Investigation and Manipulation of Dislocations in Heteroepitaxial Diamond-on-Iridium Crystals

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Outline

- Diamond for Radiation and Particle Detectors
- Growth of Heteroepitaxial Diamond Films on Ir/YSZ/Si(001)
- Defects in Crystals
- Dislocations in CVD Diamond
- Detection of Dislocations
- Reduction of Dislocation Density
- Further investigations
Diamond for Radiation and Particle Detectors

- Influence of material quality on detector performance

  Local variation of sensitivity
  → Electronic activity of crystal lattice defects and impurities (recombination centres)

X-ray response of a homoepitaxial single crystal diamond detector

![Image of X-ray response of a homoepitaxial single crystal diamond detector](image1)

J. Morse, CARAT Workshop 2009

X-ray response of a polycrystalline diamond detector

![Image of X-ray response of a polycrystalline diamond detector](image2)

Fig. 3. Image of the X-ray sensitivity as measured on a 1 x 1 mm area in the corner of the electrical contact. Grey scale is logarithmic and given in nA, for a 2.2 x 10^6 pA s^-1 flux at 5.5 keV.

P. Bergonzo et al., DRM 11 (2002), 418
Diamond for Radiation and Particle Detectors

- Influence of material quality on detector performance

  Local variation of sensitivity

  → Electronic activity of crystal lattice defects and impurities (recombination centres)

X-ray response of a homoepitaxial single crystal diamond detector

→ Homogeneous response

X-ray response of a polycrystalline diamond detector

→ Inhomogeneous response

Fig. 3. Image of the X-ray sensitivity as measured on a 1 x 1 mm area in the corner of the electrical contact. Grey scale is logarithmic and given in nA, for a 2.2 x 10^6 ph s^-1 flux at 5.5 keV.

J. Morse, CARAT Workshop 2009

P. Bergonzo et al., DRM 11 (2002), 418
Diamond for Radiation and Particle Detectors

- **Influence of material quality on detector performance**

  Local variation of sensitivity
  → Electronic activity of crystal lattice defects and impurities (recombination centres)

X-ray response of a homoepitaxial single crystal diamond detector

X-ray response of a polycrystalline diamond detector

→ Possibilities for measuring and influencing the defect concentration?

J. Morse, CARAT Workshop 2009

P. Bergonzo et al., DRM 11 (2002), 418
Heteroepitaxial Diamond Films on Ir/YSZ/Si(001)

- **Method of growth**
  
  Microwave enhanced plasma chemical vapour deposition (MWPCVD)

Schematic and photograph of CVD reactor
Heteroepitaxial Diamond Films on Ir/YSZ/Si(001)

- **Substrate**
  
  Multi-layer system Ir/YSZ/Si(001) (YSZ = yttria-stabilised zirconia)
  → Good thermal compatibility of diamond and silicon
  → Monocrystalline growth of diamond on iridium
  → Cost-saving (Ir price!) due to thin film technology

![Schematic of diamond/Ir/YSZ/Si multi-layer system](image)

Thermally induced stress due to mismatched thermal expansion coefficients of diamond and substrate

![Graph showing thermal stress](image)
Heteroepitaxial Diamond Films on Ir/YSZ/Si(001)

- **Nucleation**
  
  Bias-Enhanced Nucleation (BEN)
  
  → Applicable to 4 inch Ir/YSZ/Si(001) wafers
  
  → Nucleation density up to $10^{11}$ cm$^{-2}$

Schematic of BEN set-up

4 inch substrates for BEN process
Heteroepitaxial Diamond Films on Ir/YSZ/Si(001)

- Growth

→ Formation of defect-rich monocrystalline diamond film
→ Improvement of crystalline quality with film thickness

Schematic: Transition to heteroepitaxial diamond single crystal

SEM images of Dia/Ir/SrTiO$_3$/Si(001)
Heteroepitaxial Diamond Films on Ir/YSZ/Si(001)

