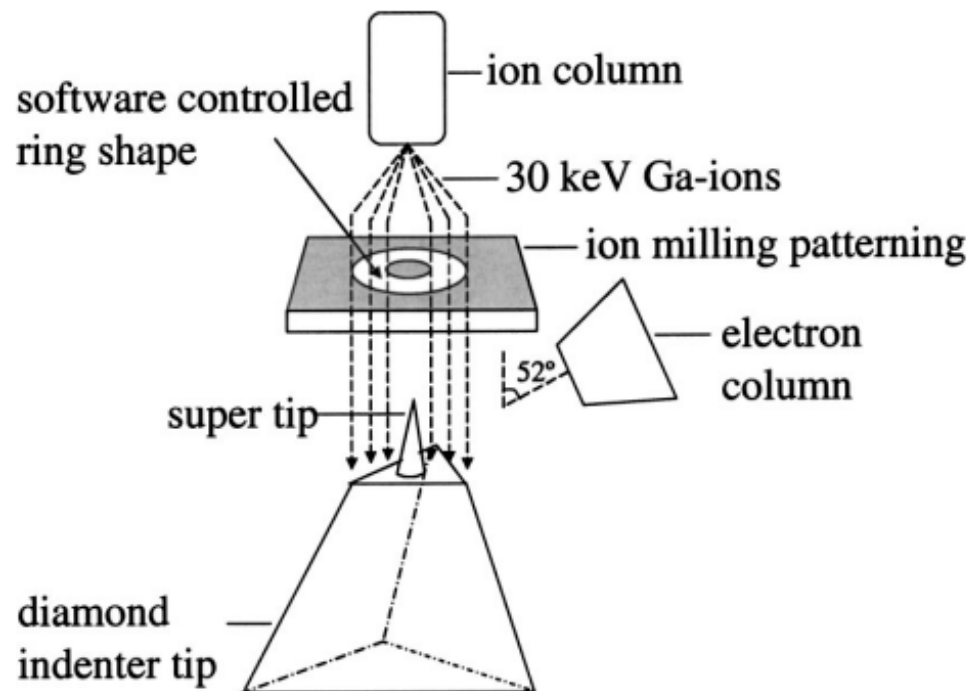
FIB in Diamond: State of the Art

Micro & nano-indenters

Use of the focused ion beam technique to produce a sharp spherical diamond indenter for sub-10 nm nanoindentation measurements

Ning Yu and Andreas A. Polycarpou^{a)}

668 J. Vac. Sci. Technol. B 22(2), Mar/Apr 2004



FIB in Diamond: State of the Art

Micro- & nano-indenters

IOP PUBLISHING

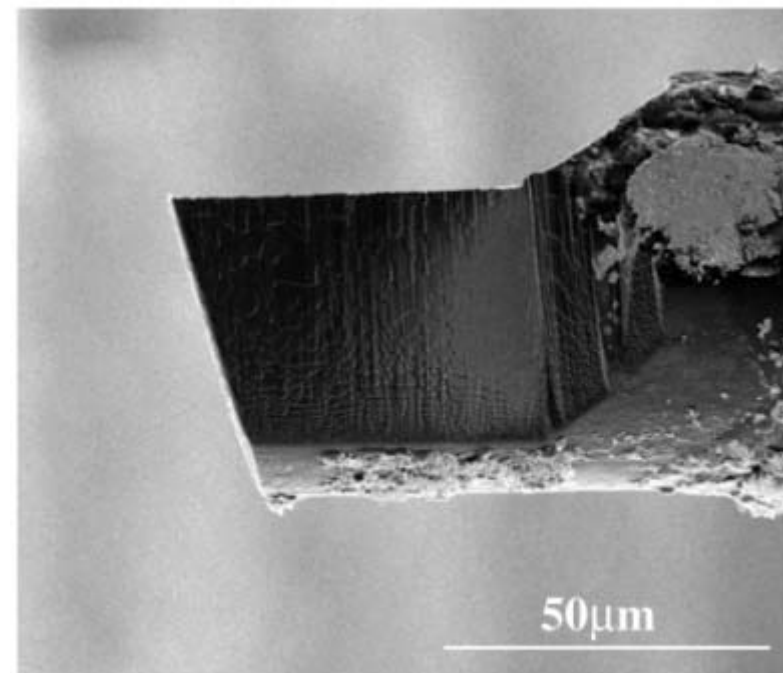
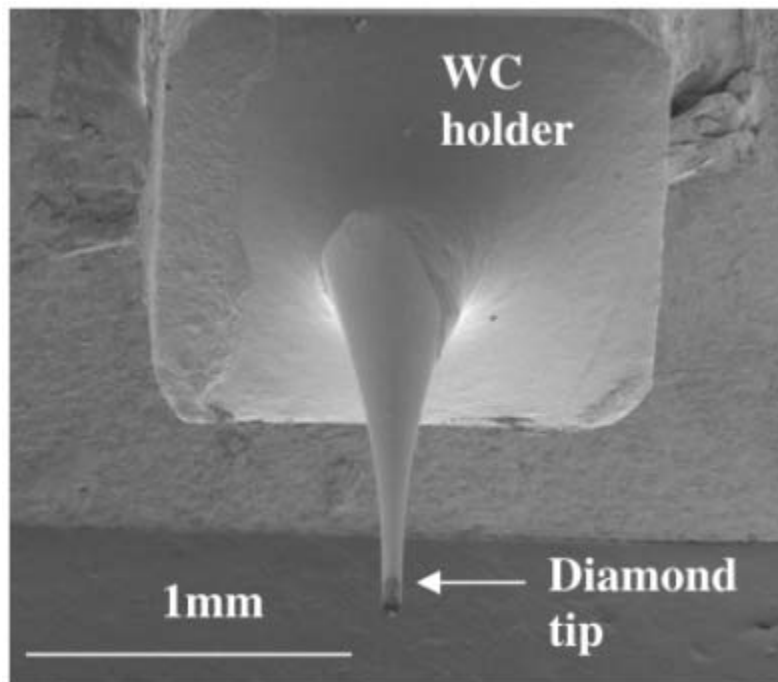
JOURNAL OF MICROMECHANICS AND MICROENGINEERING

J. Micromech. Microeng. 18 (2008) 075017 (10pp)

[doi:10.1088/0960-1317/18/7/075017](https://doi.org/10.1088/0960-1317/18/7/075017)

Fabrication of a micro-size diamond tool using a focused ion beam

X Ding¹, G C Lim¹, C K Cheng², David Lee Butler^{1,3}, K C Shaw¹, K Liu¹
and W S Fong¹



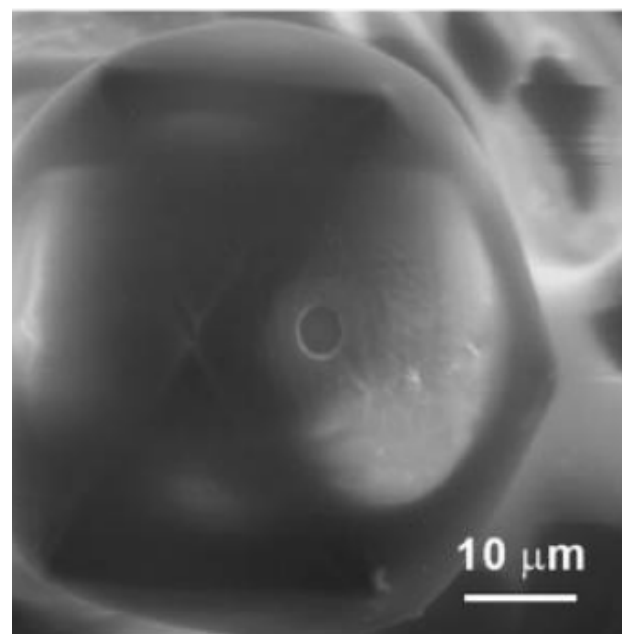
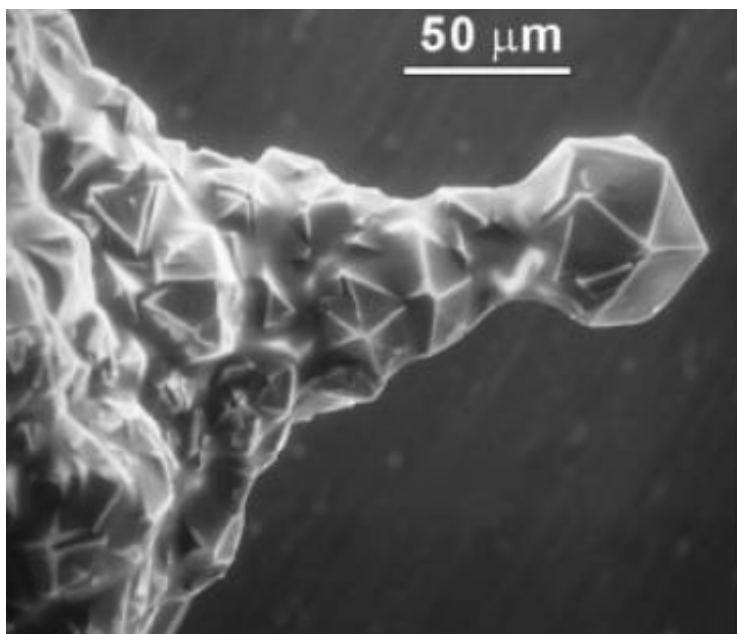
FIB in Diamond: State of the Art

Micro-electrodes

Anal. Chem. 2009, 81, 5663–5670

Focused Ion Beam Fabrication of Boron-Doped Diamond Ultramicroelectrodes

Jingping Hu,[†] Katherine B. Holt,[‡] and John S. Foord^{*,†}



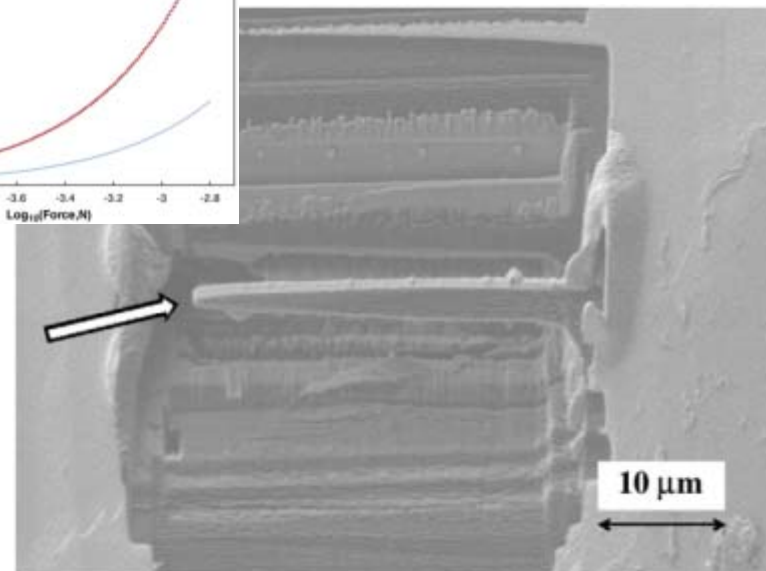
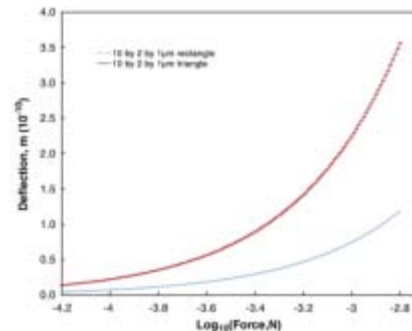
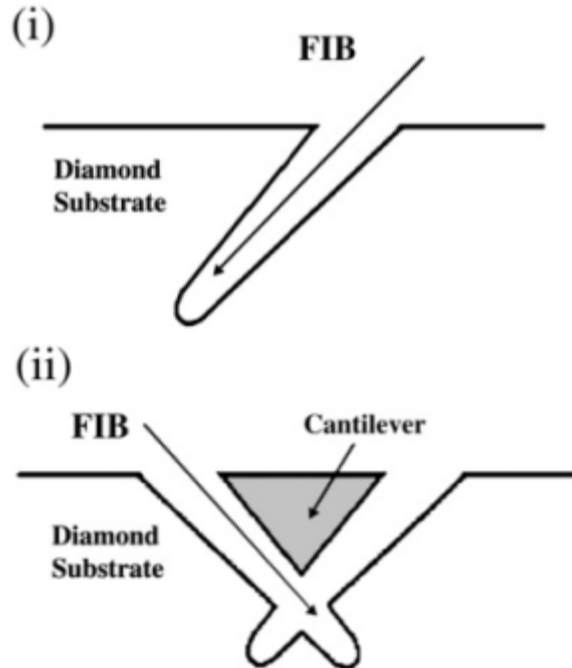
FIB in Diamond: State of the Art

Micro-mechanical structures



Fabrication and characterisation of triangle-faced single crystal diamond micro-cantilevers

Benjamin Z. Kupfer, Rezal K. Ahmad, Aiman Zainal, Richard B. Jackman*

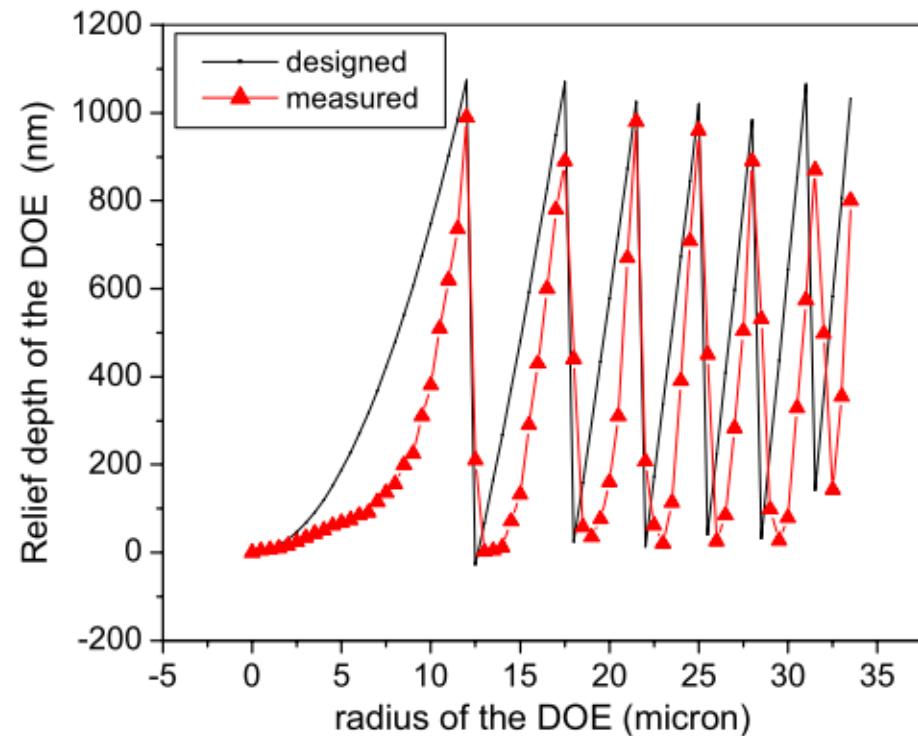
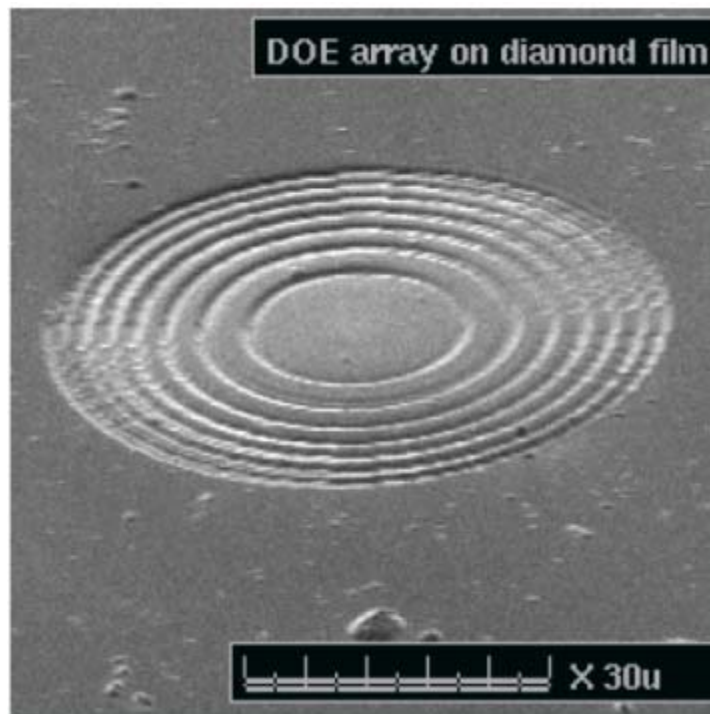


