1st CARAT Workshop @ GSI Darmstadt 2009 13-15 December 2009

Heteroepitaxial diamond on Ir/YSZ/Si(001): general developments and specific aspects for detector applications

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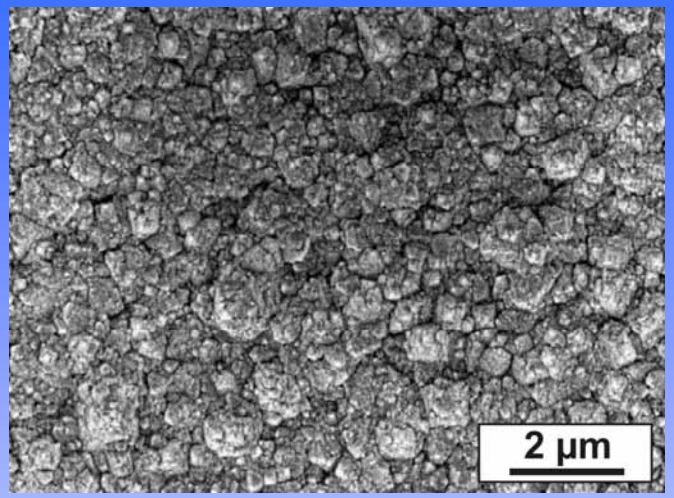
OUTLINE

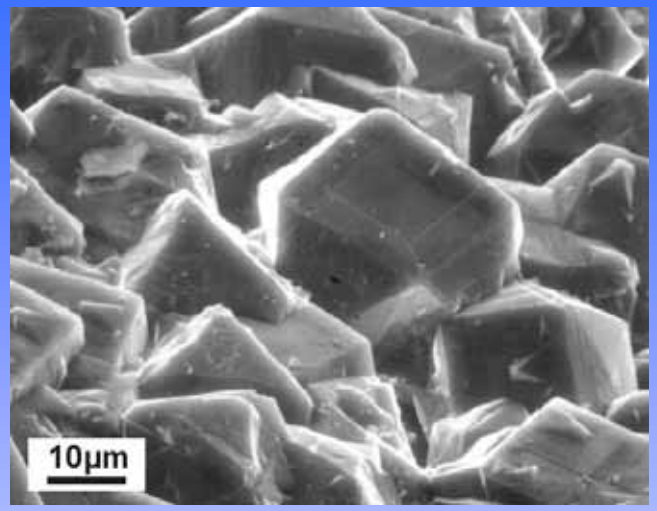
- Competing concepts for growth of diamond with low defect density
- Heteroepitaxy of diamond on iridium: a brief introduction
- Potential fields of applications for heteroepitaxial diamond
- Diamond films on Iridium for CARAT: specific aspects for detectors
- Outlook on future work

TEXTURED / EPITAXIAL DIAMOND FILMS

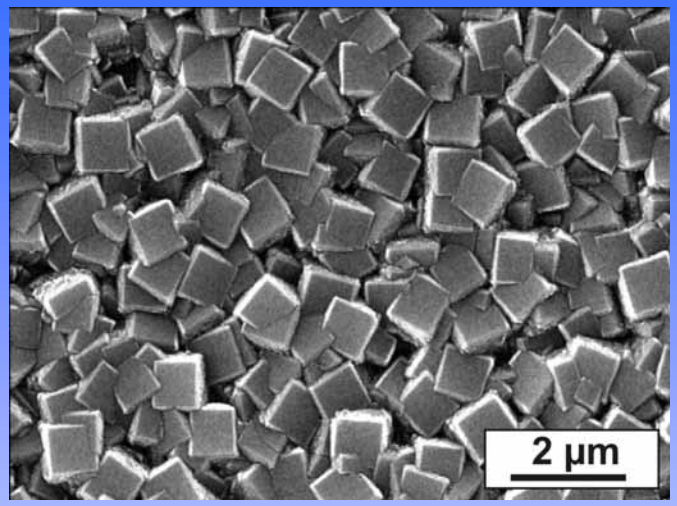
nanocrystalline

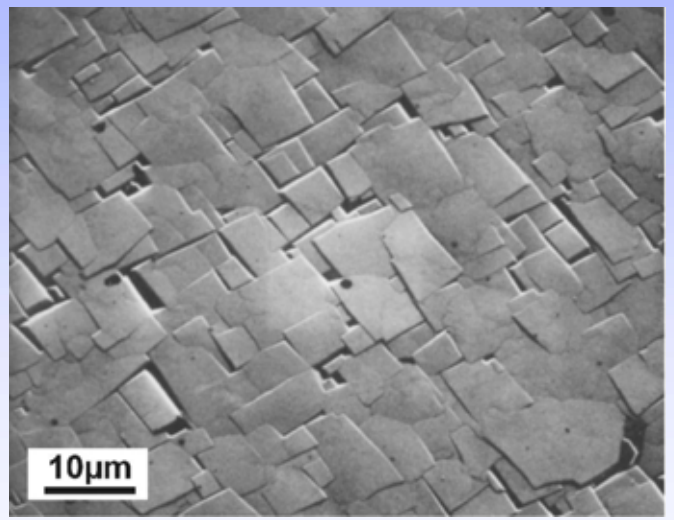


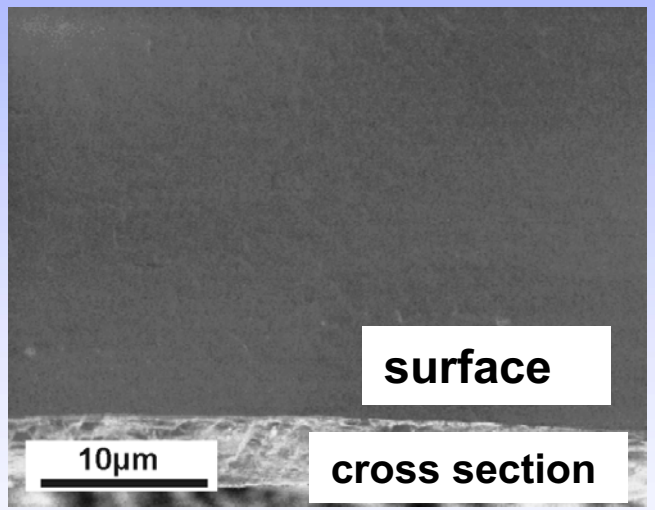
microcryst. (110)-fiber text.