- **State of the art**
  - Large-area growth
  - Step by step towards 30 x 30 mm²

10 x 10 mm² flawless detector crystal

20 x 20 mm² neutron monochromator crystals ("poor" quality)

18 x 18 mm² attempt for detector crystal (medium quality, still many inclusions)
State of the art
Detector performance
→ Resolution of discrete mixed nuclide α particle energy spectrum

**MIXED NUCLIDE α - SOURCE**

CCE\(_{\text{hole-drift}} \approx 93\%\)

δE/E ≈ 1.5 %

E. Berdermann, CARAT-Workshop 2011
Defects in Crystals

Point defects (0-dimensional)  
e.g. vacancies and interstitials

Line defects (1-dimensional)  
e.g. dislocations

Planar defects (2-dimensional)  
e.g. grain boundaries

Bulk defects (3-dimensional)  
e.g. voids (= clusters of vacancies)

C. Kittel: Einführung in die Festkörperphysik, 14th ed. (2006)

http://www.spaceflight.esa.int/impress/text/education/Glossary/Glossary_G.html
Dislocations in CVD Diamond

- **Basics**
  
  Fundamental dislocation types

  **Edge dislocation**

  **Screw dislocation**

  Insertion of additional incomplete lattice plane

Dislocations in CVD Diamond

- **Basics**

  Classification by Burgers vector $\mathbf{b}$ and dislocation line direction $\mathbf{l}$

  Determination of Burgers vector $\mathbf{b}$ of an edge dislocation

  Dislocation line $\mathbf{l}$ (blue) of an edge dislocation

  http://de.wikipedia.org/wiki/Versetzung_(Materialwissenschaft)


  Edge dislocation: $\mathbf{b} \perp \mathbf{l}$
  Screw dislocation: $\mathbf{b} \parallel \mathbf{l}$
  Mixed dislocation: else
Dislocations in CVD Diamond

- Simplest types of dislocations in diamond

Perfect dislocations of the \{111\}\langle110\rangle slip system

- Screw dislocation, e.g. \( \mathbf{l}_s = [1 -1 0], \mathbf{b}_s = \frac{1}{2} [1 -1 0] \)
- \(60^\circ\) dislocation, e.g. \( \mathbf{l}_{60} = [1 -1 0], \mathbf{b}_{60} = \frac{1}{2} [0 -1 1] \)
- Edge dislocation, e.g. \( \mathbf{l}_e = [-1 -1 2], \mathbf{b}_e = \frac{1}{2} [-1 1 0] \)

This is only the tip of the iceberg!

A. Blumenau et al., Defects and Diffusion Forum 226-228 (2004) 11
Dislocations in CVD Diamond

- **Threading dislocations**

  Consequence of growth mechanism of heteroepitaxial diamond films
  → Generation of defect bands composed of small angle grain boundaries

Plan view TEM images of thin heteroepitaxial CVD diamond films

→ Dislocation lines penetrating the whole diamond film nearly parallel to the growth direction

→ Initially very high dislocation density, comparable to nucleation density (10^{11} \text{ cm}^{-2}), decreasing with film thickness

C. Kittel: Einführung in die Festkörperphysik, 14th ed. (2006)
Detection of Dislocations

- Common methods

**X-ray topography**
- M. Gaukroger et al., DRM 17 (2008) 262

**TEM**
- A. Blumenau et al., Defects and Diffusion, Forum 226-228 (2004) 11

Dislocation density (cm\(^{-2}\))

<table>
<thead>
<tr>
<th>Density (cm(^{-2}))</th>
<th>0</th>
<th>(10^5)</th>
<th>(10^{10})</th>
<th>(10^{13})</th>
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</thead>
<tbody>
<tr>
<td>X-ray topography</td>
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<td>TEM</td>
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<tr>
<td>Etching</td>
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</tbody>
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Values taken from K. Sangwal: Etching of Crystals (1987)
Detection of Dislocations