FIB in Diamond: State of the Art

Diffractive optical elements

Investigation of diffractive optical element fabricated on diamond film by use of focused ion beam direct milling

Yongqi Fu Ngoi Kok Ann Bryan Opt. Eng. 42(8) 2214 (August 2003)



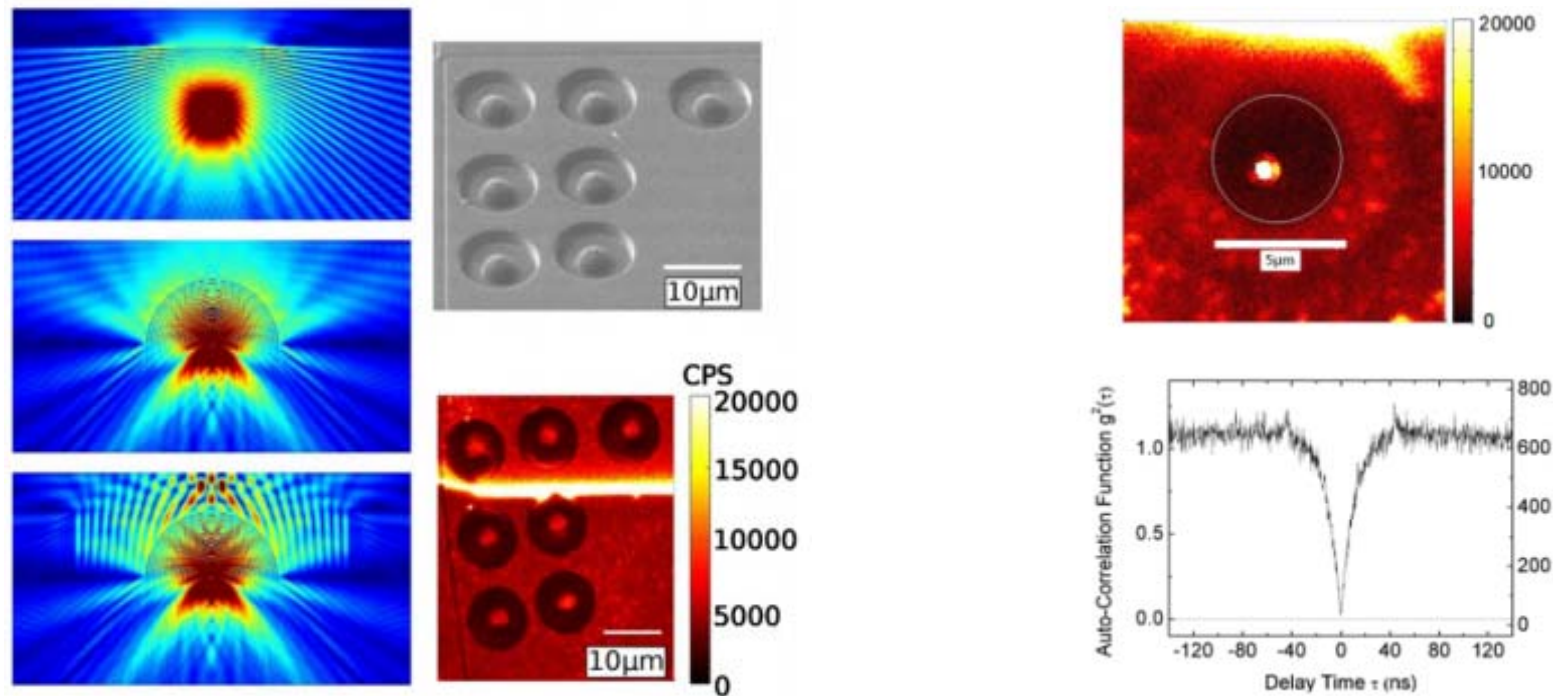
FIB in Diamond: State of the Art

Integrated micro-optics

APPLIED PHYSICS LETTERS 97, 241901 (2010)

Strongly enhanced photon collection from diamond defect centers under microfabricated integrated solid immersion lenses

J. P. Hadden,^{a)} J. P. Harrison, A. C. Stanley-Clarke, L. Marseglia, Y.-L. D. Ho, B. R. Patton, J. L. O'Brien, and J. G. Rarity



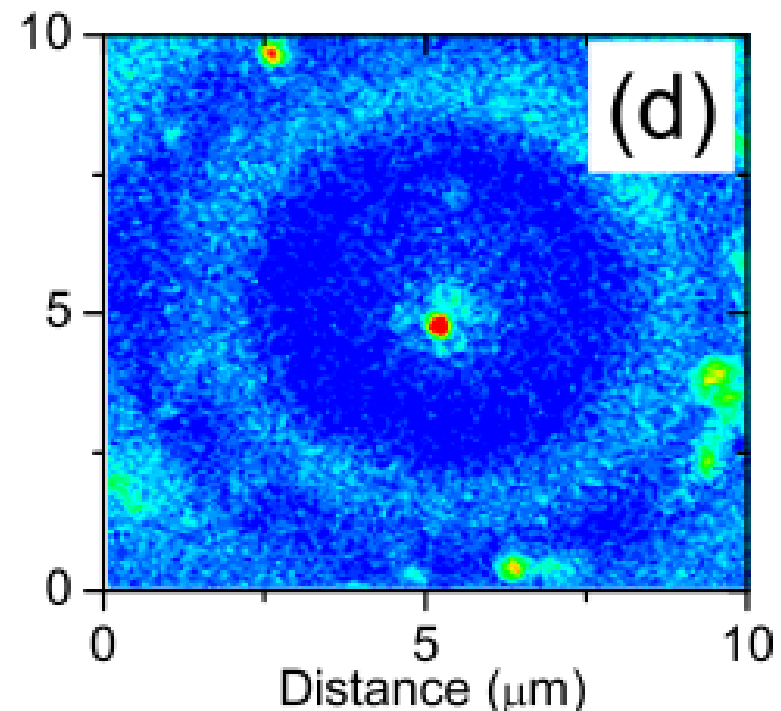
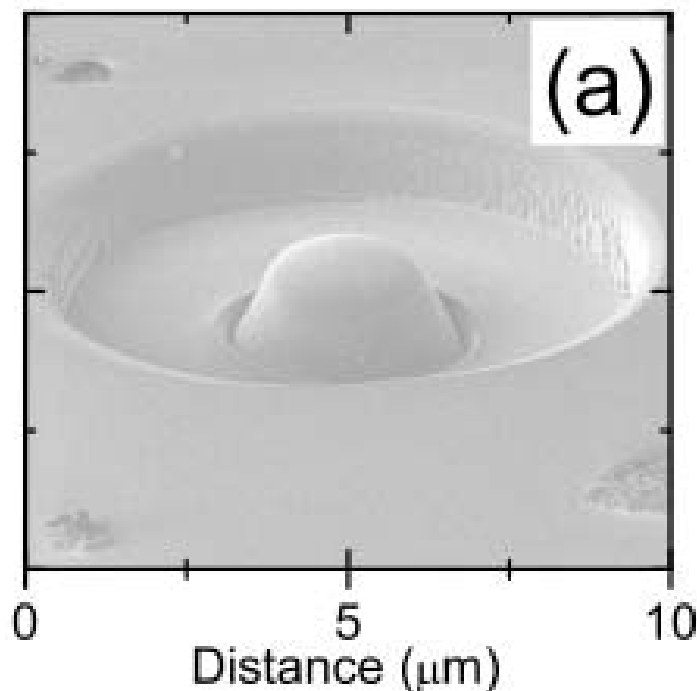
FIB in Diamond: State of the Art

Integrated micro-optics

APPLIED PHYSICS LETTERS 98, 133107 (2011)

Nanofabricated solid immersion lenses registered to single emitters in diamond

L. Marseglia,^{1,a)} J. P. Hadden,¹ A. C. Stanley-Clarke,¹ J. P. Harrison,¹ B. Patton,¹
Y.-L. D. Ho,¹ B. Naydenov,² F. Jelezko,² J. Meijer,³ P. R. Dolan,⁴ J. M. Smith,⁴ J. G. Rarity,⁴
and J. L. O'Brien



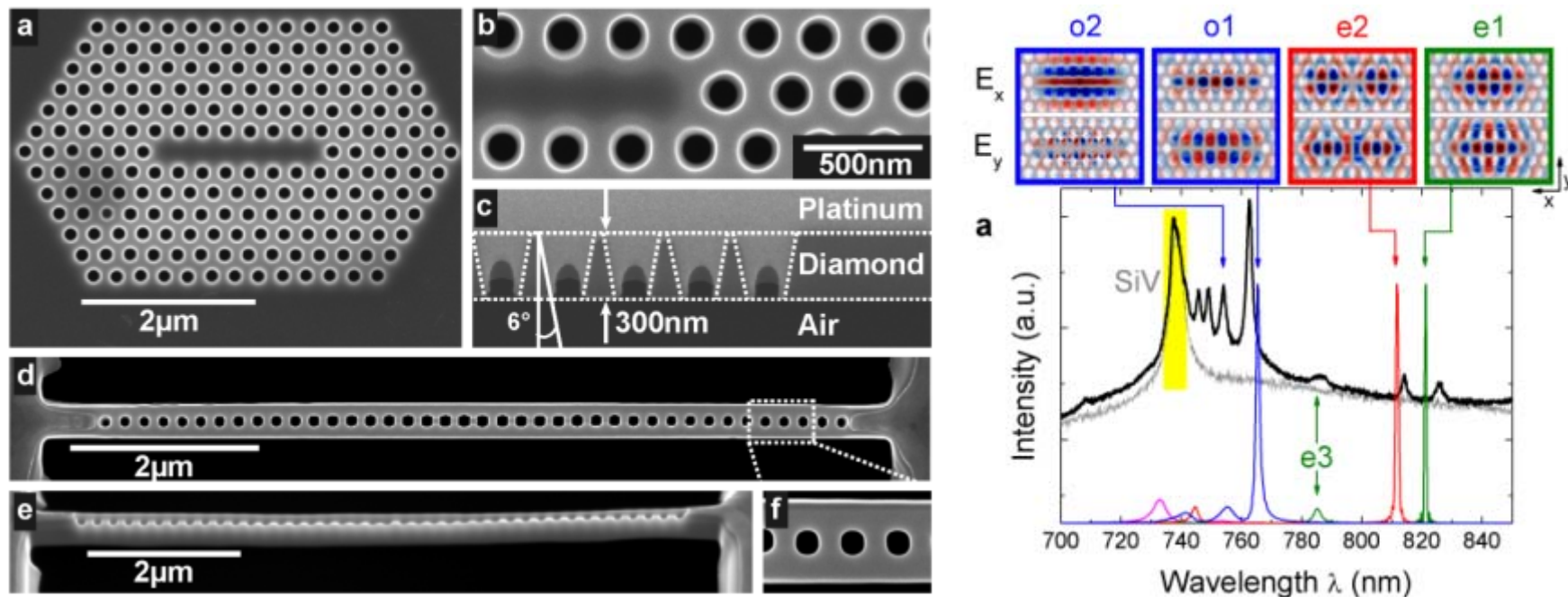
FIB in Diamond: State of the Art

Photonic structures

One- and two-dimensional photonic crystal micro-cavities in single crystal diamond

Janine Riedrich-Möller¹, Laura Kipfstuhl¹, Christian Hepp¹, Elke Neu¹,
Christoph Pauly², Frank Mücklich², Armin Baur³, Michael Wandt³,
Sandra Wolff⁴, Martin Fischer⁵, Stefan Gsell⁵, Matthias Schreck⁵, and
Christoph Becher^{1*}

Nature Nanotechnology 7, 69-74 (2012)



FIB in Diamond: State of the Art

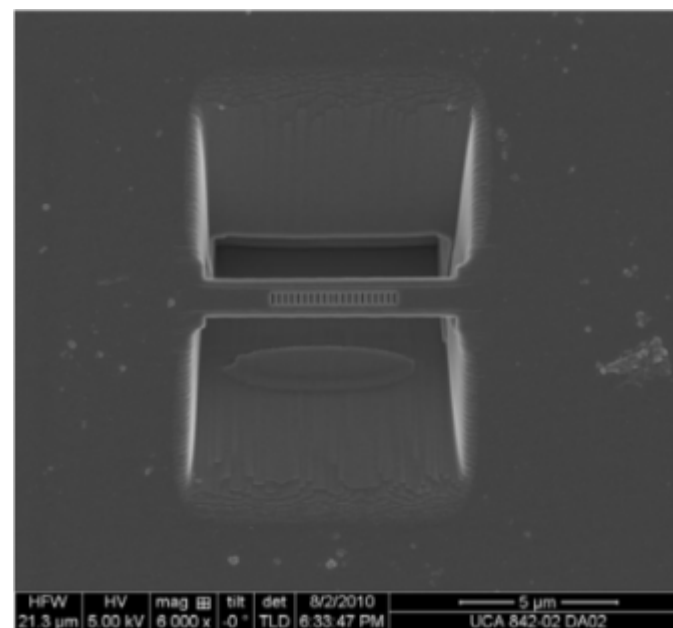
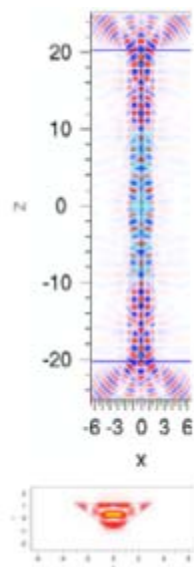
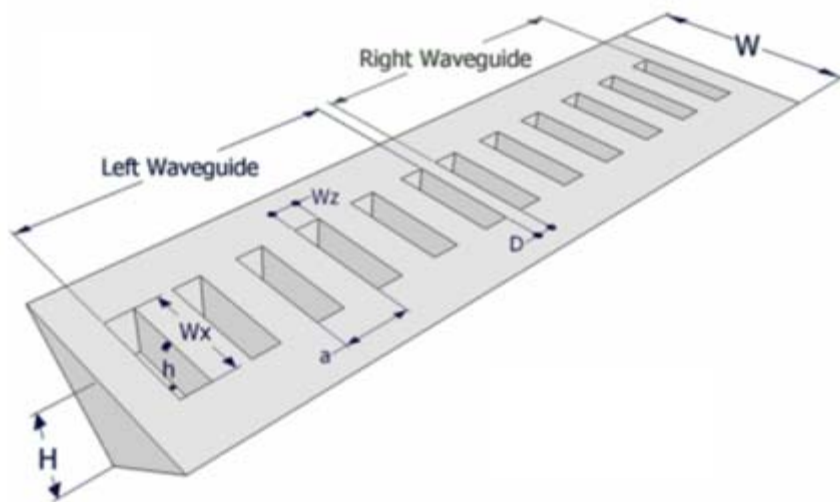
Photonic structures