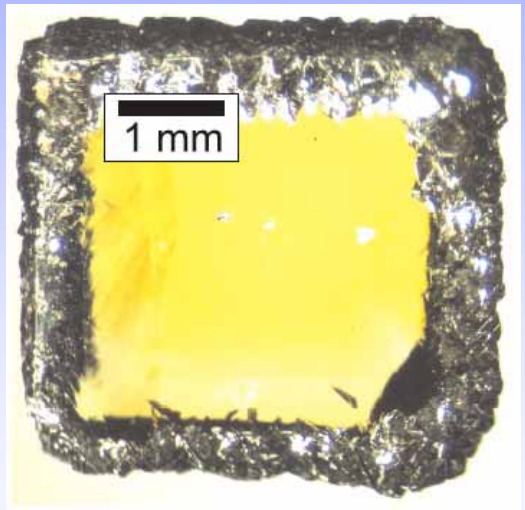


(100)-fiber texture







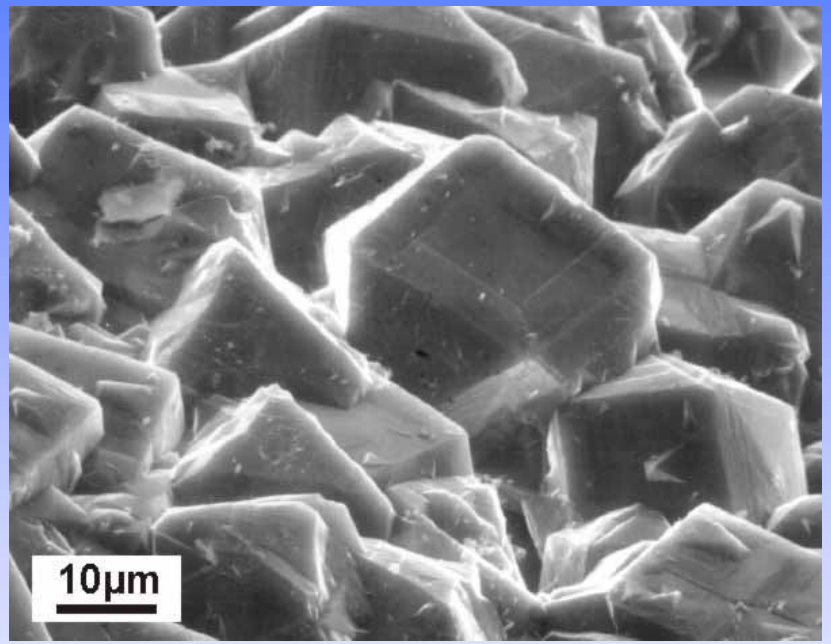


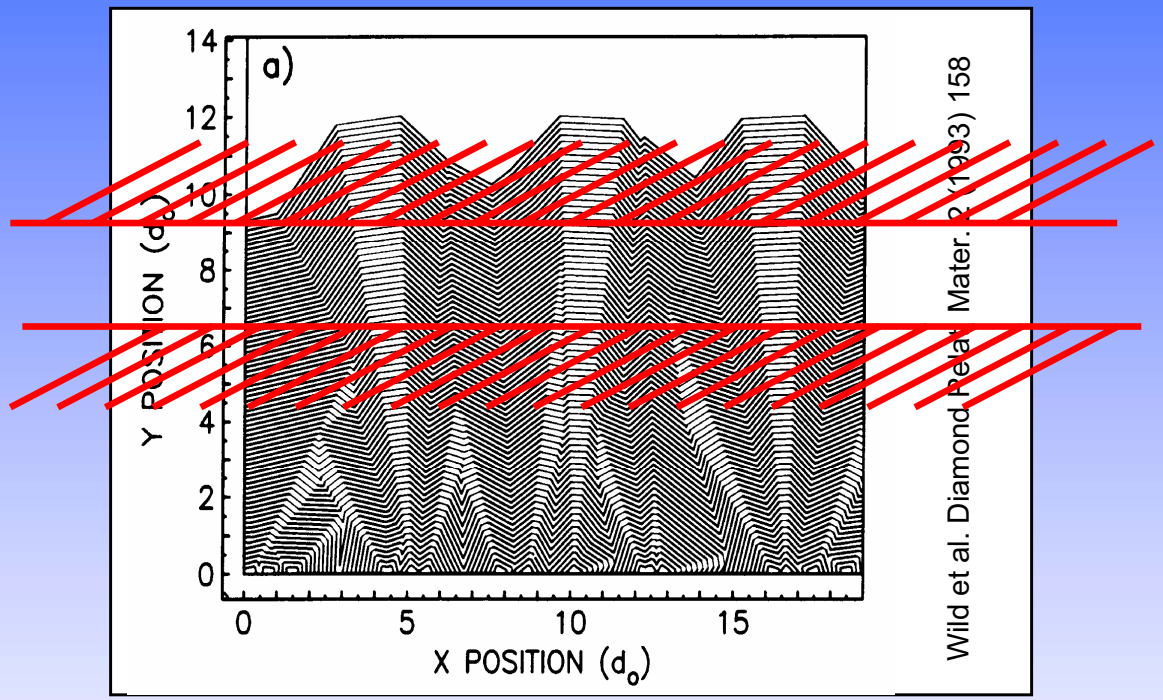
heteroepitaxial Dia/Si(001)

heteroepit.: Dia/Ir/$SrTiO_3$(001)

homoepitaxy on Ib HPHT

COARSE GRAIN POLYCRYSTALLINE FILMS

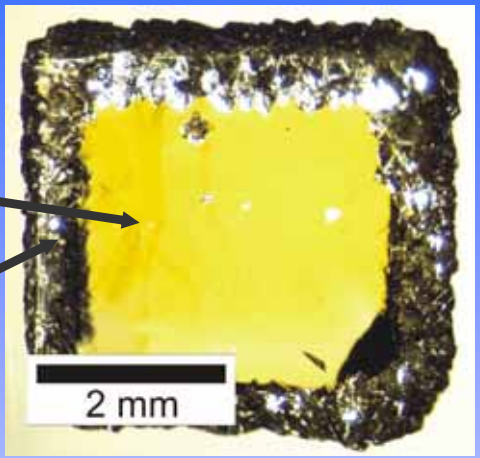




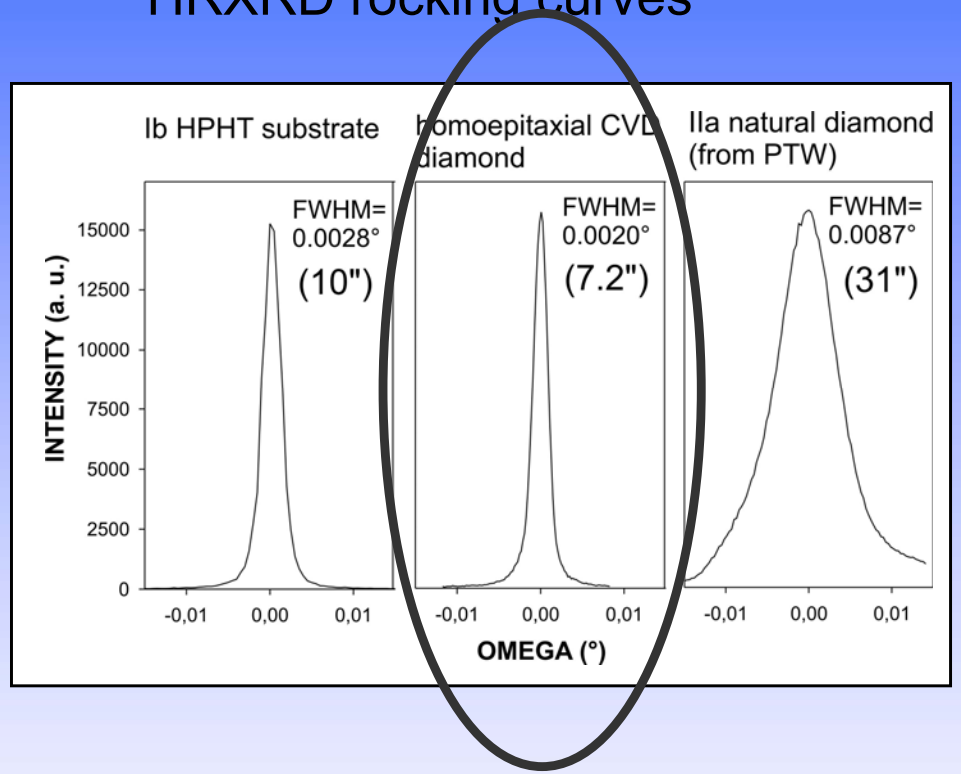
HOMOEPITAXY

Yellow colour from subjacent substrate

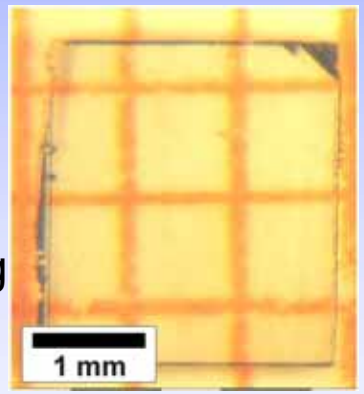
Polycryst-alline rim



HRXRD rocking curves

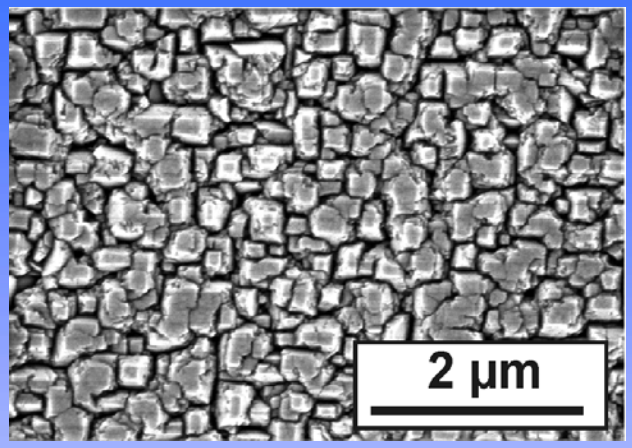


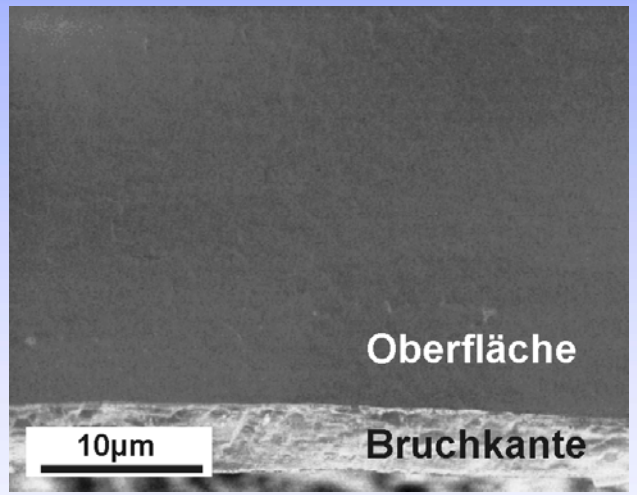
Free-standing CVD layer after laser cutting and grinding / polishing