- **Etching**
  
  Plasma etching of diamond
  
  → Similar to CVD growth process
  
  → Addition of $\text{CO}_2$ instead of $\text{CH}_4$ in the gas phase
  
  → Formation of **etch-pits** at intersection of surface and dislocation line

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**Advantages:**

→ Compatible with existing CVD reactors
→ Fast
→ Suitable for wide range of dislocation densities

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J. Achard et al., PSS(A) 206 (2009) 1949
Detection of Dislocations

- Experimental observations on DoI films
  
  Etch pit distribution on “low-quality” crystals for neutron monochromators

[Image of a dark-field optical microscope image showing preferential orientation of dislocation bands]
Detection of Dislocations

- Experimental observations on DOI films

Etch pit distribution on “low-quality” crystals for neutron monochromators

SEM image
Detection of Dislocations

- Experimental observations on DoI films

Etch pit distribution on “low-quality” crystals for neutron monochromators

Bi-modal etch-pit distribution:
- Agglomerated dislocation bands from former grain boundaries
- Dispersed dislocations inside former grains

Extracted etch-pit positions
Detection of Dislocations

- Experimental observations on DoI films
  
  Photoluminescence pattern on “high-quality” crystal for detectors
  → **Blue luminescence correlated with dislocations**

![DiamondView image (UV excitation), courtesy of J. Achard](image)
Detection of Dislocations

- Experimental observations on DOI films
  
  Photoluminescence pattern on “high-quality” crystal for detectors
  → **Blue luminescence correlated with dislocations**

Luminescence bands automatically extracted by Watershed Algorithm
→ characteristic **length scale ~ 35 µm**
Detection of Dislocations

- Experimental observations on DOI films
  Unexpected similarity between “low-quality” monochromator crystals and “high-quality” detector crystals!

Further investigation necessary
  → Cathodoluminescence imaging (information depth!)
  → Thickness dependent etch-pit distribution
Reduction of Dislocation Density

- Growth of thick layers

Dislocation density vs. film thickness for different heteroepitaxial material systems

→ Common 1/d dependence observed for other material systems

A. Romanov et al., PSS(B) 198 (1996) 599
Reduction of Dislocation Density

- Growth of thick layers

  Descriptive explanation:
  Interaction of inclined dislocations during growth
  → Annihilation of dislocations with opposite Burgers vectors

→ Reduction of dislocation density by growth of thick layers possible
Reduction of Dislocation Density

- Growth of thick layers

  Descriptive explanation:
  Interaction of inclined dislocations during growth
  → Annihilation of dislocations with opposite Burgers vectors

→ Reduction of dislocation density by growth of thick layers possible

→ Mathematical modelling yields experimental 1/d dependence
Reduction of Dislocation Density

- Experimental observations on DoI films
  
  Etch pit density on films of different thickness

$$\propto d^{-0.6}$$

→ Behaviour closer to $\frac{1}{\sqrt{d}}$ than $\frac{1}{d}$
Reduction of Dislocation Density

- Experimental observations on DOI films

Etch pit density on films of different thickness

$\propto d^{-0.5}$

$\propto d^{-1}$

→ Two distinct regions with different behaviours?
Reduction of Dislocation Density

- Dislocation density spectrum of diamond materials

Y. Shvyd'ko et al., Nature Photonics 5, 539 (2011)

Dislocation density (cm$^{-2}$) 10$^4$ 10$^6$ 10$^8$ 10$^9$ 10$^{11}$

Further investigations

- TEM measurements
  → Dislocation type
  → Dislocation line direction

- Creating larger etch-pit data set
  → Spatial etch-pit distribution at different growth stages
  → Dislocation density reduction and modelling of thickness dependence

- Correlation of etch-pits and Raman/luminescence measurements

- Investigation of etch-pit morphology

- Correlation with detector performance

Etch-pits of different size and shape
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