Triangular nanobeam photonic cavities in single-crystal diamond

Igal Bayn^{1,3}, Boris Meyler¹, Joseph Salzman¹ and Rafi Kalish²

New Journal of Physics **13** (2011) 025018



FIB in Diamond: State of the Art

Photonic structures

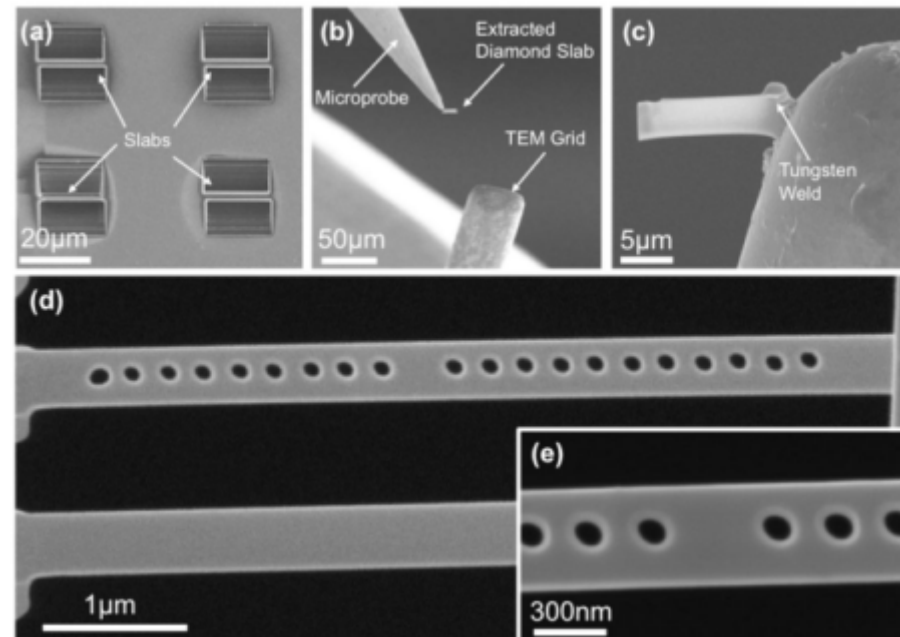
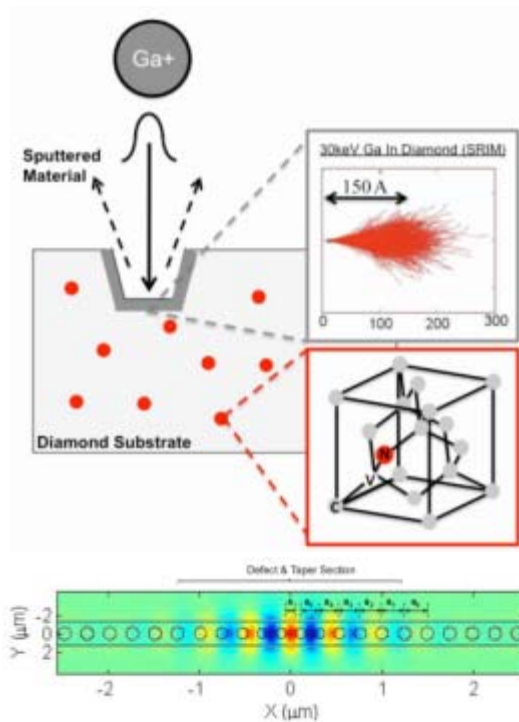
Design and Focused Ion Beam Fabrication of Single Crystal Diamond

Nanobeam Cavities

Thomas M. Babinec¹, Jennifer T. Choy¹, Kirsten J. M. Smith^{1,2}, Mughees Khan^{1,3}, Marko

Lončar¹

arXiv: 1008.1431



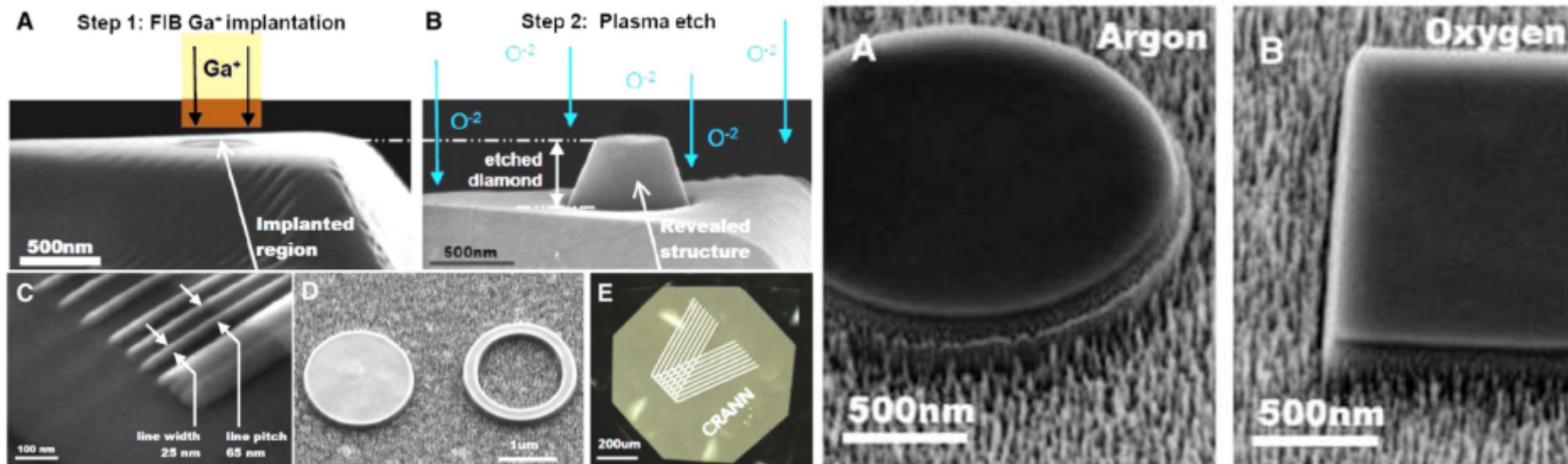
FIB in Diamond: State of the Art

Direct-write negative lithography



A direct-write, resistless hard mask for rapid nanoscale patterning of diamond

Warren McKenzie¹, John Pethica, Graham Cross*

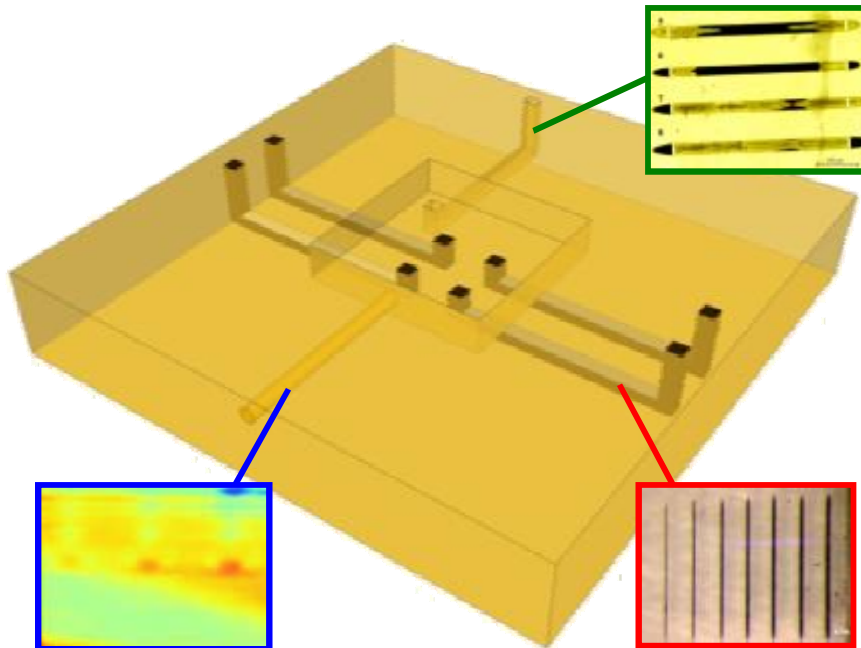


Outline

- Diamond
 - Synthesis
 - Properties
 - Applications
- IBL in diamond
 - MeV ion lithography in diamond
 - keV ion beam lithography in diamond
- **Activities at the University of Torino**
 - Electrical features
 - Optical features
 - Microfluidics

Activities at the University of Torino

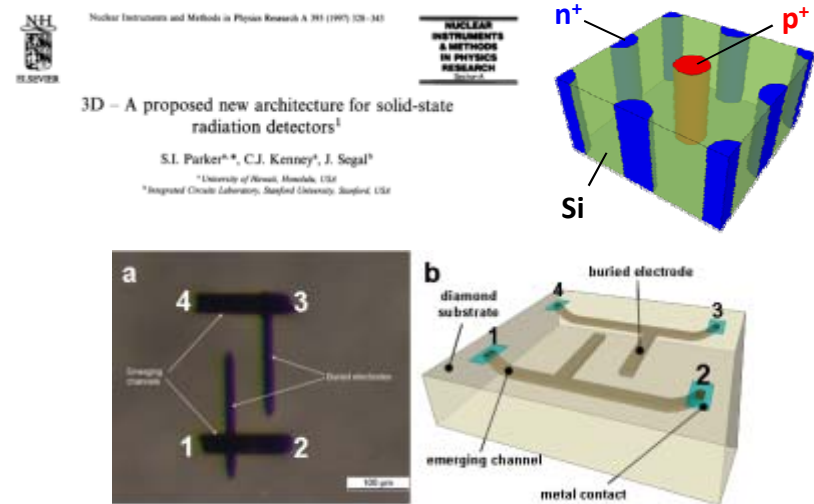
Cellular bio-sensing devices



A bio-compatible and transparent diamond active substrate for interfacing with single excitable cells:

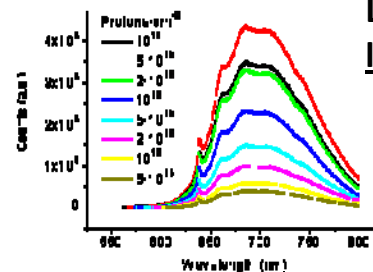
- ✓ **electrical** interfacing: sub-superficial micro-electrodes
- ✓ **optical** interfacing: integrated waveguiding structures
- ✓ **chemical** interfacing: microfluidic devices

Three-dimensional particle detectors



A novel geometry for induced charge collection in diamond detectors

Single photon sources



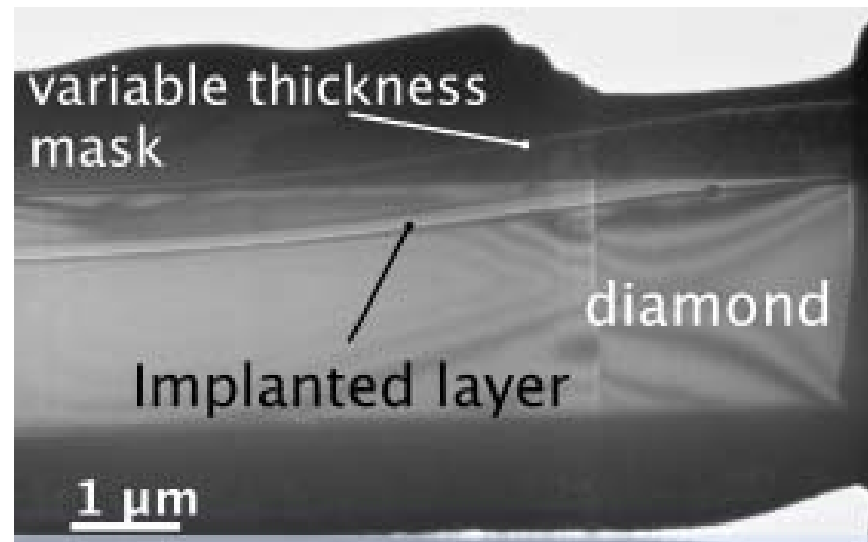
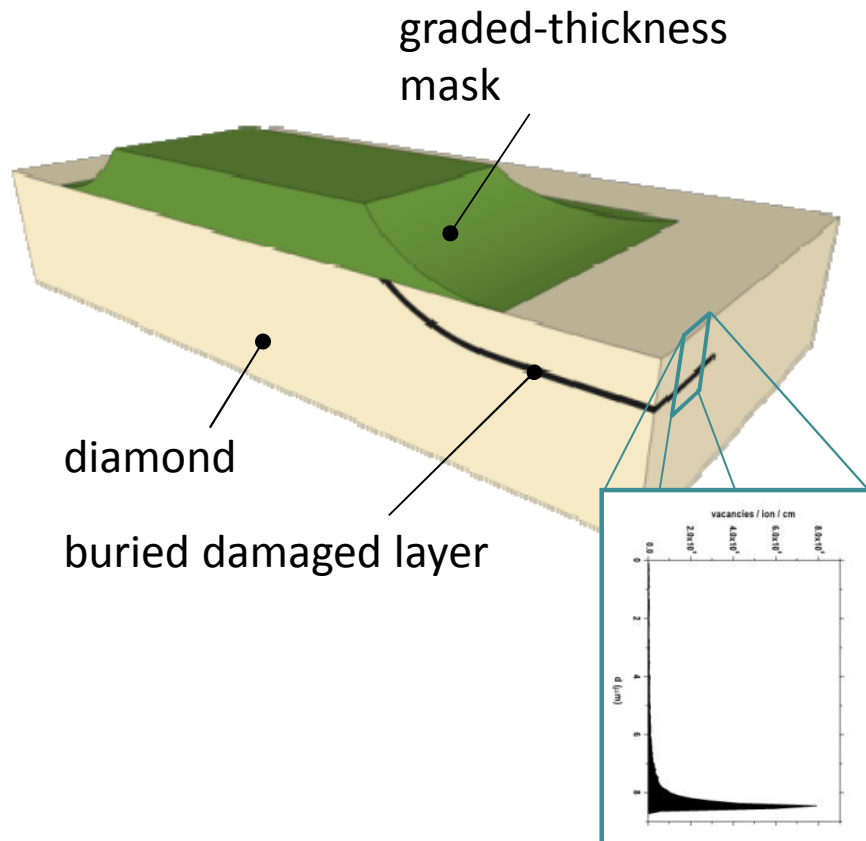
Light emitters based on luminescent centers in diamond

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Electrical features

Implantation with a scanning MeV ion micro-beam
through graded-thickness mask



✓ definition of buried graphitic microchannels in single-crystal diamond

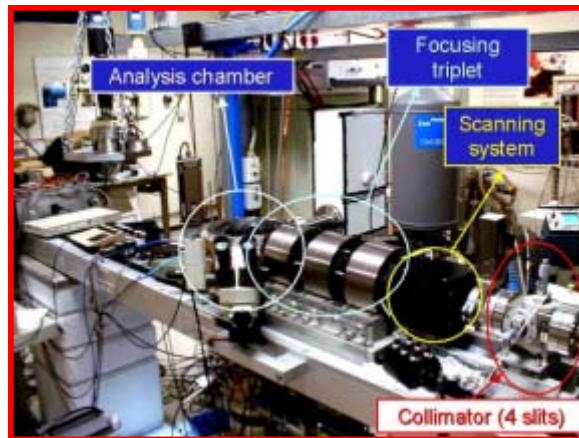
Electrical features

Ion implantation performed at the
MP2-UniMelb, **LNL-AN200** and **Ruđer Bošković Institute** ion microbeam lines