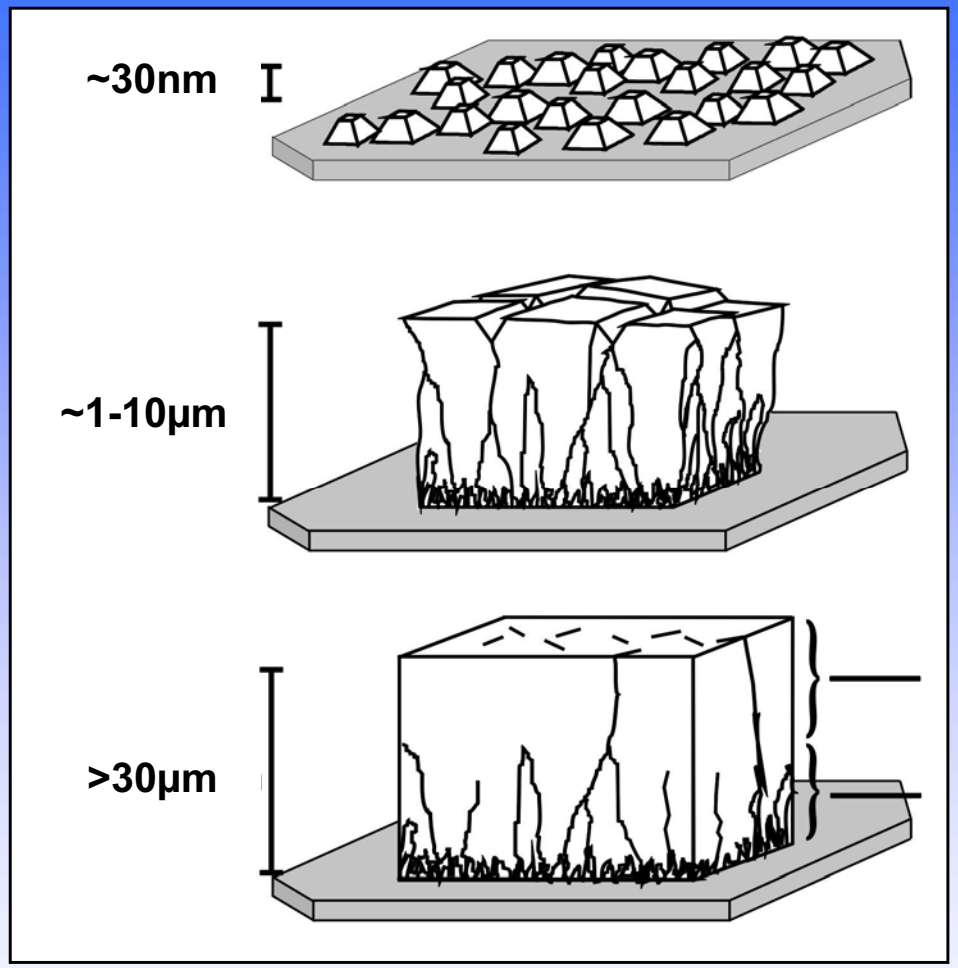
Homoepitaxial diamond with electronic / detector grade quality has been shown by other groups → achievable size??

SINGLE CRYSTAL DIAMOND FILMS BY HETEROEPITAXY





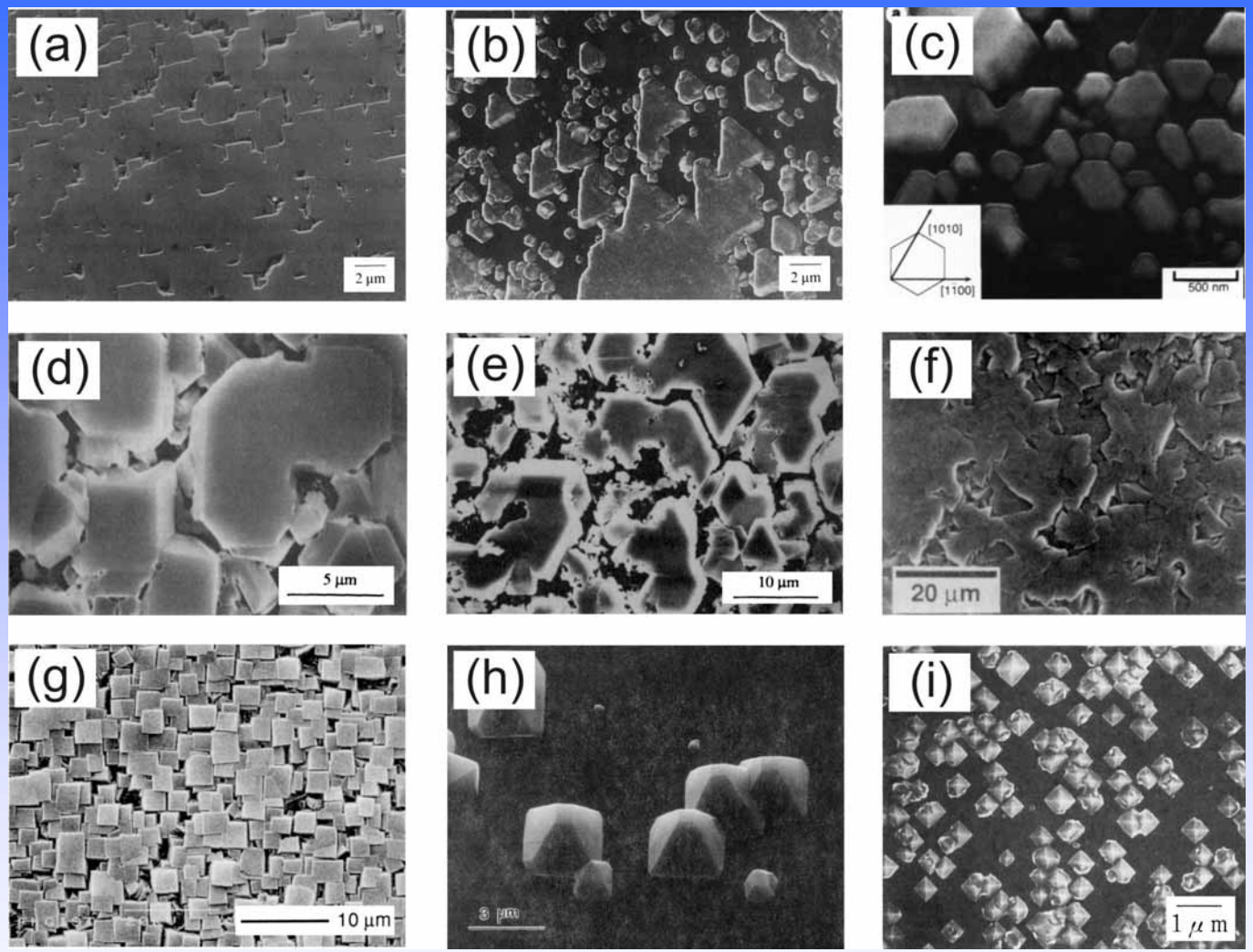
Dia/Ir/$SrTiO_3$(001)



Schema: Transition to a single crystal film in heteroepitaxy

A brief introduction to diamond heteroepitaxy

HETEROEPITAXY OF DIAMOND: SEARCHING FOR THE IDEAL SUBSTRATE MATERIAL



(a) c-BN(001)

(b) c-BN(111)

(c) Al_2O_3(0001)

(d) Ni(001)

(e) Ni(111)

(f) Pt(111)

(g) Si(001)

(h) ß-SiC(001)

(i) Ir(001)

Nucleation procedure: (a)-(c): no specific treatment (d)-(f): seeding with carbon / diamond powder (g)-(i) bias enhanced nucleation (BEN)

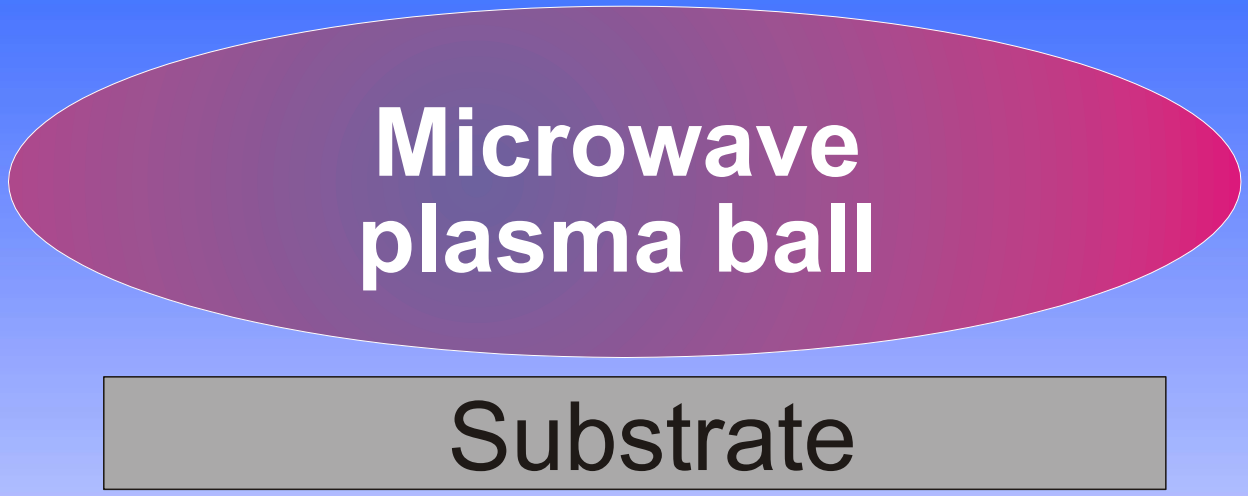
HETEROEPITAXY OF DIAMOND: SEARCHING FOR THE IDEAL SUBSTRATE MATERIAL

Substrate	First Publication	Current state of the art
Diamond on β-SiC:	Stoner & Glass 1992	Tilt 0.6° Twist: ~2.5°
Diamond on silicon:	Jiang & Klages 1992	Tilt ~ 1° Twist ~4°
Diamond on Ir:	Ohtsuka, Suzuki, Sawabe, Inuzuka 1996	Tilt, Twist: ~ 0.1 - 0.3°