**Structural
TEM characterization**

**0.5 MeV He⁺
F=1·10¹⁷ cm⁻²**



**Cellular bio-sensing
3D particle detectors**

**1.8 MeV He⁺
F=1·10¹⁶ - 1·10¹⁷ cm⁻²**

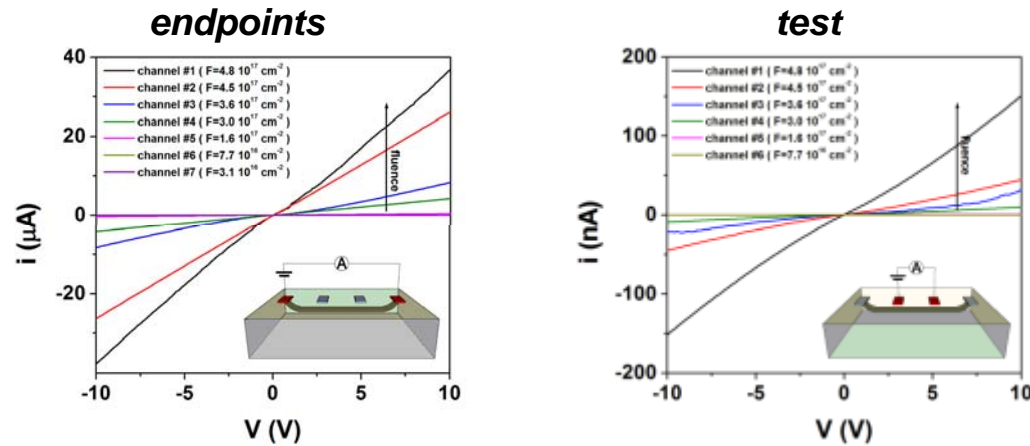


3D particle detectors

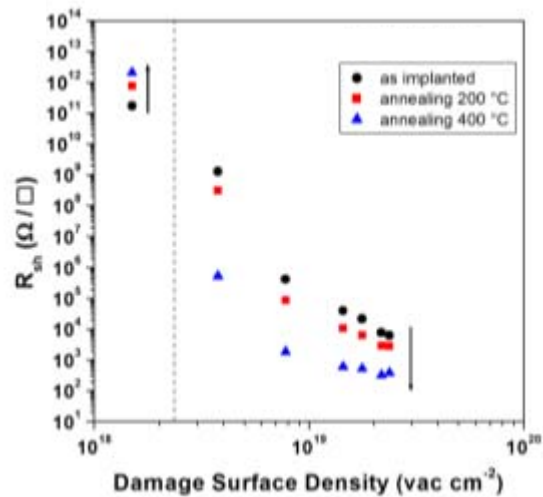
**6 MeV C³⁺
F=4·10¹⁶ cm⁻²**

Electrical features

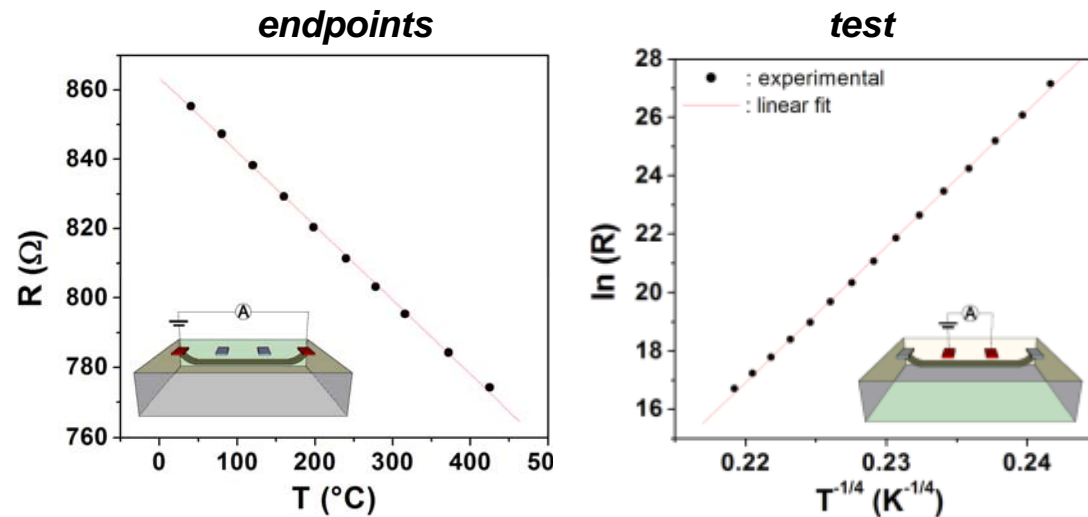
IV curves @ increasing fluences



Annealing behavior

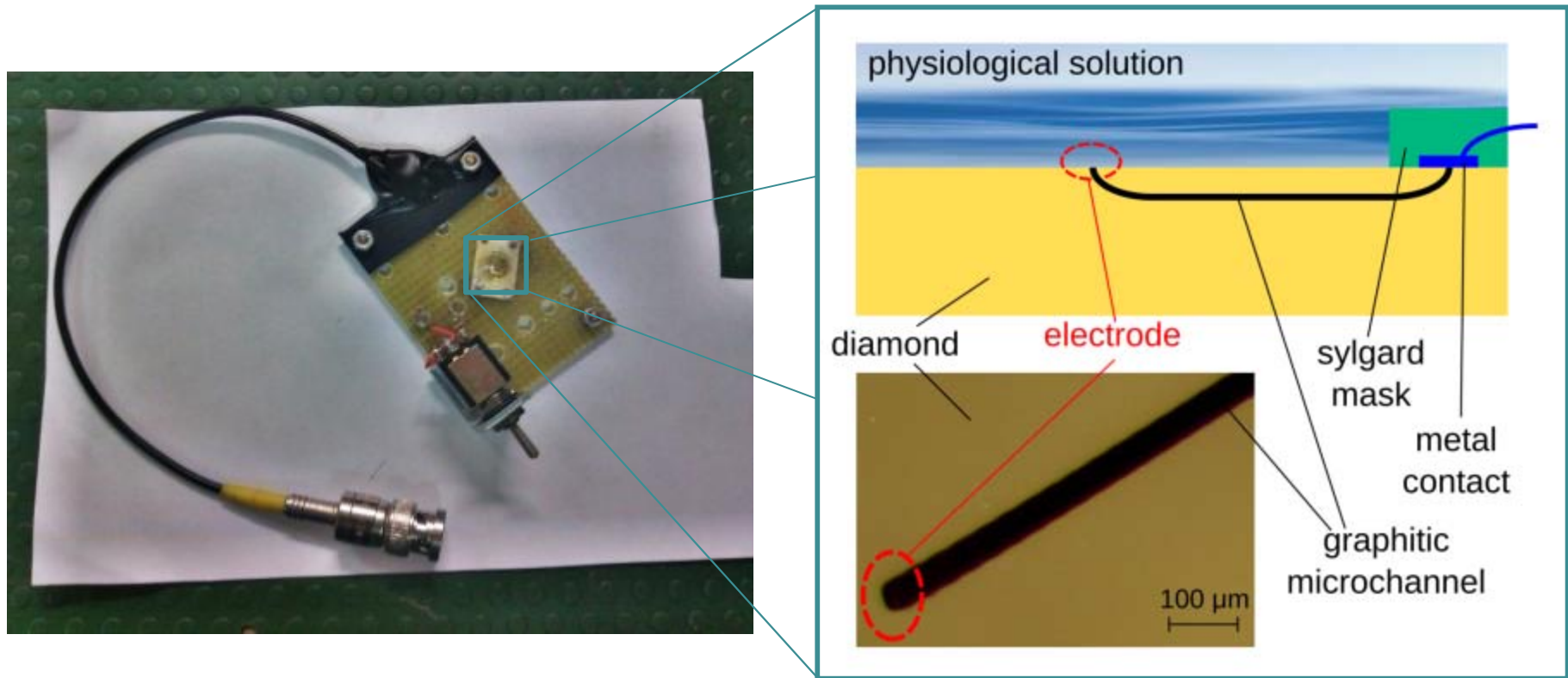


IV measurements in temperature



Electrical features

Amperometric detection of **exocytosis** activity from **neuroendocrine** cells

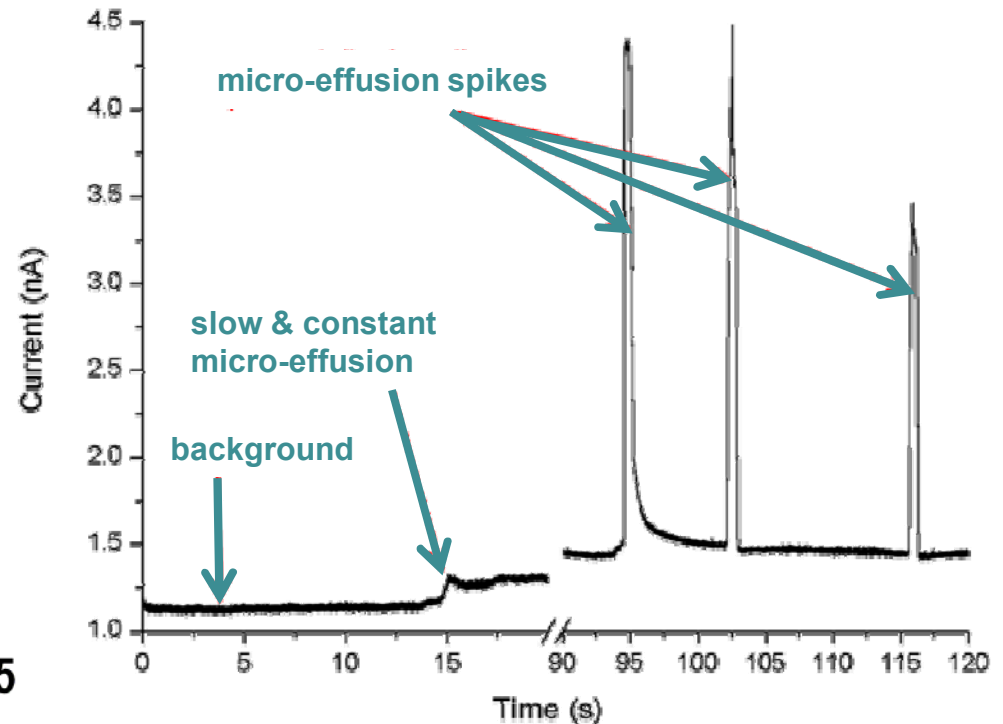
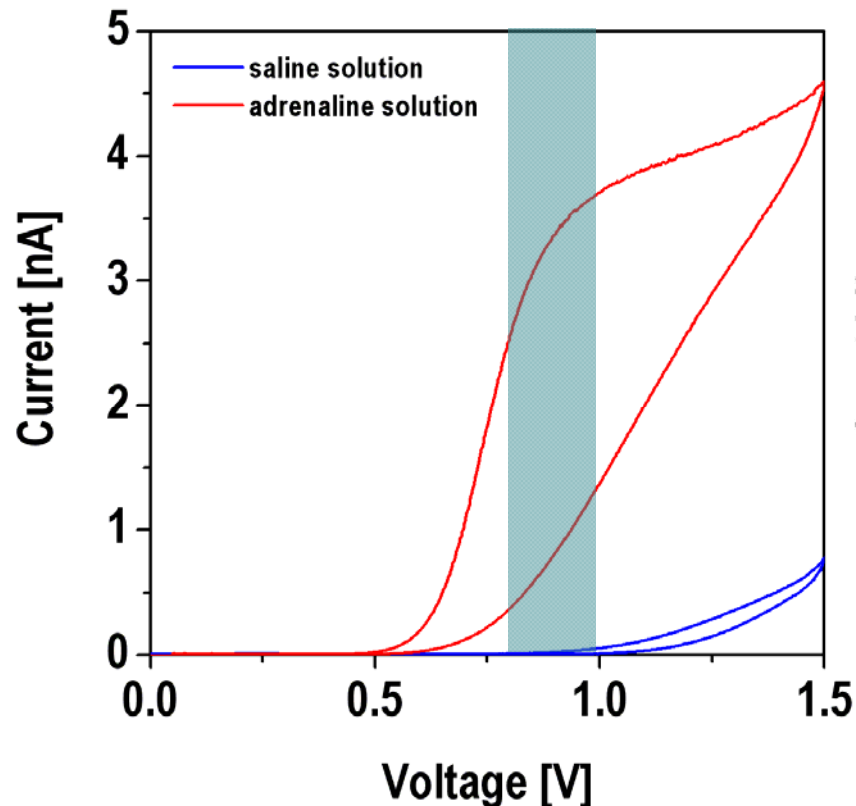


- $3 \times 3 \times 1.5 \text{ mm}^2$ type Ib single crystal diamond grown by HPHT technique

Electrical features

Cyclic voltammetry: oxidation of catecholamines (adrenaline, noradrenaline, etc.) at the biased electrode

Preliminary sensitivity test: micro-effusion of adrenaline with a syringe



Saline solution: NaCl (128 mM), MgCl₂ (2 mM), glucose (10 mM), HEPES (10 mM), CaCl₂ (10 mM), KCl (4 mM)

Adrenaline solution: saline solution, adrenaline (10 mM)

Electrical features

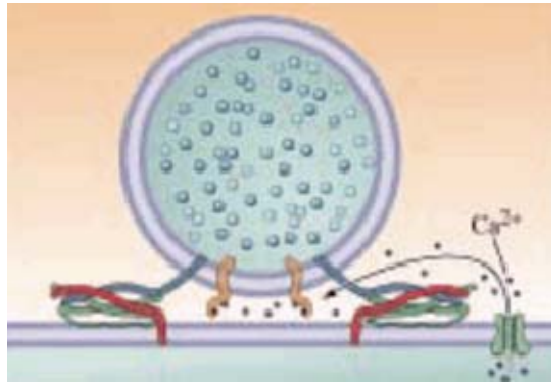
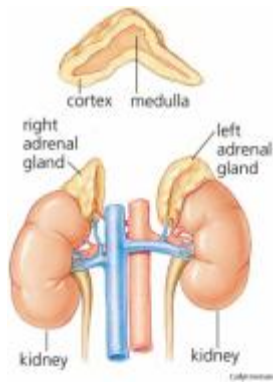
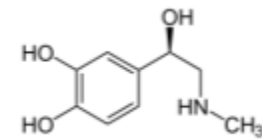
Amperometric detection of **exocytosis** (*quantal release of molecules*) from **chromaffin cells** (*neuroendocrine cells located into the medulla part of the renal gland*): oxidation of adrenaline at the **0.8 V** biased micro-electrode

Chromaffine cells: $\varnothing \sim 10 \mu\text{m}$

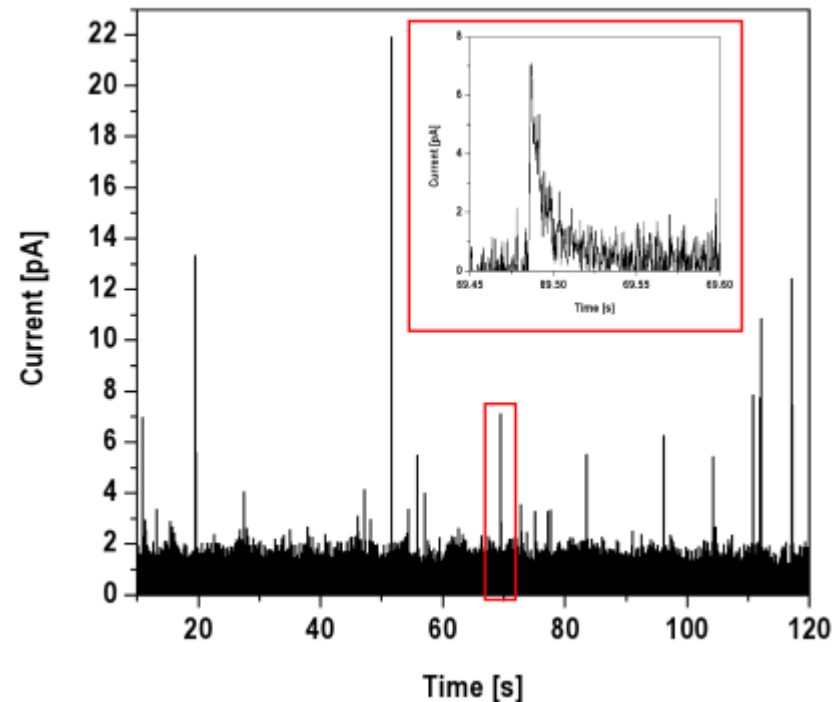
containing 50-300 nm chromaffin granules

adrenaline concentration in the granules: $0.5 - 1 \text{ M}$

exocytosis: process related to synaptic transmission



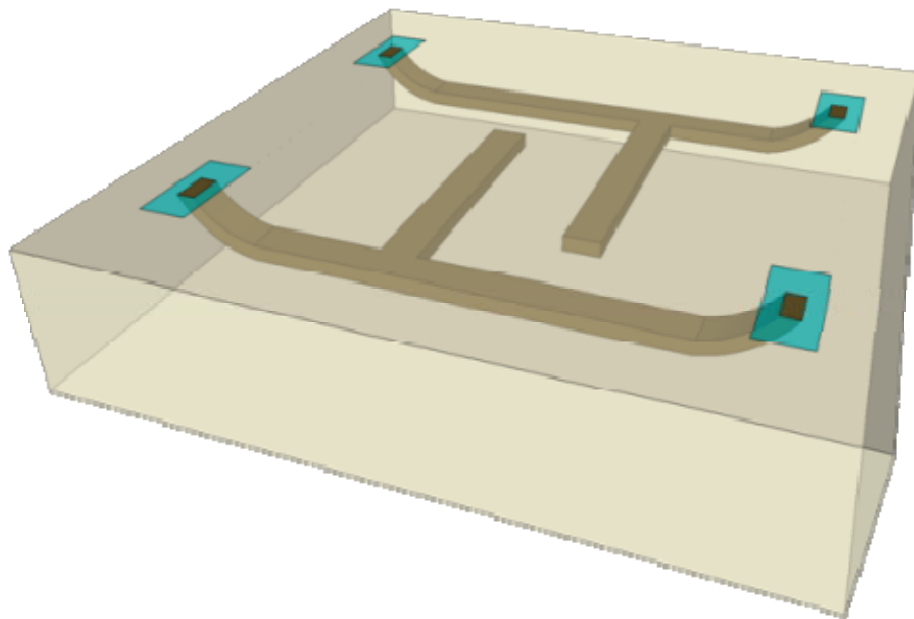
Signal detection: good performance (i.e. comparable to standard carbon fibers) in a fully **miniaturized, bio-compatible** and **transparent** device



F. Picollo et al., "Fabrication of a Diamond-Based Cellular Biosensor by Ion Beam Lithography to detect quantal exocytic events from chromaffin cells", *in preparation* (2012)

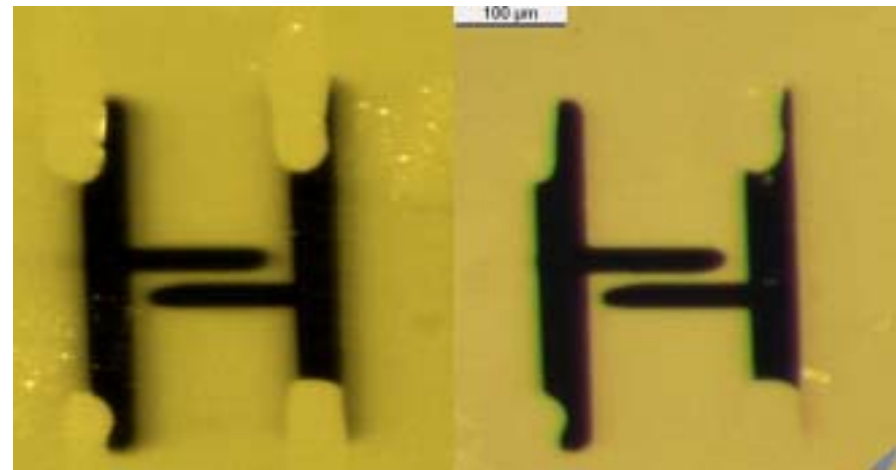
Electrical features

Buried graphitic electrodes in 3D particle detectors



before annealing

after annealing



implantation @ LNL

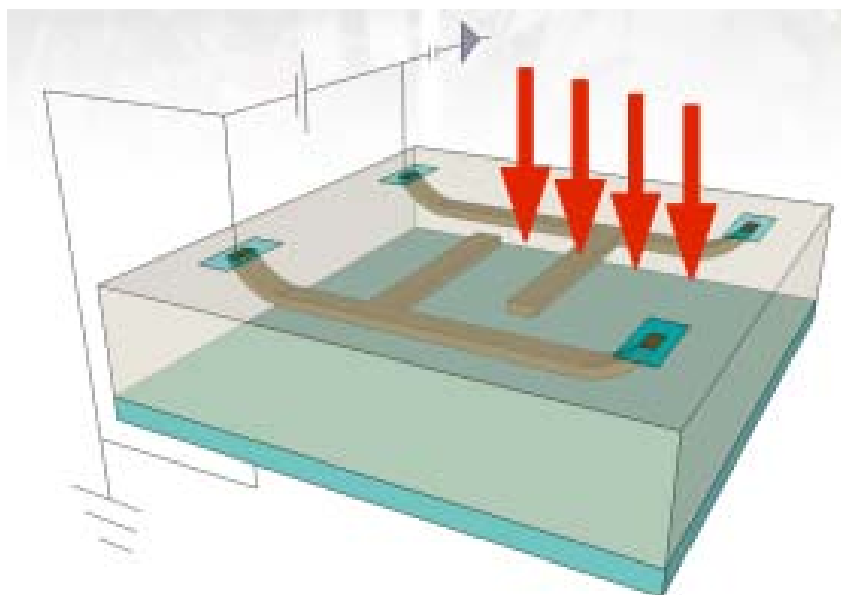
Implantation conditions:

1.8 MeV He⁺ (F = 3·10¹⁷ cm⁻²) at LNL

6 MeV C³⁺ (F = 4·10¹⁶ cm⁻²) at RBI

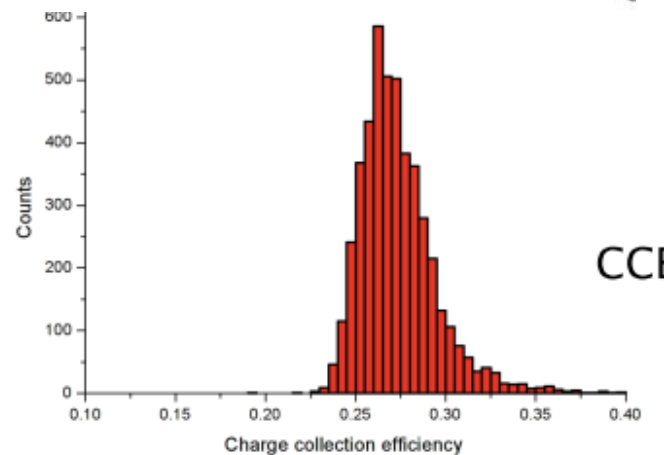
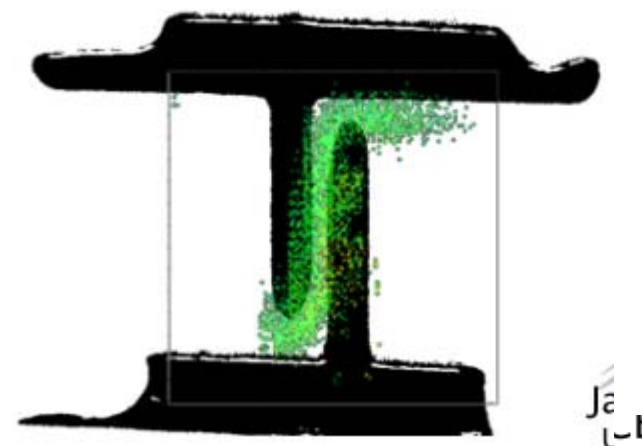
Electrical features

Charge collection characterization with Ion Beam Induced Charge (IBIC) measurements



IBIC probe ions: 2.0 MeV He⁺ at LNL
2.0 MeV H⁺ at RBI

measurements @ LNL

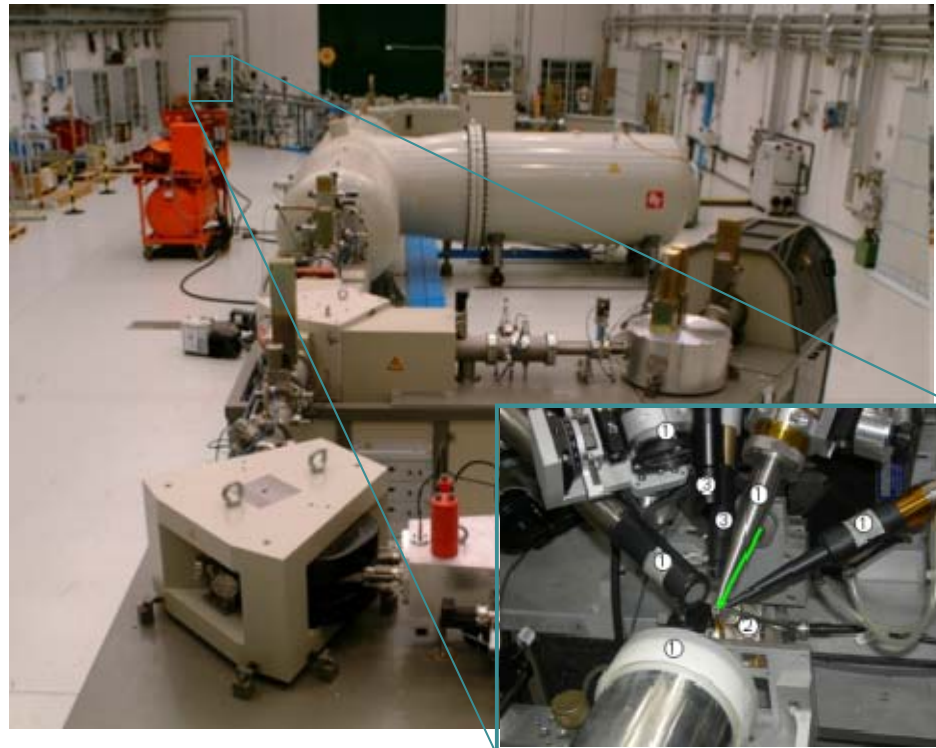


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 - **Optical features**
 - Microfluidics

Optical features

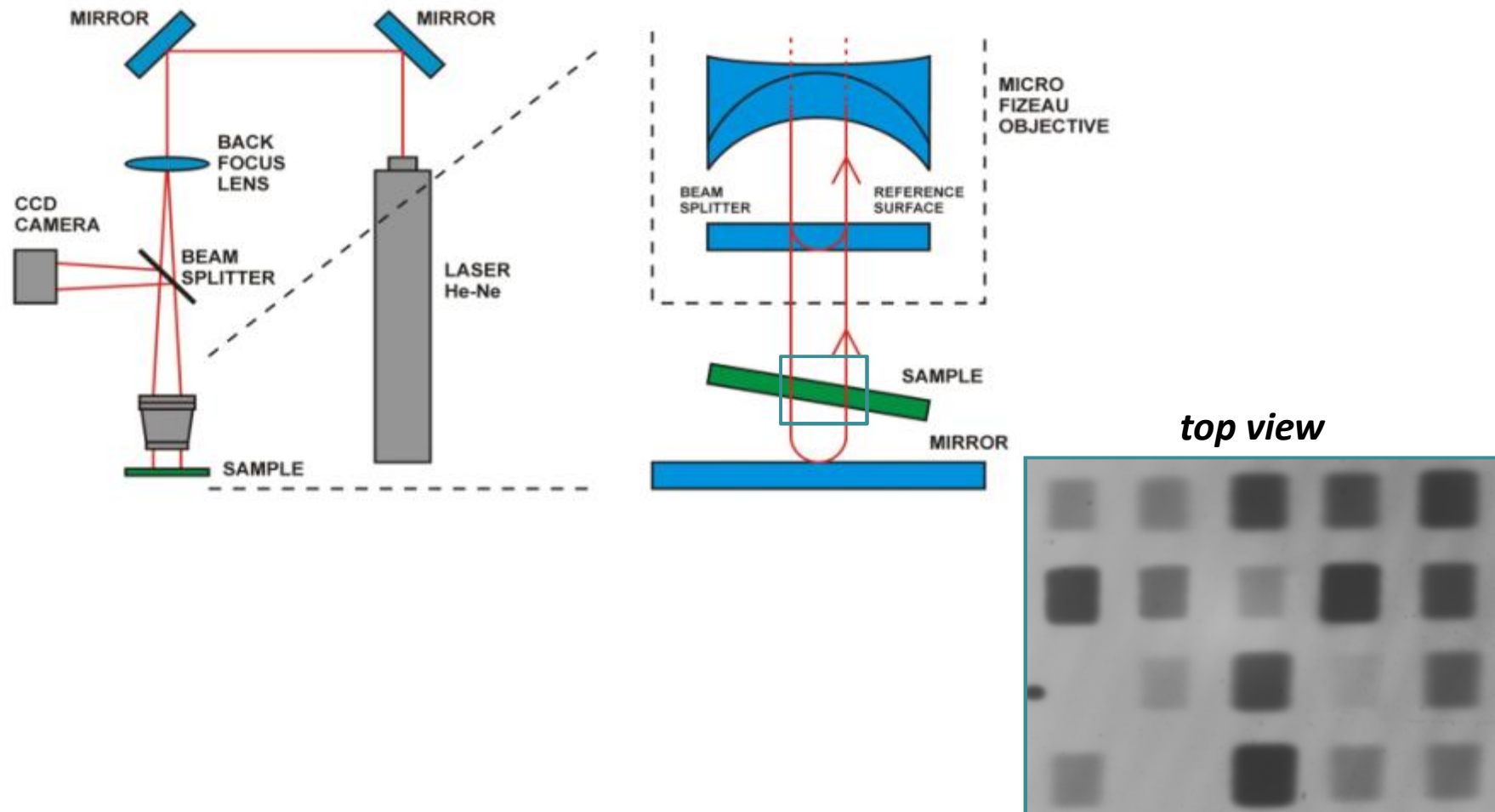
Ion implantation performed at the
LABEC external ion microbeam line



2, 3 MeV H implantation at increasing fluences ($F = 10^{13} - 10^{17} \text{ cm}^{-2}$)
over $100 \times 100 \mu\text{m}^2$ areas