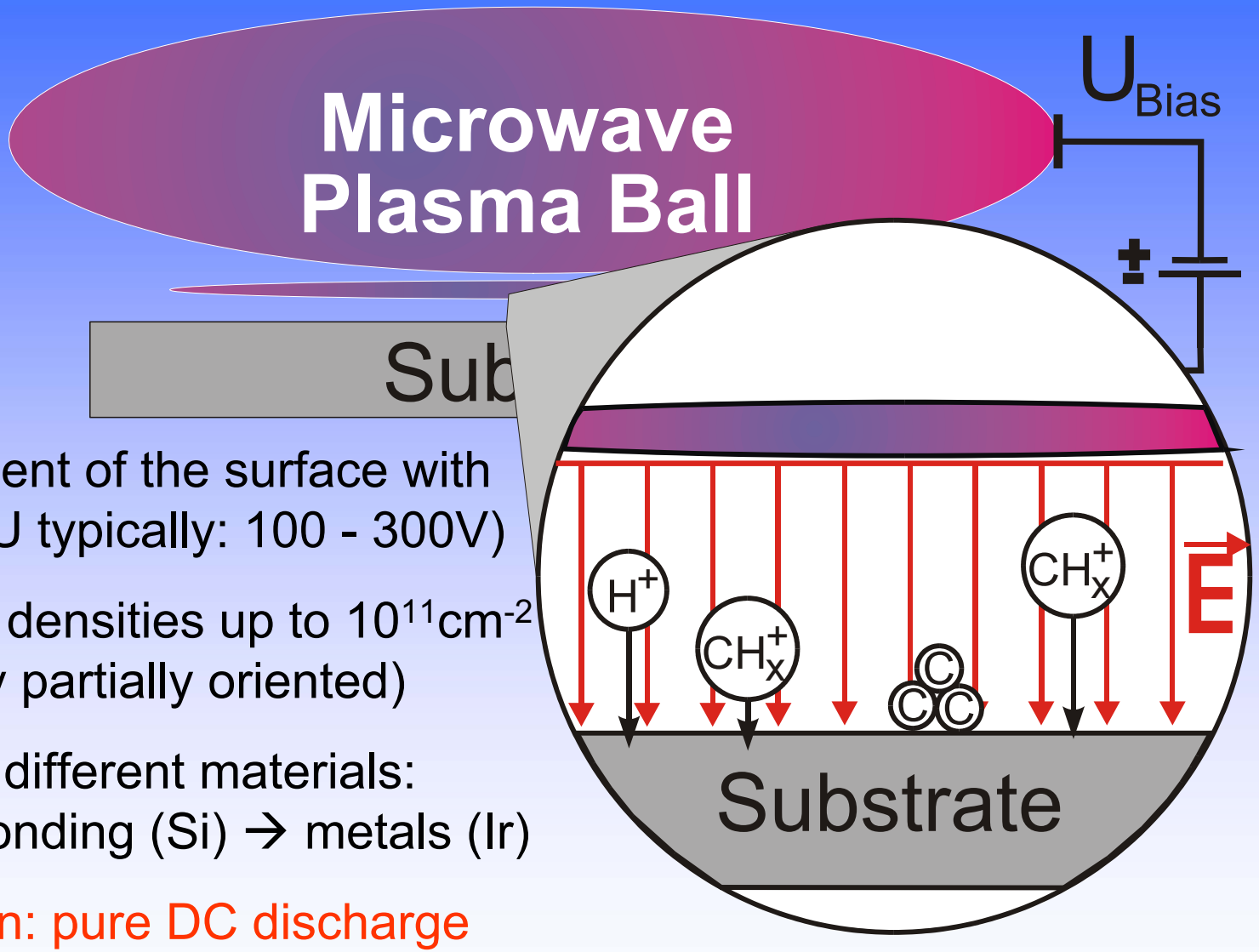
Further materials: c-BN, Cu, Ni, Co, TiC, Ni_3Si, Ni_3Ge, Al_2O_3

Iridium, the very best candidate for diamond heteroepitaxy

BIAS ENHANCED NUCLEATION (BEN)



BIAS ENHANCED NUCLEATION (BEN)



Bombardment of the surface with pos. ions (U typically: 100 - 300V)

Nucleation densities up to $10^{11}cm^{-2}$ (on Si, only partially oriented)

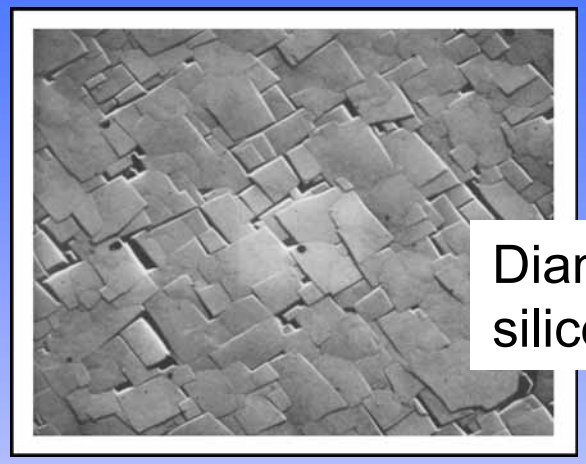
Epitaxy on different materials: covalent bonding (Si) → metals (Ir)

Modification: pure DC discharge

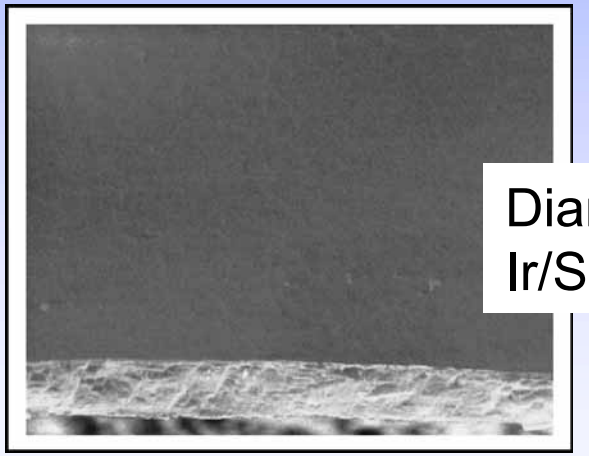
COMPARISON:
DIAMOND ON Si⇔DIAMOND ON Ir/$SrTiO_3$

The film surface

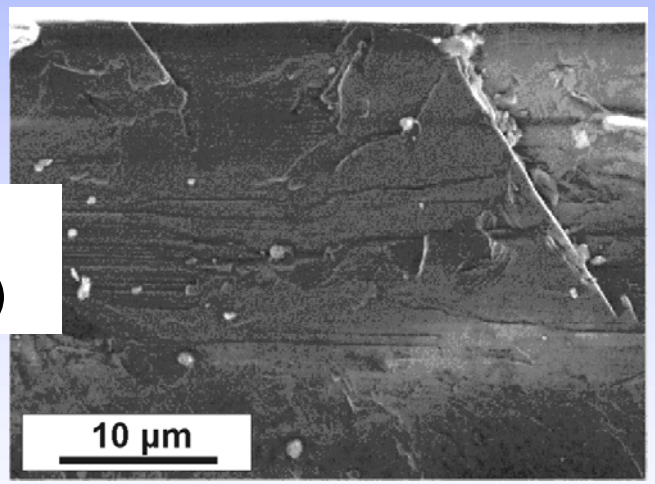
The cross section



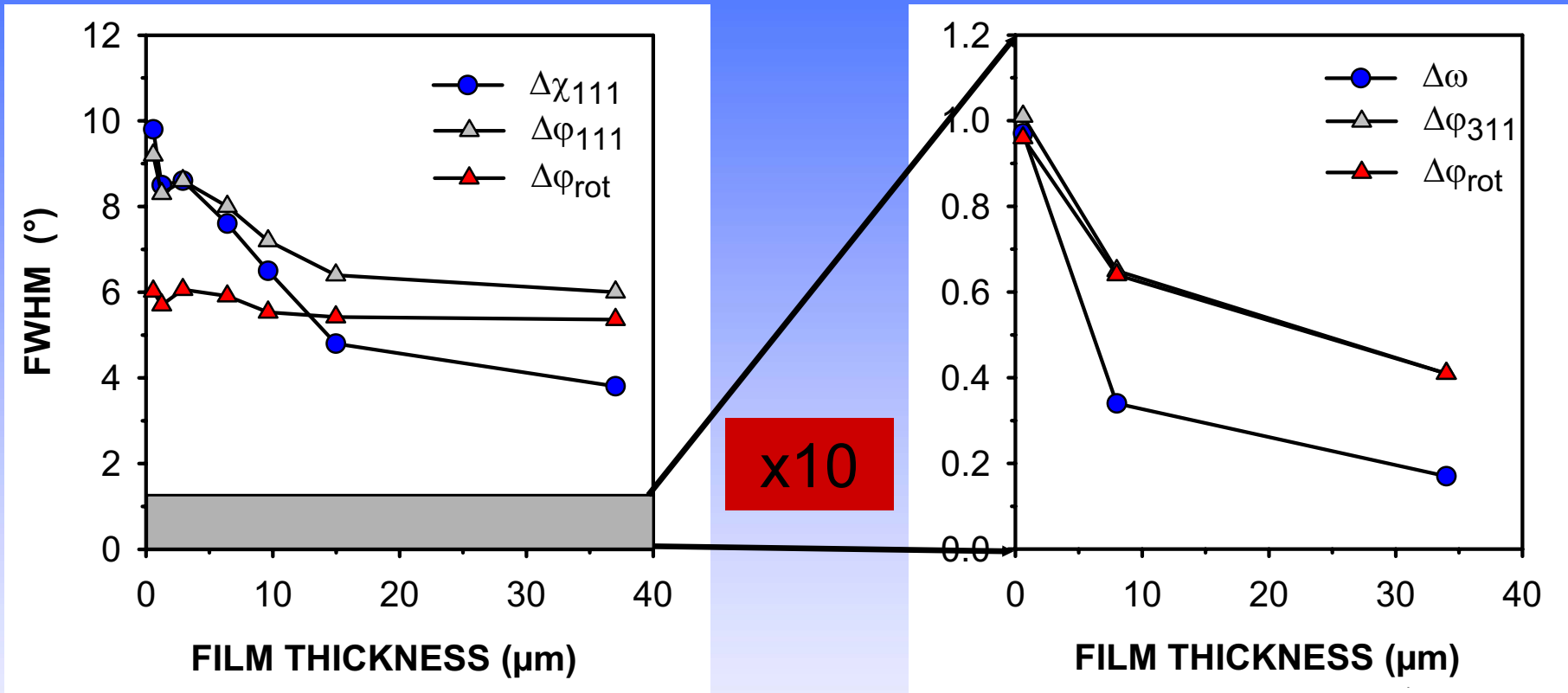
Diamond on silicon



Diamond on Ir/$SrTiO_3$(001)



DIFFERENCES IN THE TEXTURE DEVELOPMENT: DIAMOND ON Si ⇔ DIAMOND ON $Ir/SrTiO_3$



- One order of magnitude lower mosaicity
- Tilt and Twist decrease with thickness !!!!

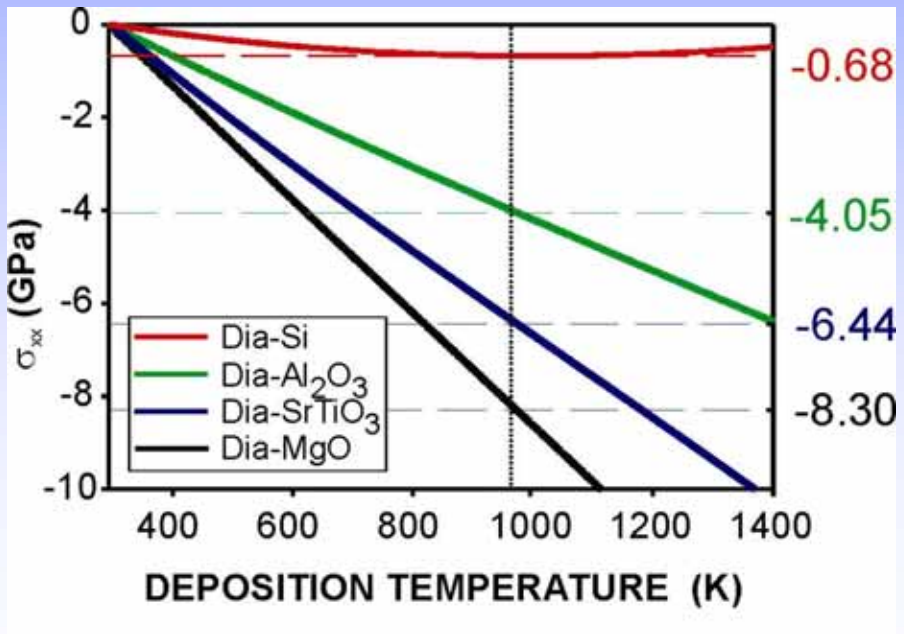
The technological challenge: finding an appropriate multilayer system

OXIDE SINGLE CRYSTALS vs. BUFFER LAYERS ON SILICON

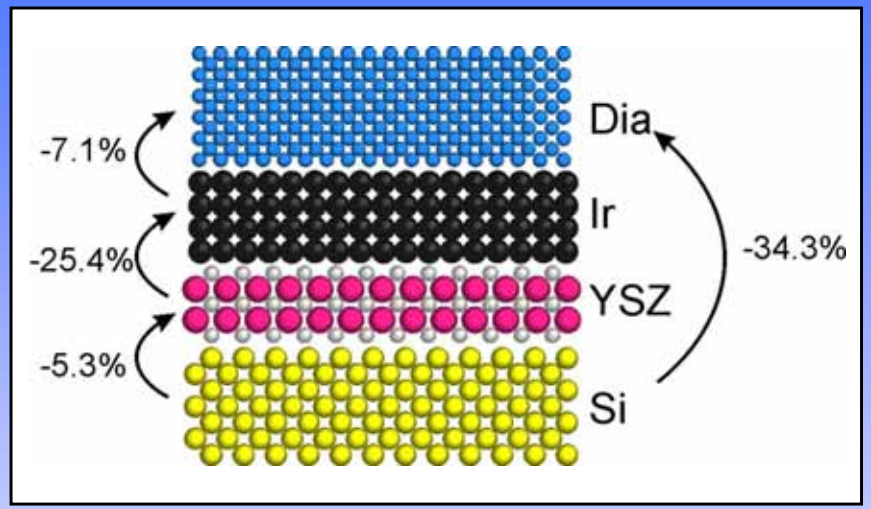
Requirements:

a) Growth of single crystal iridium films

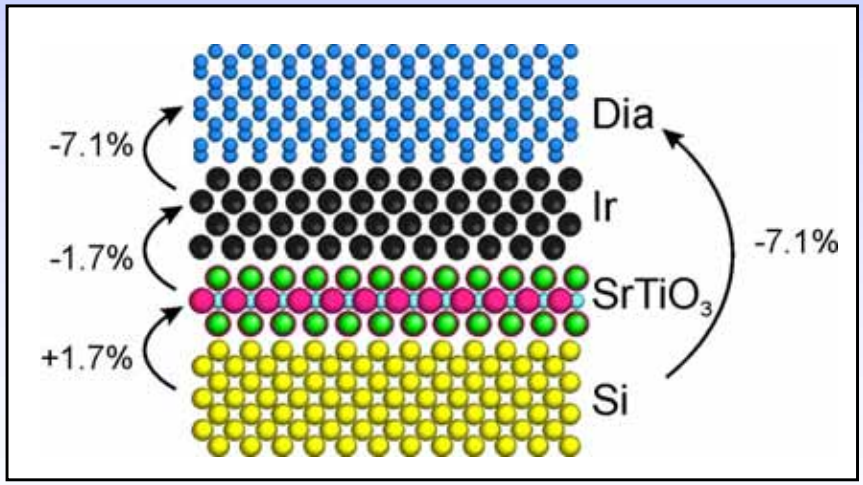
b) Thermal compatible with diamond



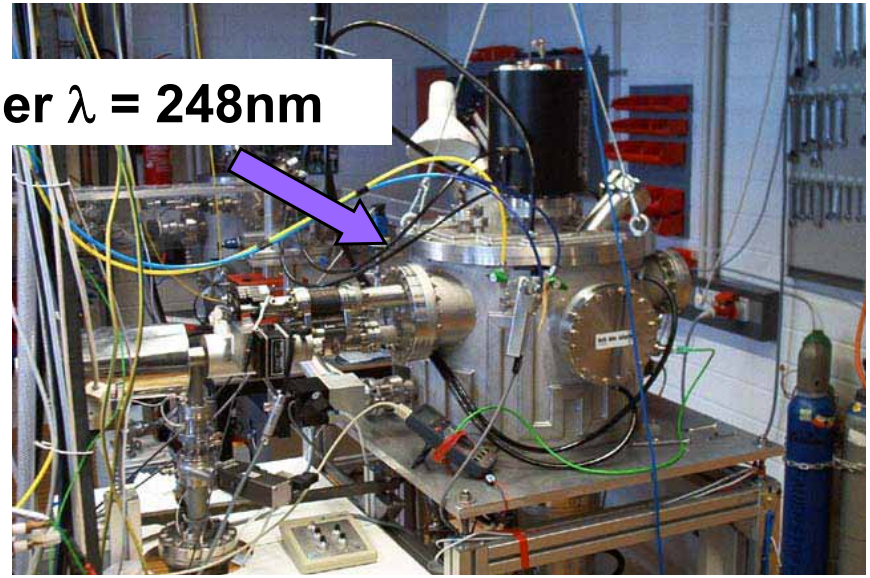
Dia/Ir/YSZ/Si(001)



Dia/Ir/$SrTiO_3$/Si(001)