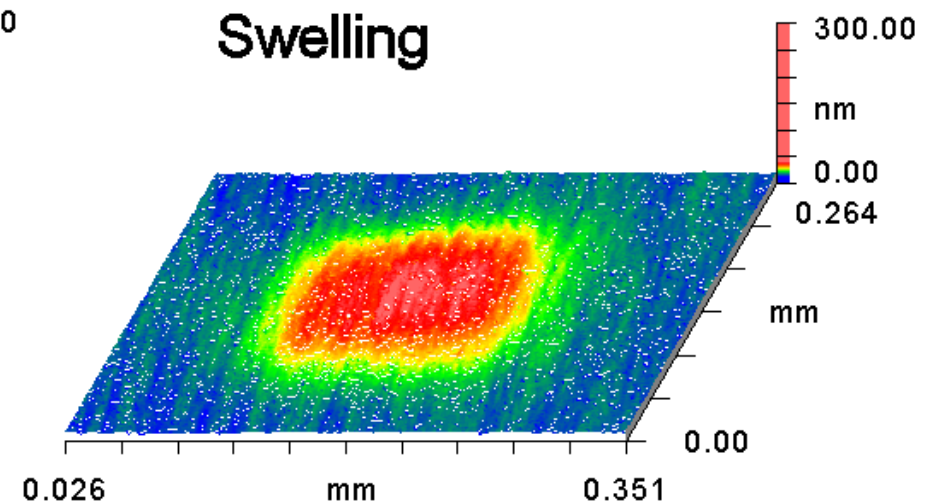
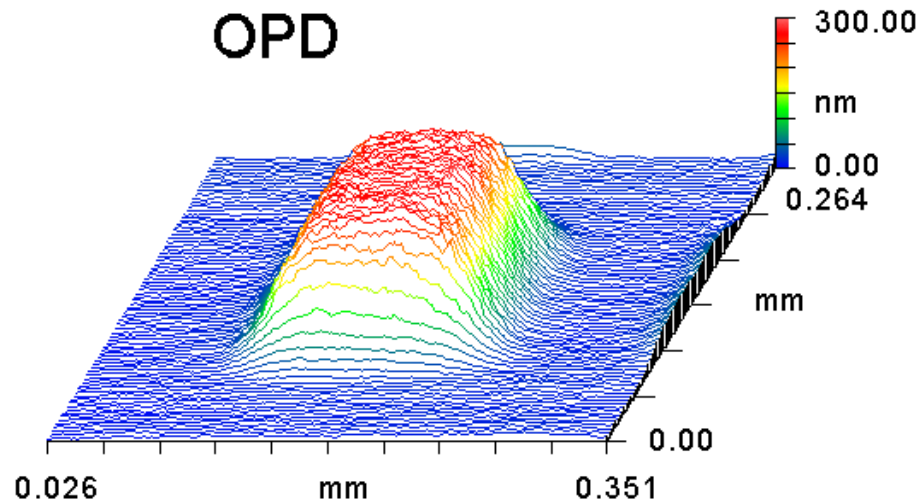
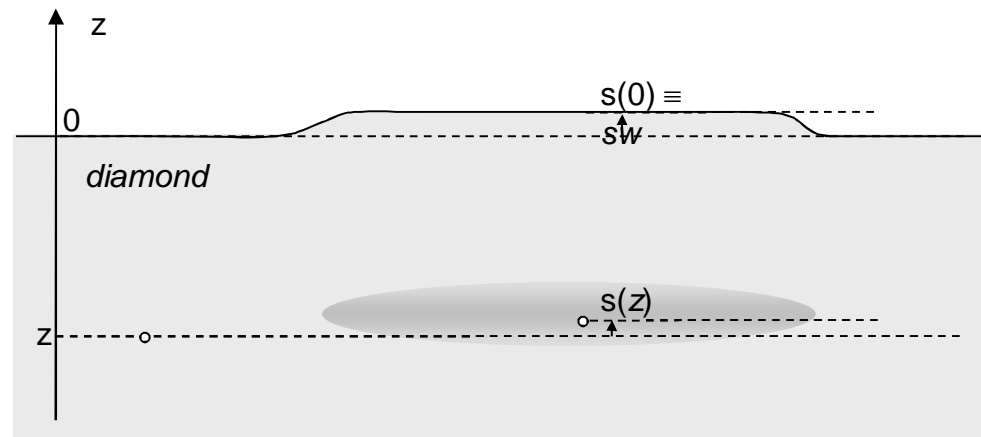
Optical features

Laser interferometric characterization



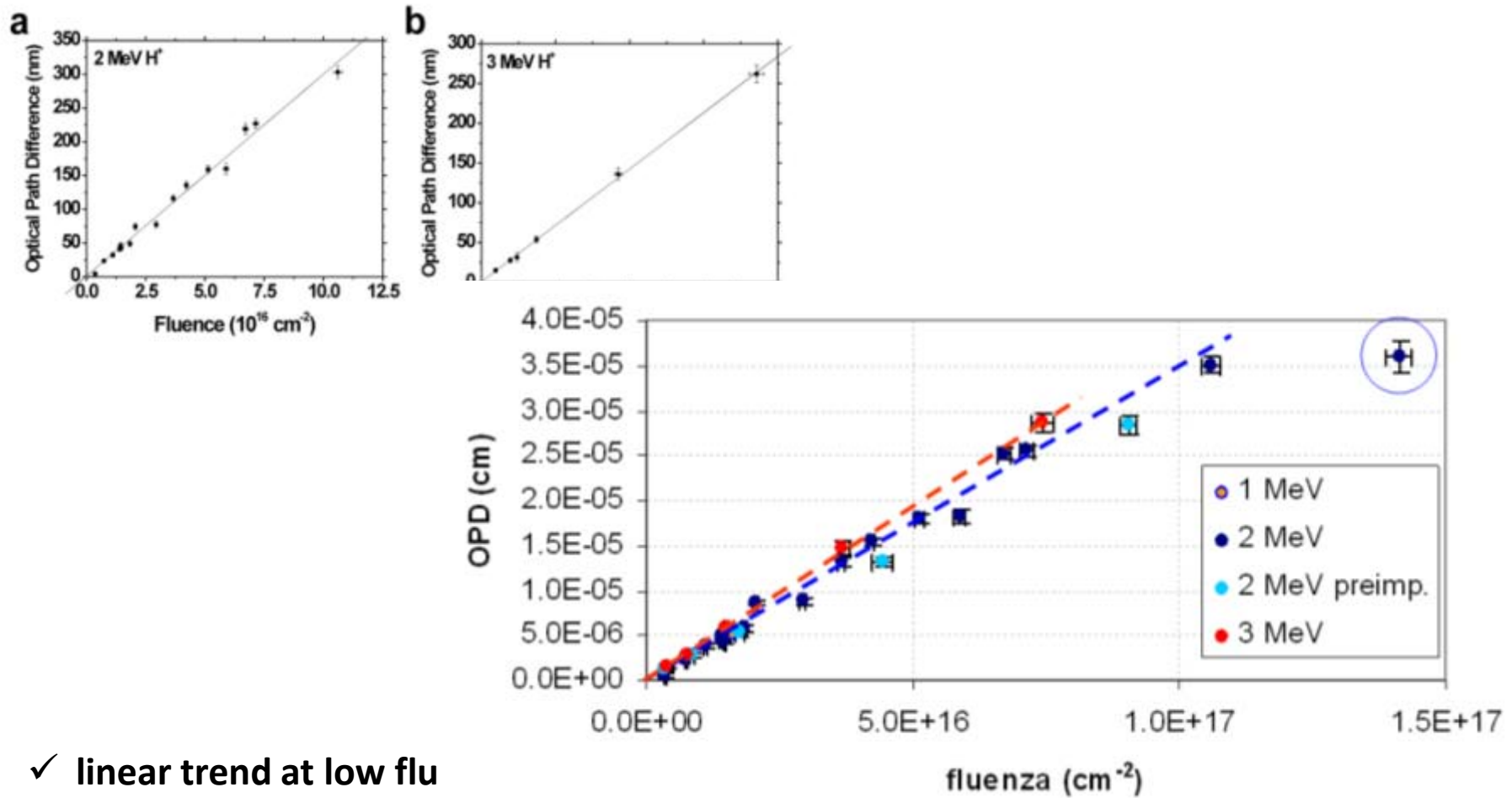
Optical features

Laser interferometric characterization



Optical features

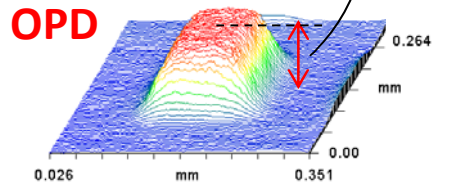
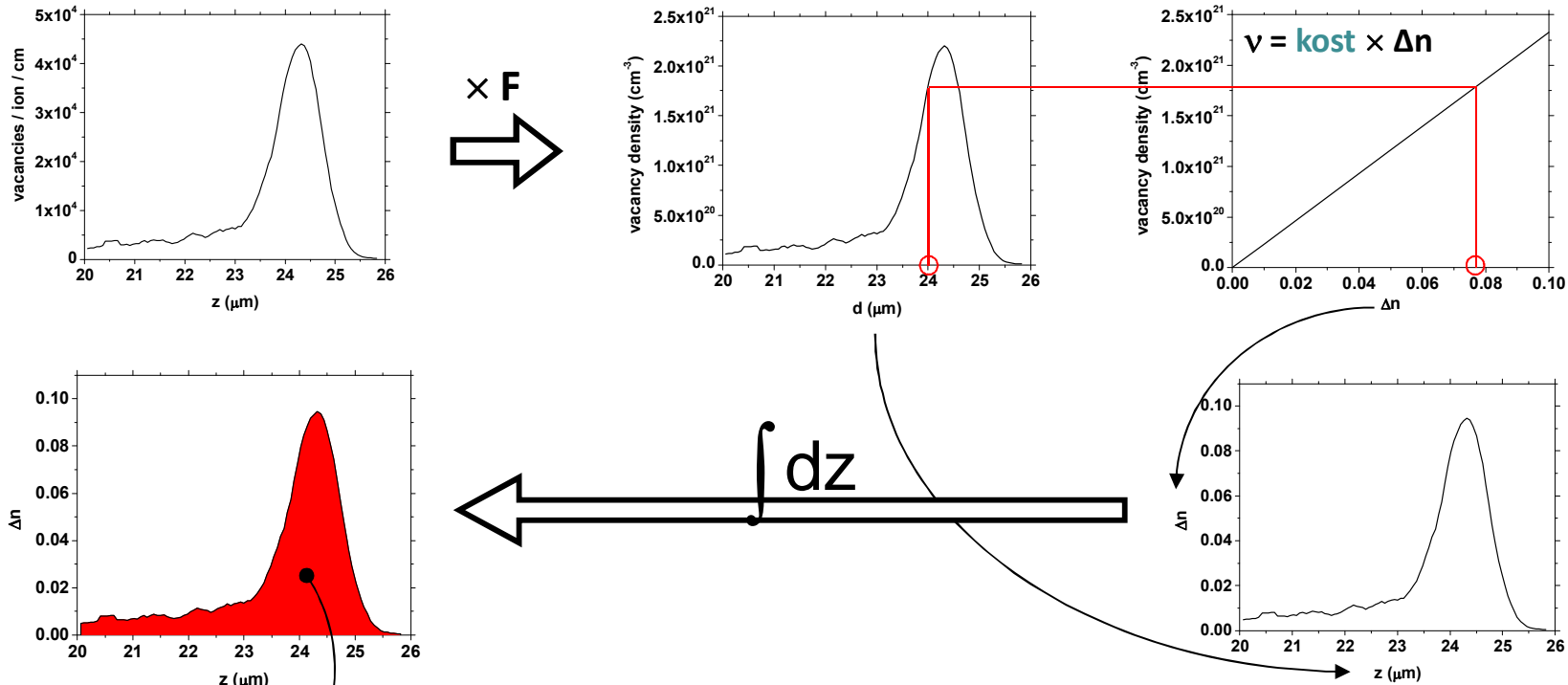
Optical path difference (OPD) vs fluence



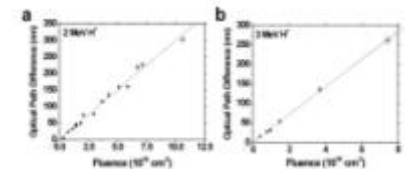
- ✓ linear trend at low flu
- ✓ sub-linear trend at high fluences
- ✓ different trends for 2 & 3 MeV protons

Optical features

Model: integration of the OPD along the depth direction



for a given value of **kost**: $\text{OPD} = \text{OPD} (F)$
 \rightarrow linear fitting of the experimental data
 $\Delta n = (4.3 \pm 0.3) \cdot 10^{-23} \times v [\text{cm}^{-3}]$



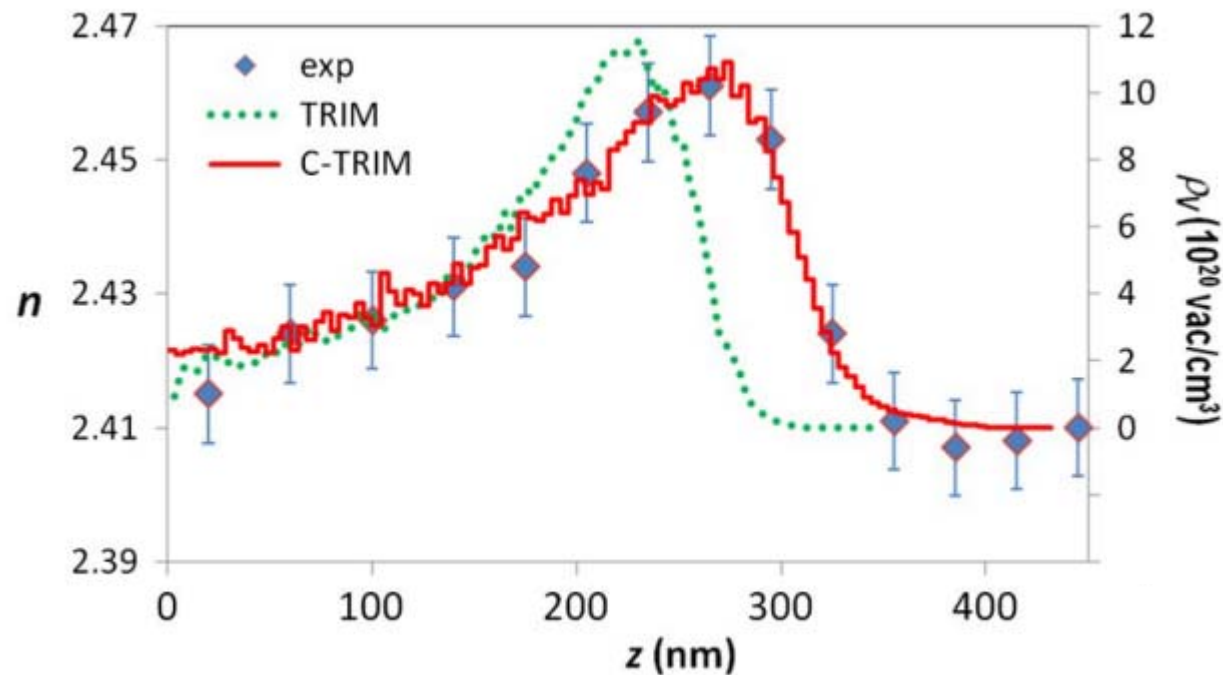
P. Olivero et al., *Diamond and Related Materials* **19**, 428 (2010)
 S. Lagomarsino et al., *Optics Express* **20** (17), 19382 (2012)