GROWTH OF YSZ BY PULSED LASER DEPOSITION



PLD setup

KrF Excimer Laser

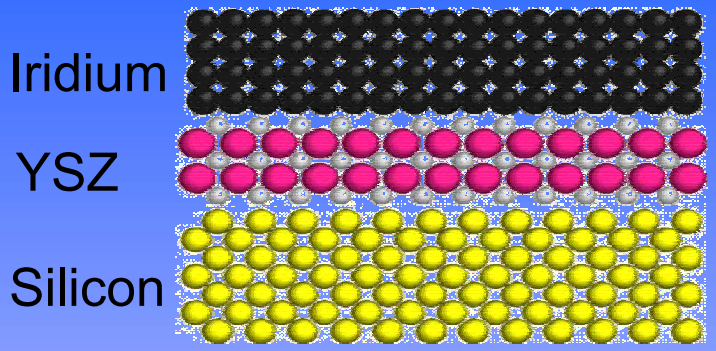
25 ns / 850 mJ

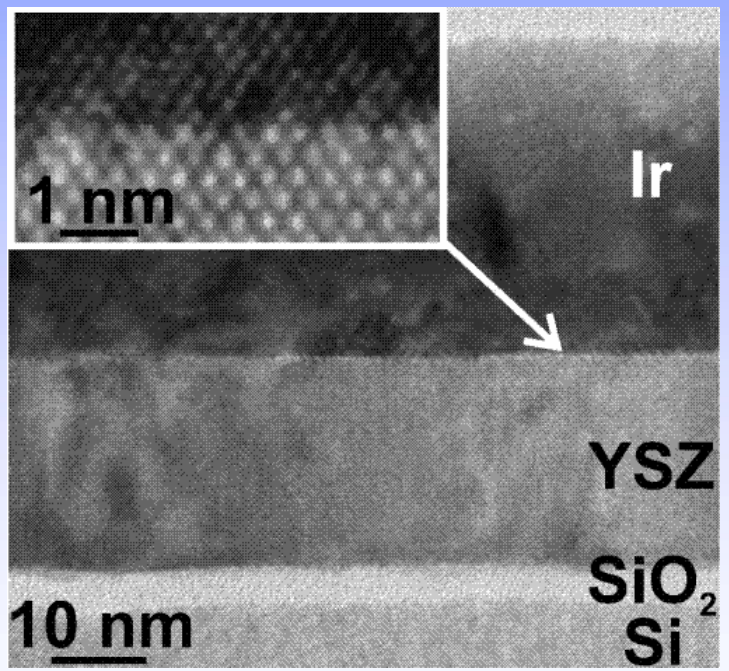
Ablation targets

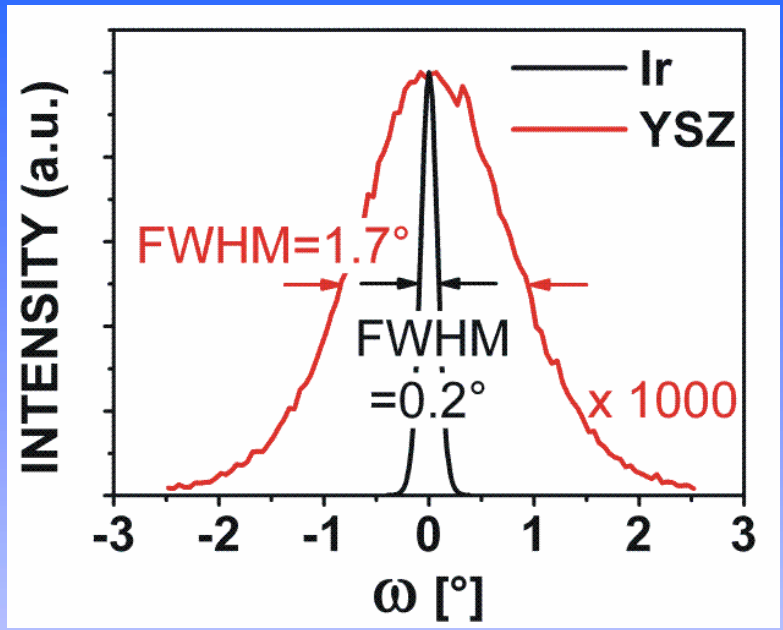
Yttria stabilized zirconia (YSZ)

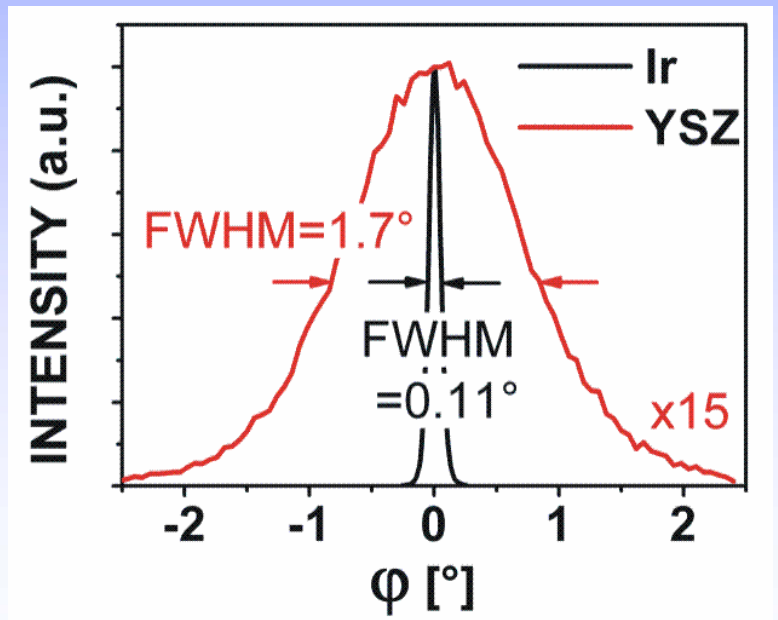
- No removal of the SiO_2 required
- Ablation target: ZrO_2 stabilized by Y_2O_3
- 5×10^{-2} Pa O_2 (first 600 pulses without O_2)
- Substrate temperature: 825°C
- Thickness: 20 - 40 nm

Ir/YSZ/Si(001): STRUCTURE & TEXTURE

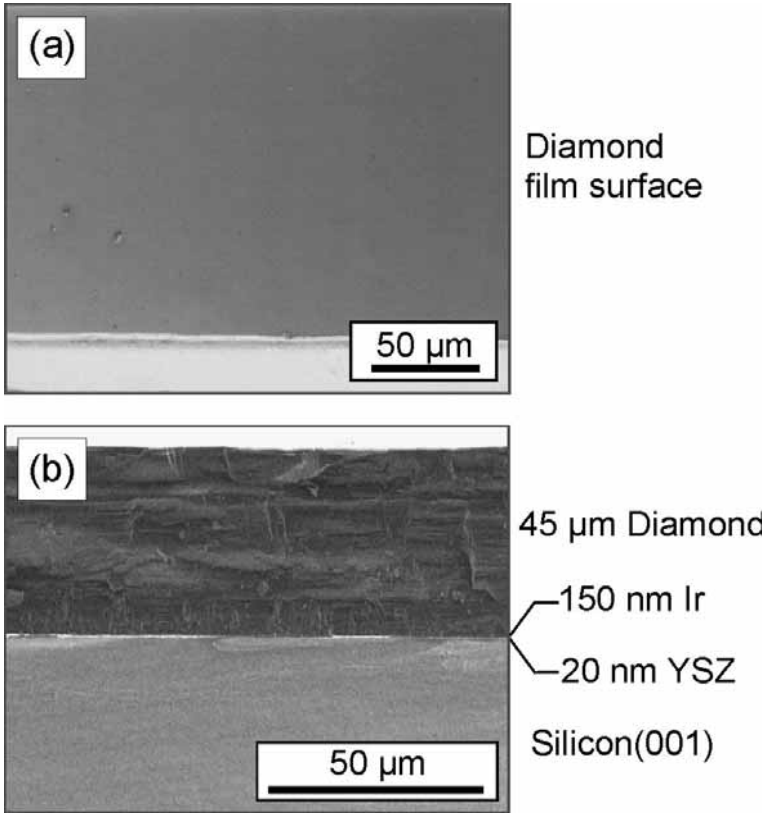






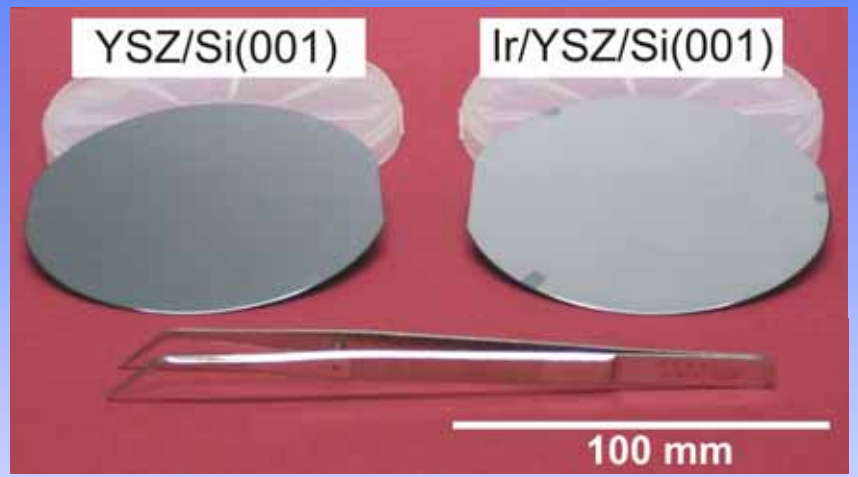


THICK DIAMOND FILMS ON Ir/YSZ/Si(001)



45 µm thick diamond film with good adhesion to the substrate

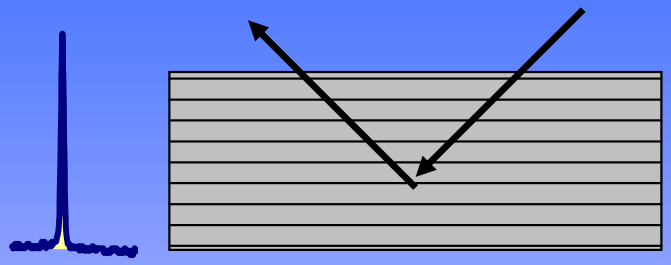
4" growth substrates



Diamond:

Tilt; Twist: 0.1 – 0.3°

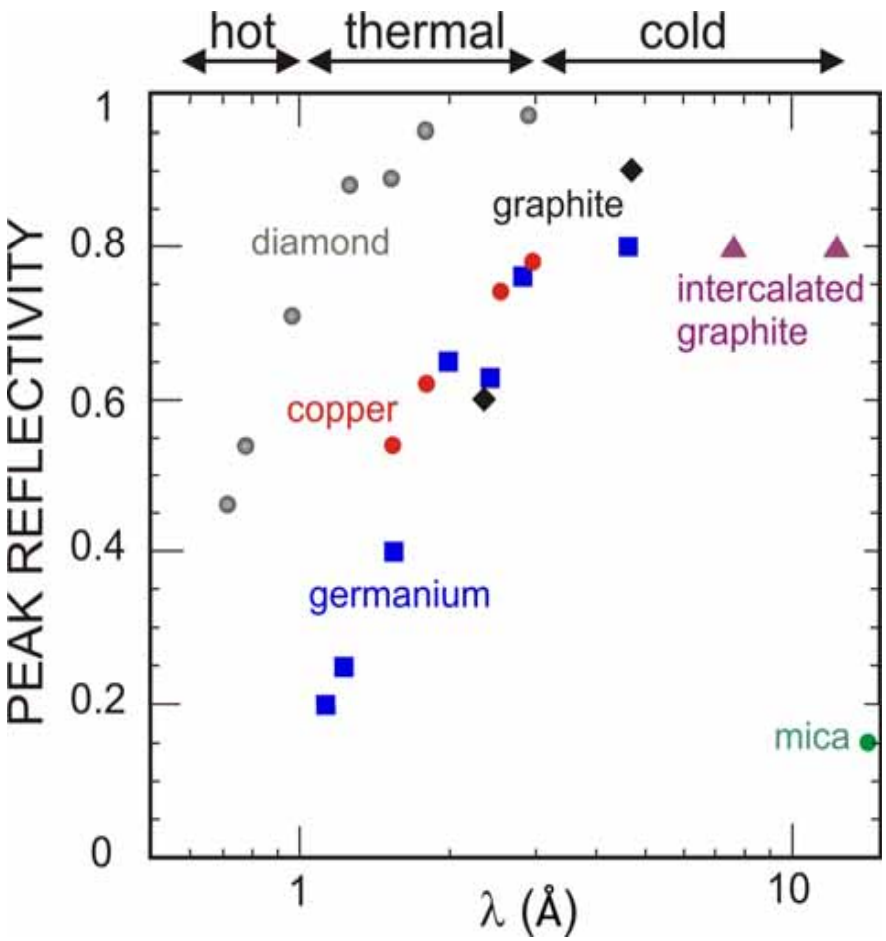
DIAMOND MOSAIC CRYSTALS FOR NEUTRON MONCHROMATORS



width < 10^{-3}° *Perfect crystal*

Mosaic crystals match neutron beam divergence and optimize integrated reflectivity at monochromacy

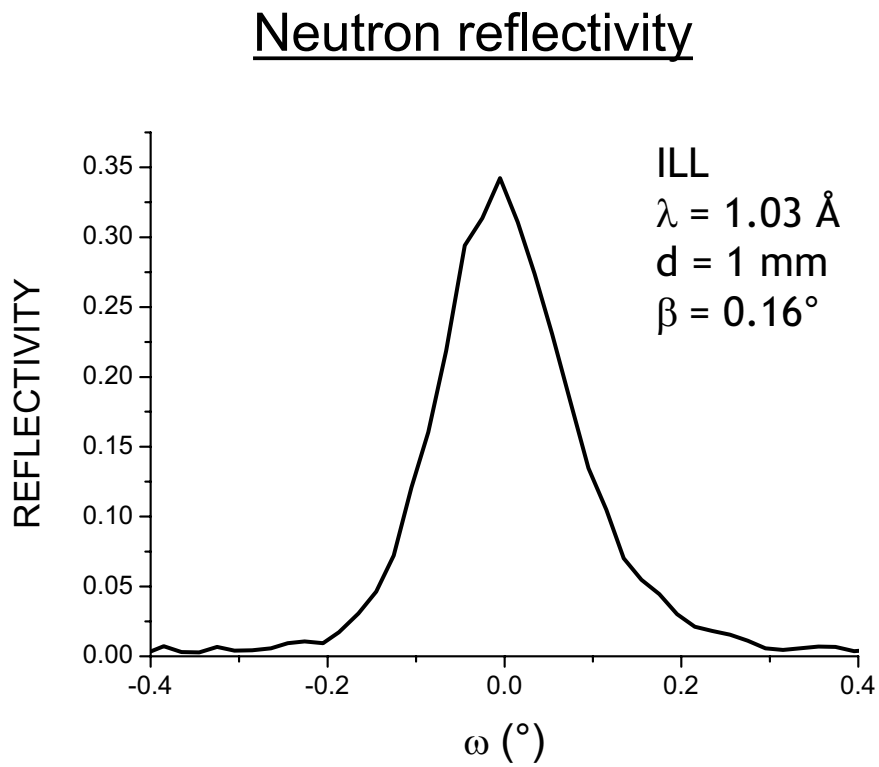
NEUTRON MONOCHROMATORS



Source: Ken Andersen, ILL, Grenoble

Possible intensity gain for thermal neutrons at 1 Å: factor 2-4

Required thickness:
several mm!!!



Maximum value up to now: 34%

Diamond films on Iridium for CARAT: specific aspects for detectors

CRUCIAL PARAMETERS FOR THE DETECTOR PERFORMANCE OF SINGLE CRYSTAL DIAMOND

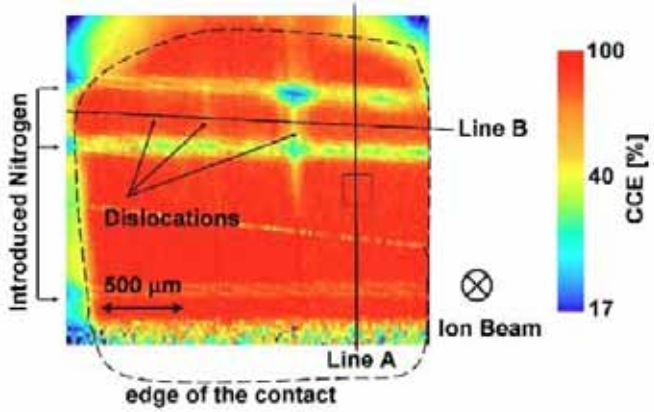


FIG. 2. (Color online) CCE map sensitive to hole transport, acquired at +125 V. The rectangular area has been used to extract the PHS spectra shown by the solid lines in Fig. 3.

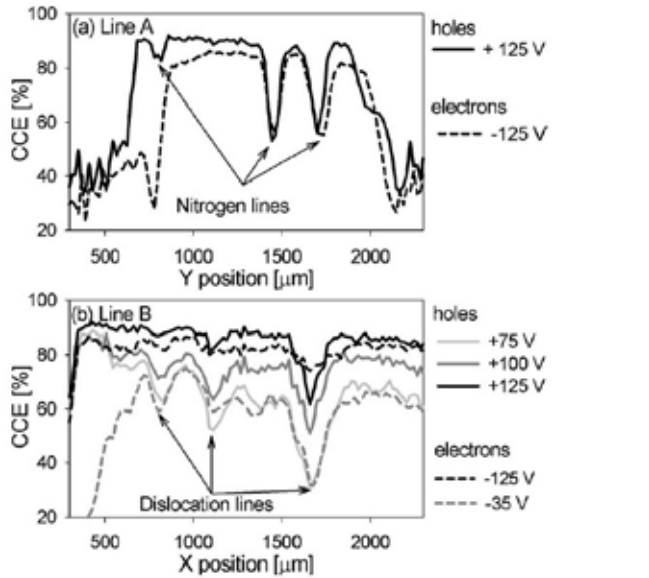
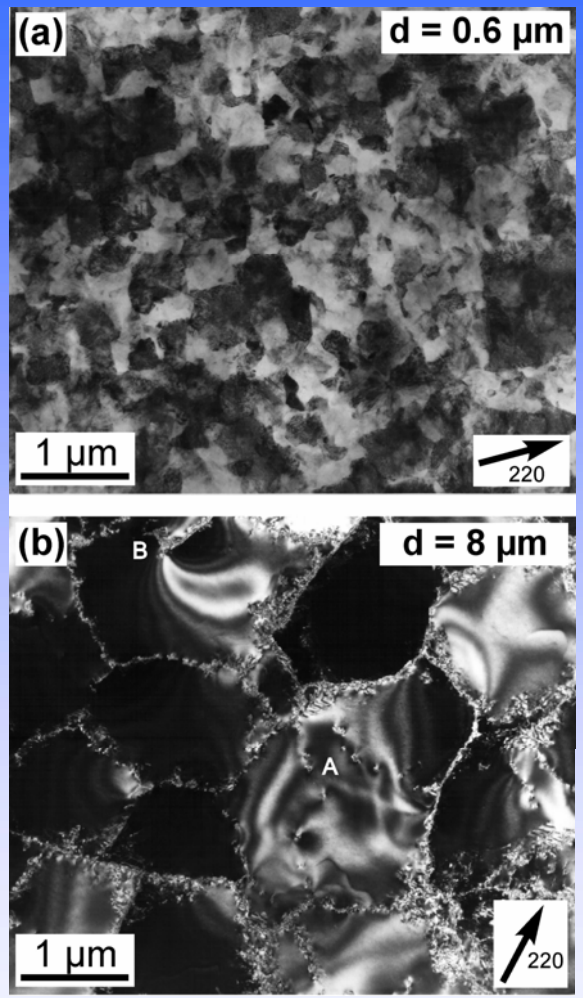