Optical features

Ellipsometric characterization of shallow-ion-implanted diamond

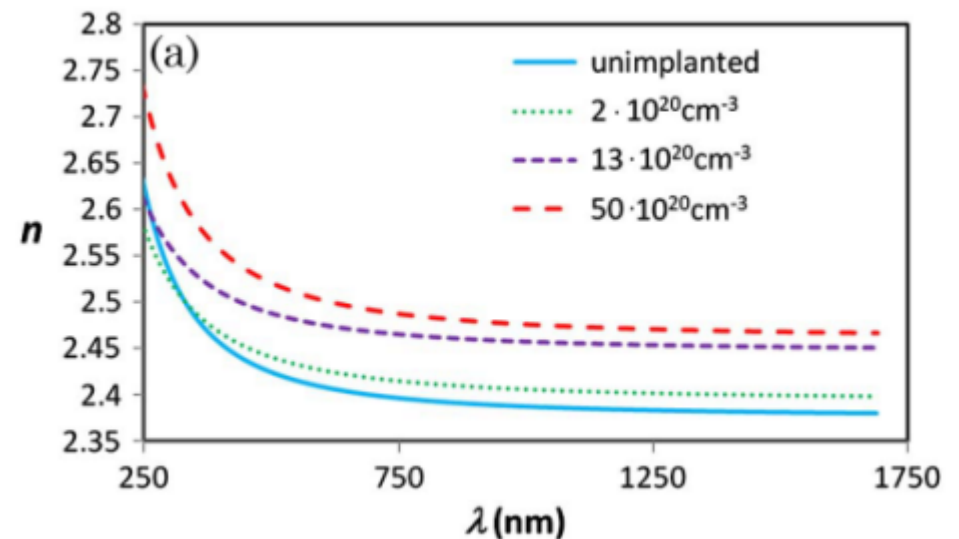
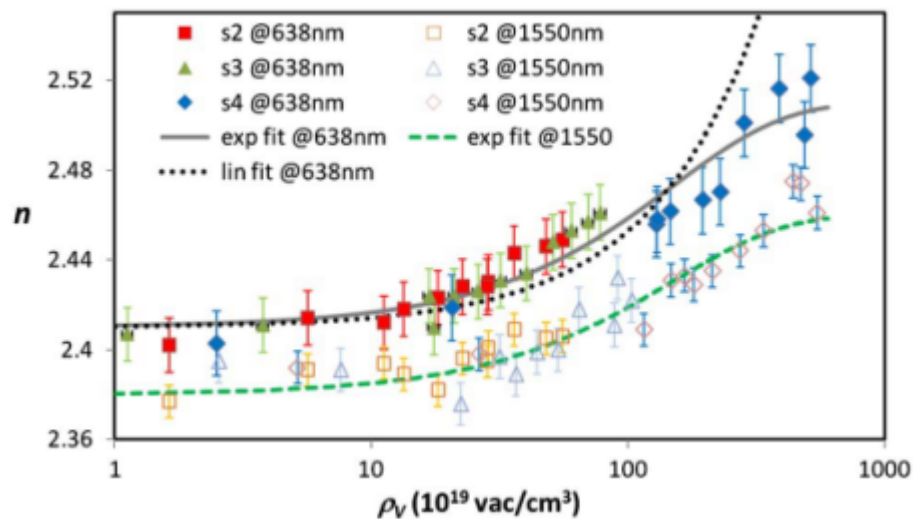
180 keV B implantation at increasing fluences ($F = 10^{13} - 5 \cdot 10^{14} \text{ cm}^{-2}$) over the whole sample surface

Woollam M2000-FI variable-angle spectroscopic ellipsometer (246 – 1690 nm)



Optical features

Ellipsometric characterization of shallow-ion-implanted diamond

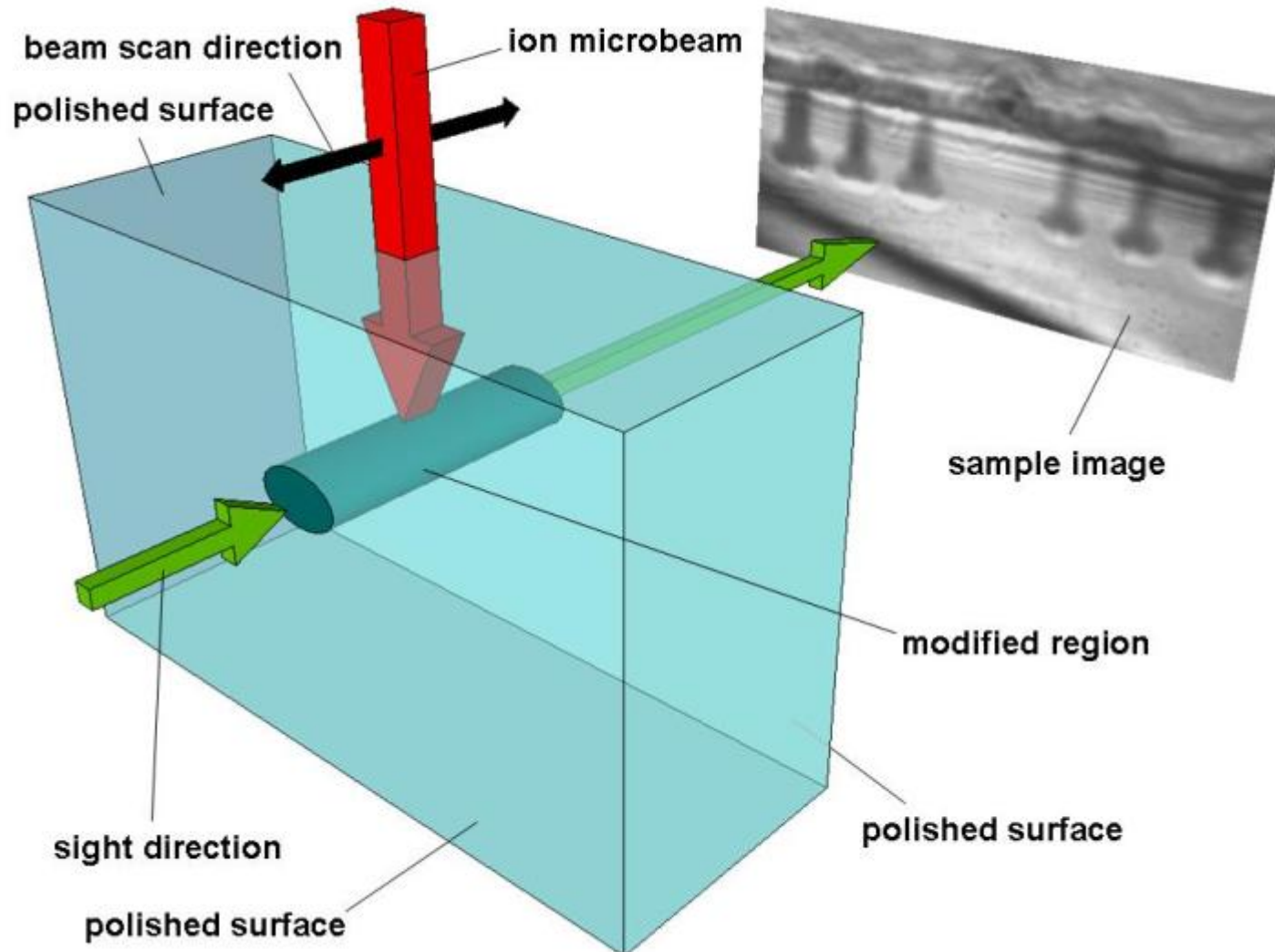


- **Linear** trend at low damage levels, **sub-linear** trend at higher fluences
- Similar changes are observed over the entire spectral range under investigation



Optical features

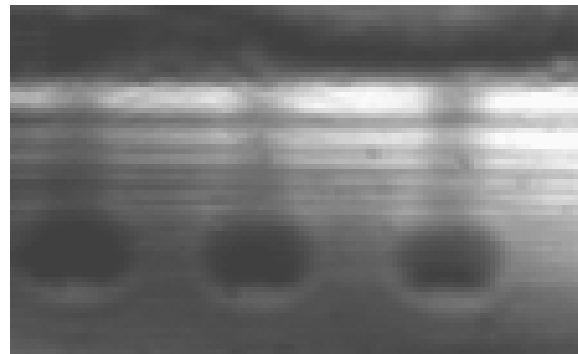
Direct writing of waveguiding structures with 5 MeV H microbeam



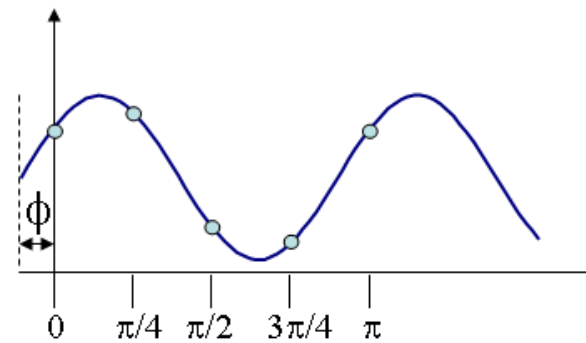
Optical features

Interferometric characterization

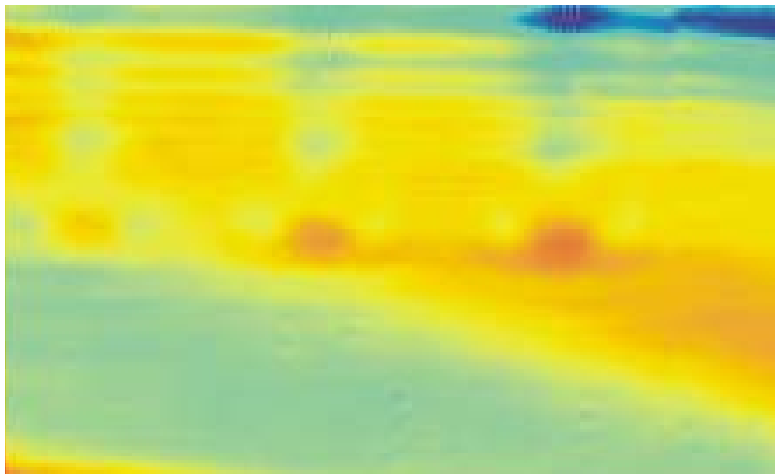
Interferometric image



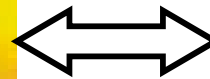
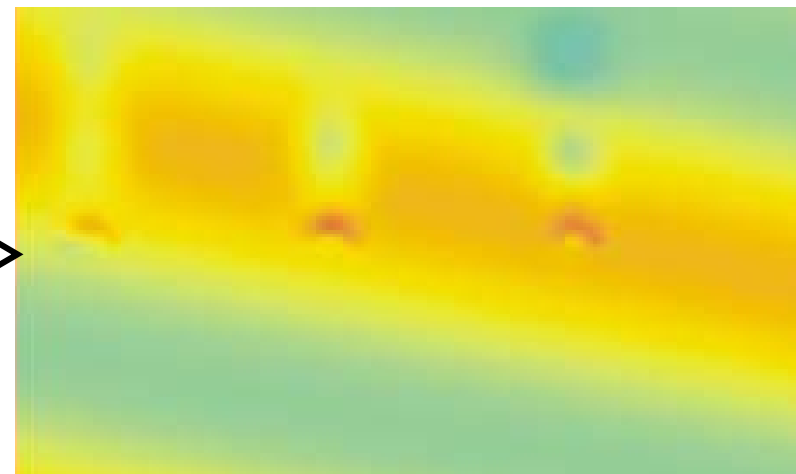
Phase shift reconstruction



Phase shift map



Finite element method



Optical features

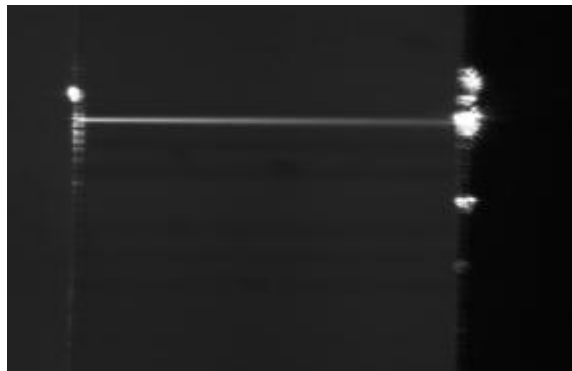
Laser-coupling characterization

$\lambda = 532 \text{ nm}$, optical objectives coupled at both the input and output points

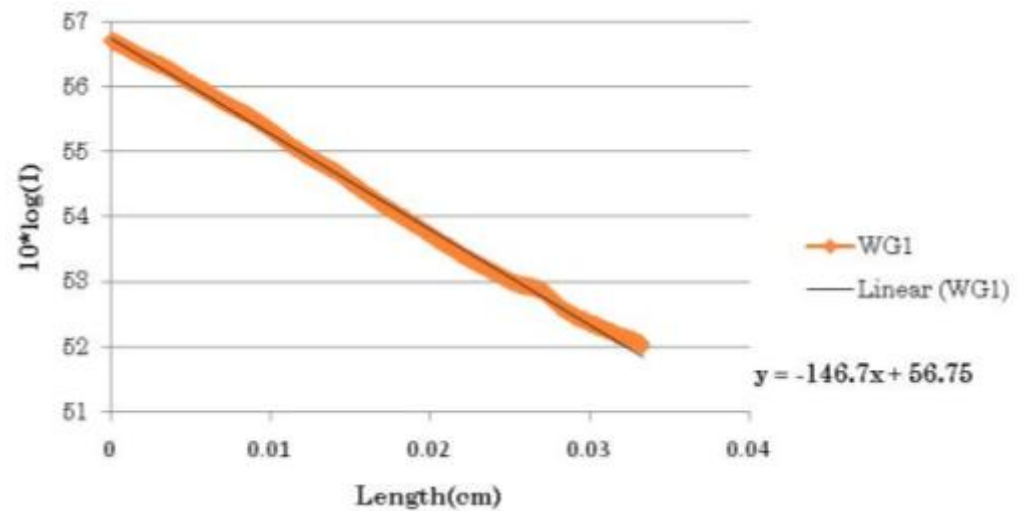
No waveguide coupling



Waveguide coupling



Strong absorption losses
($10^1 - 10^2 \text{ dB cm}^{-1}$)



CIBA-NUS



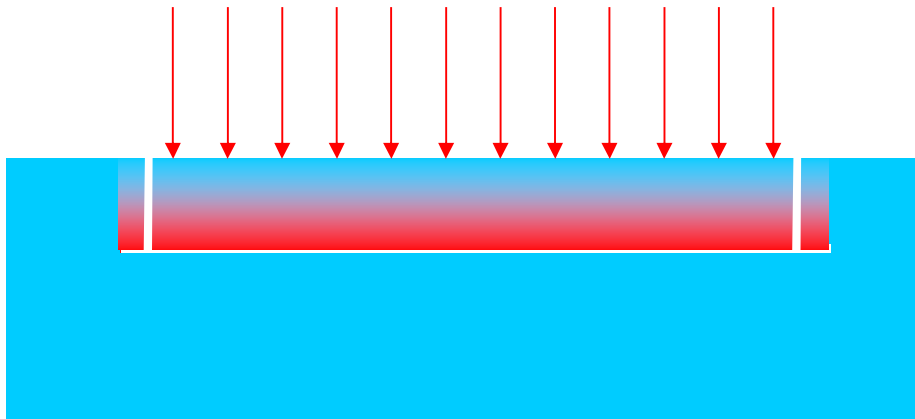
As-implanted samples: **annealing** can help reducing absorption losses while maintaining a suitable refractive-index contrast

Outline

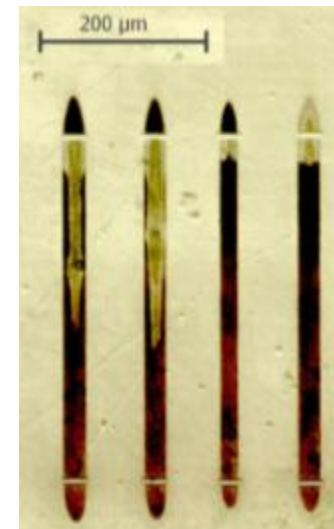
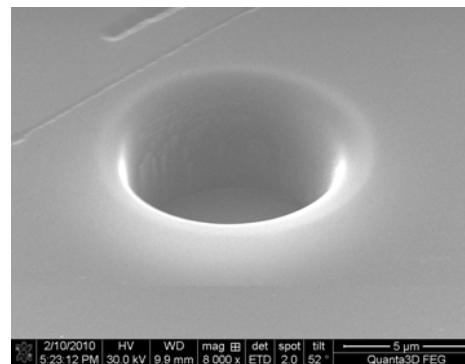
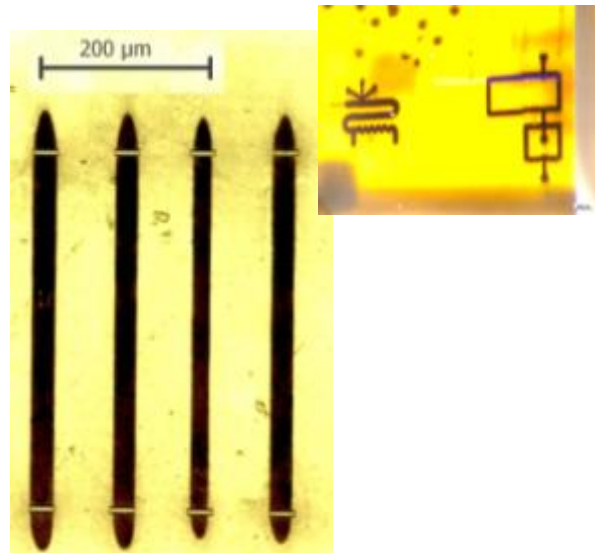
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Microfluidics

Fabrication of micro-fluidic channels



- ✓ direct ion microbeam writing of buried amorphized channels
- ✓ high temperature thermal annealing (1200 ÷ 1400 °C): graphitization
- ✓ FIB milling of access holes
- ✓ selective etching of graphite with an **electrochemical** process

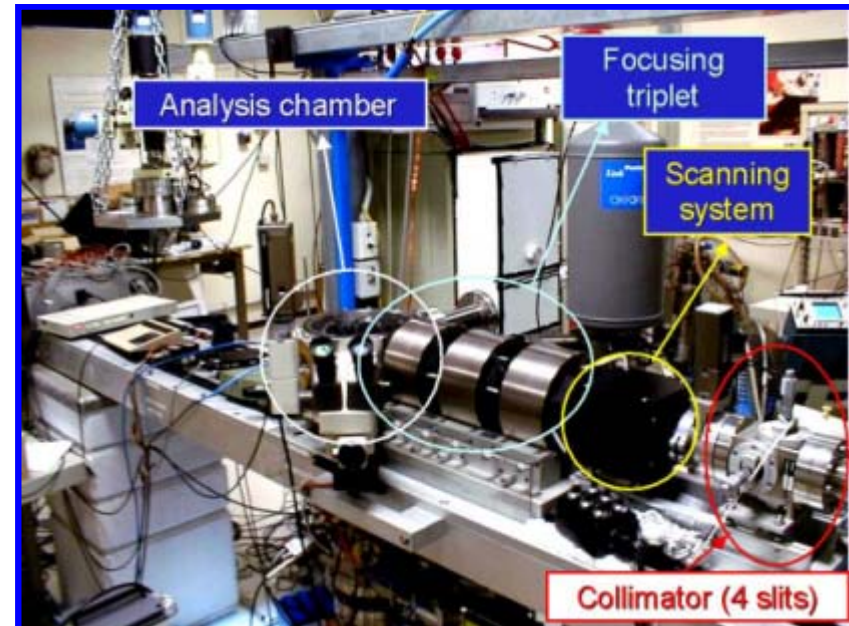


Microfluidics

Ion implantation performed at the
RBI-LIBI and **LNL-AN2000** ion microbeam lines



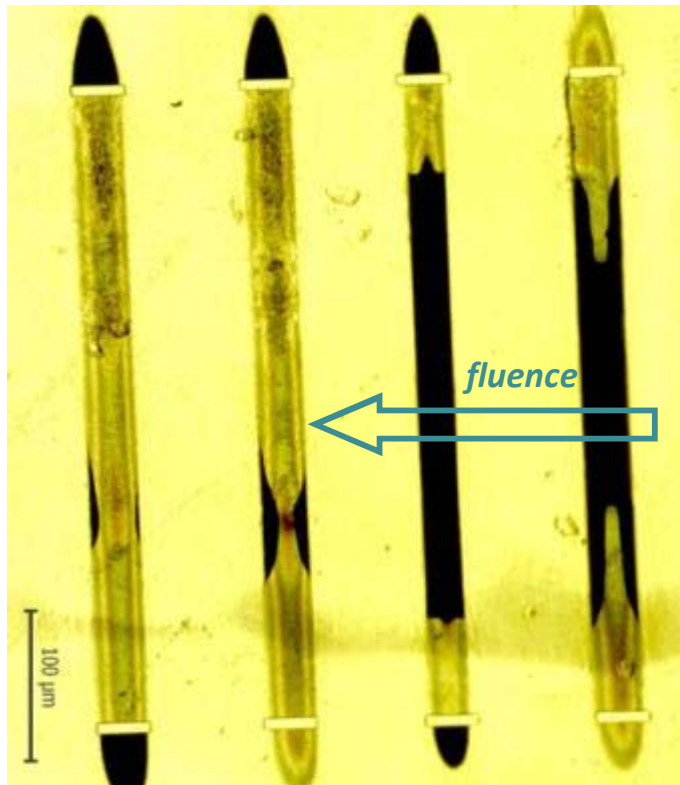
18 MeV C @ $F=2 \cdot 10^{16} \text{ cm}^{-2}$



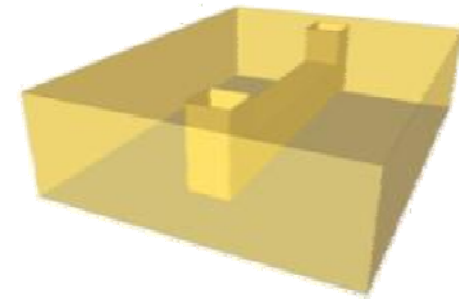
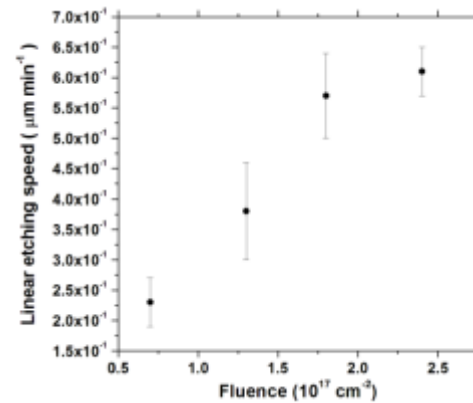
1.8 MeV He @ $F=1 \cdot 10^{17} \text{ cm}^{-2}$

Microfluidics

Fabrication of micro-fluidic channels

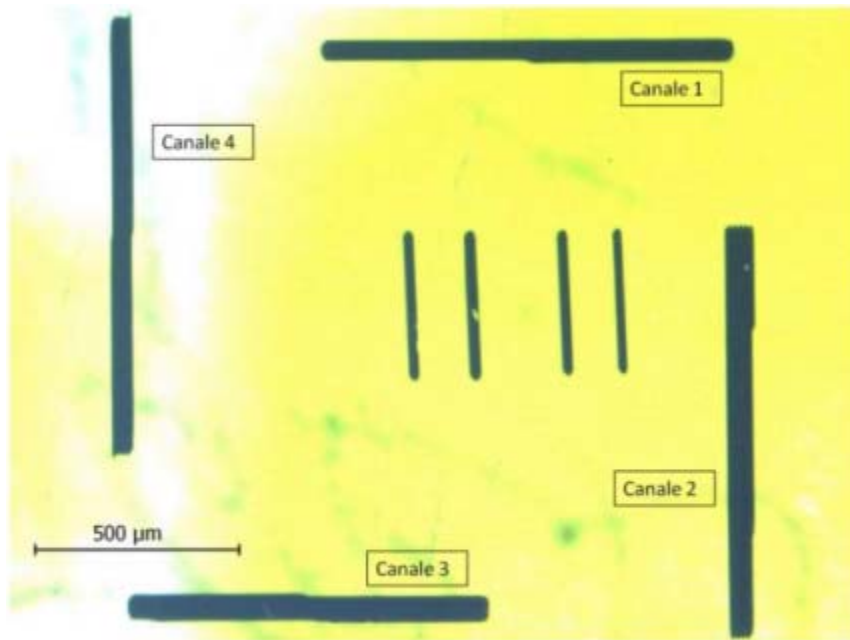


- ✓ complete graphite removal from channels implanted at the highest fluence
- ✓ different **etching rates** for channels implanted at different fluences
- ✓ possibility of creating **buried** microfluidic channels with a **monolithic** approach

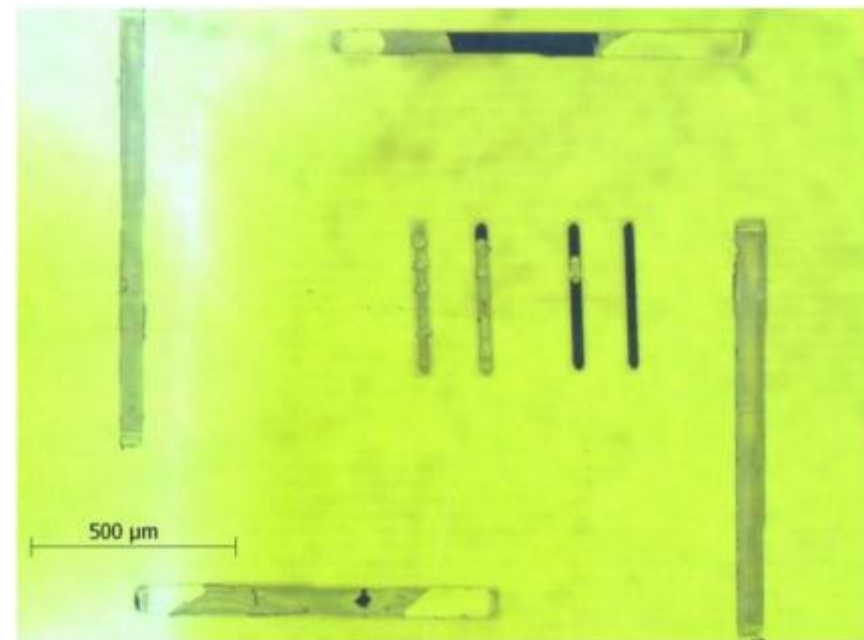


Microfluidics

mm-long microfluidic channels



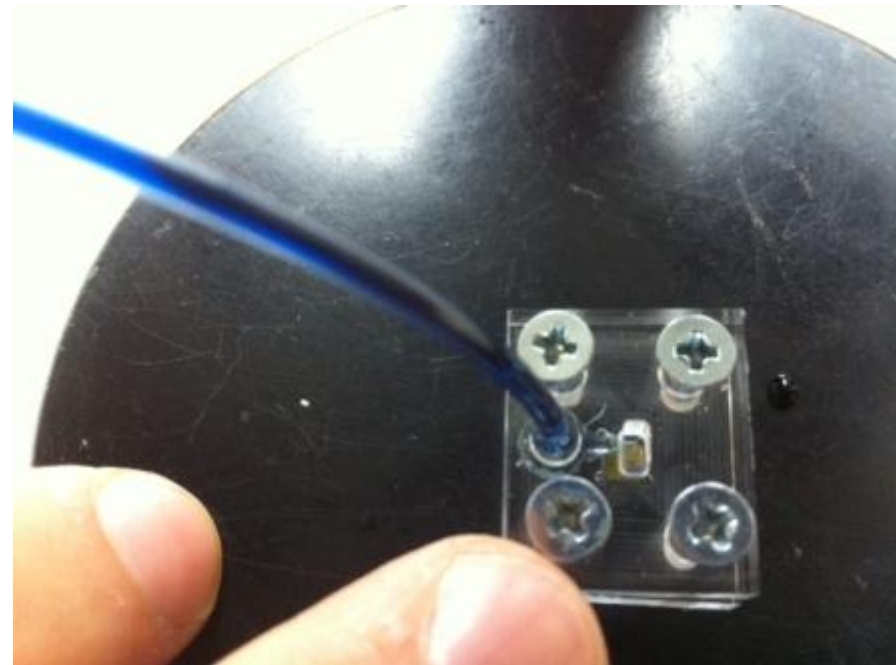
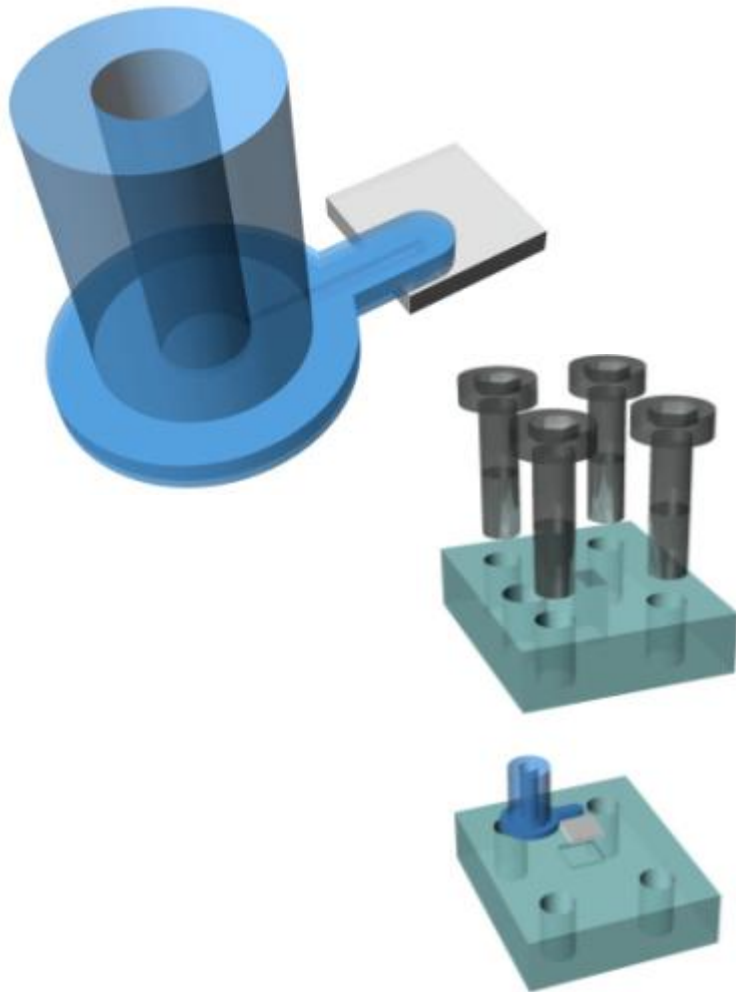
before etching



after etching

Microfluidics

liquid injection system:
mechanically clamped PDMS structure



Acknowledgments



Sample processing and characterization University of Torino

A. Battiato, F. Bosia, V. Carabelli, E. Carbone, J. Forneris, D. Gatto Monticone, A. Gilardino, A. Lo Giudice, S. Gosso, D. Lovisolo, A. Marcantoni, F. Picollo, A. Re, E. Vittone



FIB micromachining and optical characterization National Institute of Metrologic Research

G. Amato, L. Boarino, G. Brida, I. Degiovanni, E. Enrico, M. Genovese, P. Traina

MeV ion implantation & IBIC measurements

National Laboratories of Legnaro (INFN)

D. Ceccato, L. La Torre, V. Rigato



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S. Calusi, L. Giuntini, M. Massi

Ruđer Bošković Institute

V. Grilj, M. Jakšić, Ž. Pastuović, N. Skukan



RUBION Laboratory

S. Pezzagna, J. Meijer



FIB microfabrication, cross-sectional TEM MARC group, University of Melbourne

B. Fairchild, S. Praver, S. Rubanov

Optical / morphological characterization National Institute of Optics

R. Mercatelli, F. Quercioli, A. Sordini, S. Soria, M. Vannoni



Optical absorption characterization ENEA “La Casaccia”

A. Sytchkova

Optical modeling Department of Energetics, University of Florence

S. Lagomarsino, S. Sciortino



Waveguides characterization CIBA – National University of Singapore

A. Bettiol, V. S. Kumar



High-resolution X-ray diffraction Department of Physics, University of Padova

N. Argiolas, M. Bazzan



CVD diamond growth Department of Mechanical Engineering, University of Rome Tor Vergata

M. Marinelli, C. Verona, G. Verona-Rinati

Content sources

- www.srim.org
- www.wikipedia.org

- Australian National University
- School of Physics – University of Melbourne
- Solid State Physics Group – University of Torino
- University of Florida
- University of New South Wales
- Technion – Israel Institute of Technology