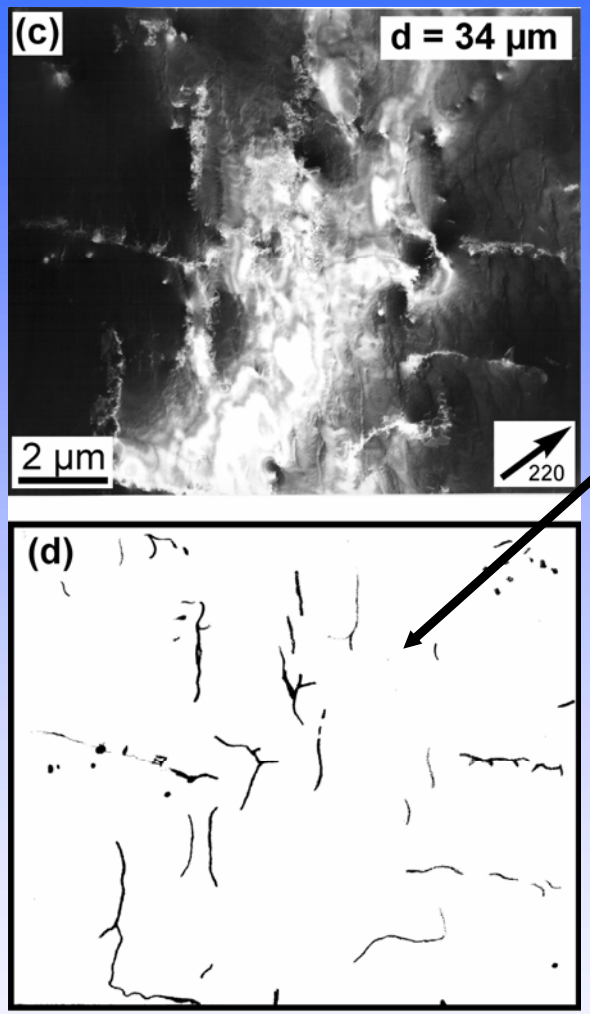
FIG. 4. CCE cross sections of various bias voltages. (a) extracted from line A and (b) extracted from line B indicated in Fig. 2.

A. Lohstroh, P. J. Sellin, S. G. Wang, A. W. Davies, J. Parkin, R. W. Martin, P. R. Edwards: Appl. Phys. Lett. 90 (2007) 102111

Nitrogen or defects formed by nitrogen as well as dislocations strongly limit the CCE in diamond

INTERNAL DEFECT STRUCTURE: TRANSMISSION ELECTRON MICROSCOPY (TEM)





Schematic sketch of defect bands from TEM image in (c)

During textured growth:

closed network of grain boundaries

isolated short defect bands

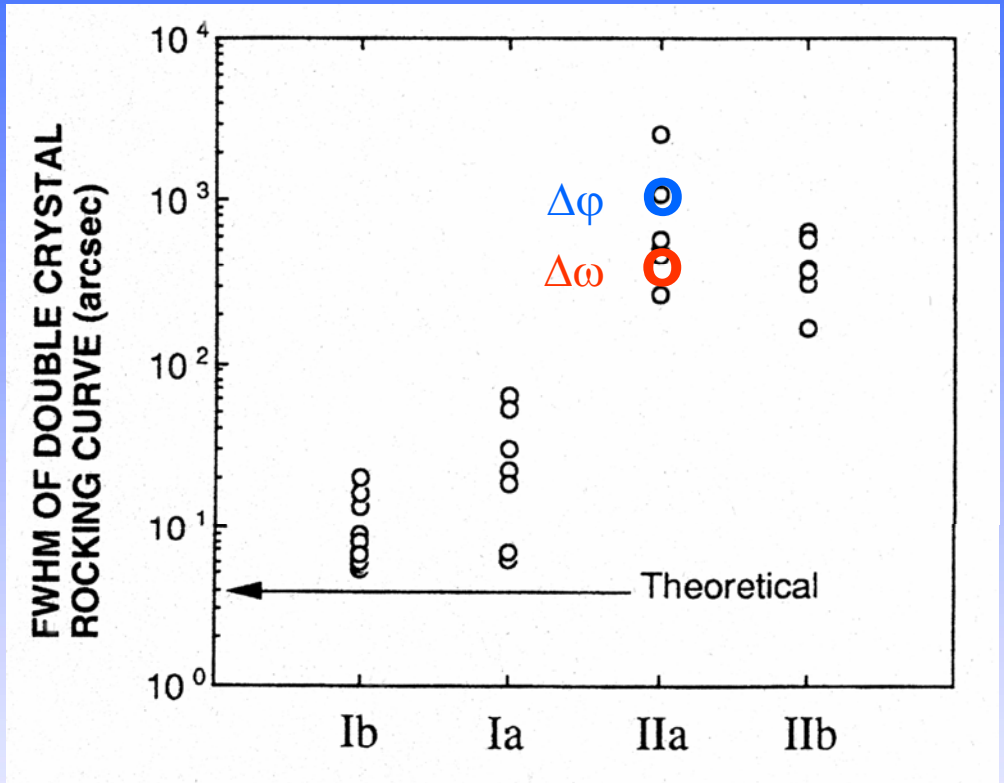
(quasi single crystal)

Estimated density of dislocations for the 34 µm film: ~ 5–10x10^{8}cm^{-2}

Reduction by ~ 2 orders of magnitude during textured growth

COMPARISON WITH NATURAL DIAMOND SINGLE CRYSTALS

Mosaic spread



Typical rocking curve widths for different types of diamond crystals

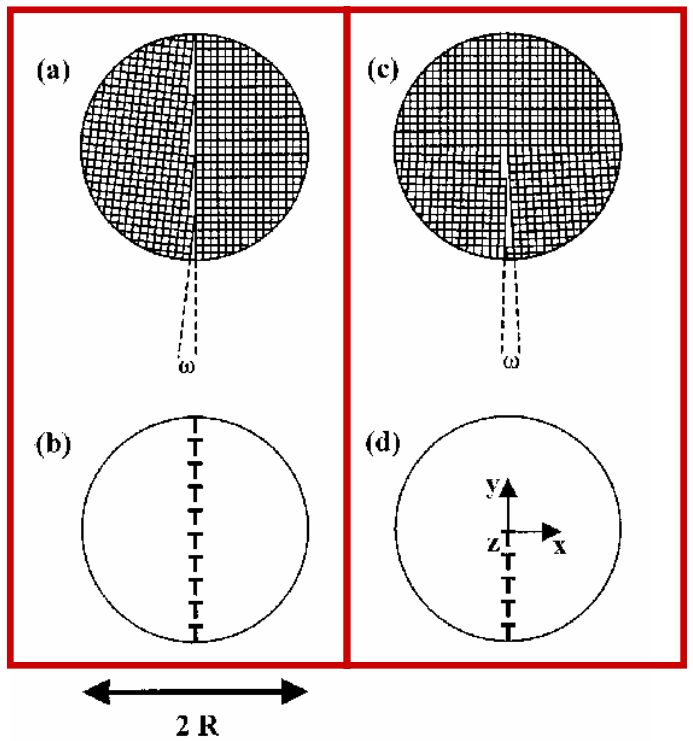
Ref. S. Fujii et al. Appl. Phys. A 61 (1995) 331

Dislocation densities:

Our films: 5-10 x $10^8 cm^{-2}$

Large natural type IIa-diamonds:
typical > $10^8 cm^{-2}$
Ref.: A.R. Lang in *The Properties of Diamond* ed. by J. E. Field (Academic, London 1979)

COMPARISON: GRAIN BOUNDARY ⇔ DISCLINATION

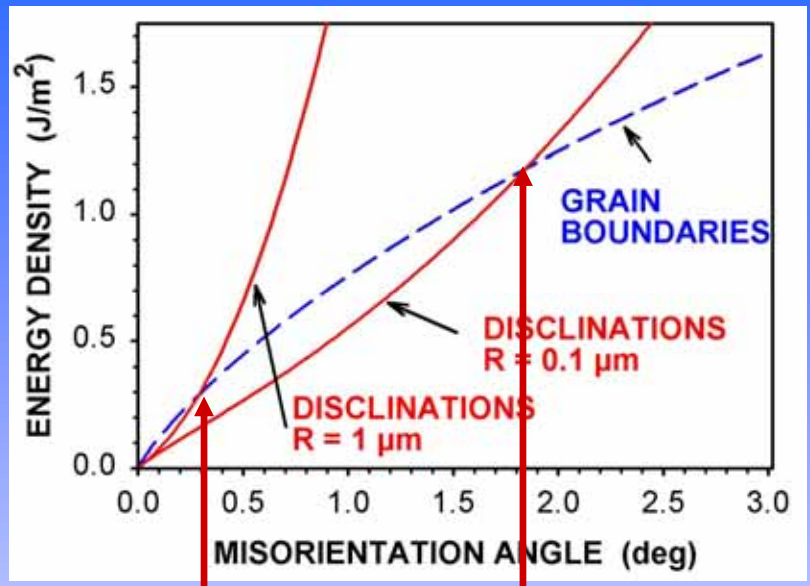


$E^{grain\ bound.} \propto \omega \ln(c\omega^{-1})\ 2\ R\ L$

$E^{disclination} \propto \omega^2 R^2 L$

$(+\ 1/2\ E^{grain\ bound})$

Energy density E/2RL



Disclinations energetically favoured

Grain boundaries energetically favoured

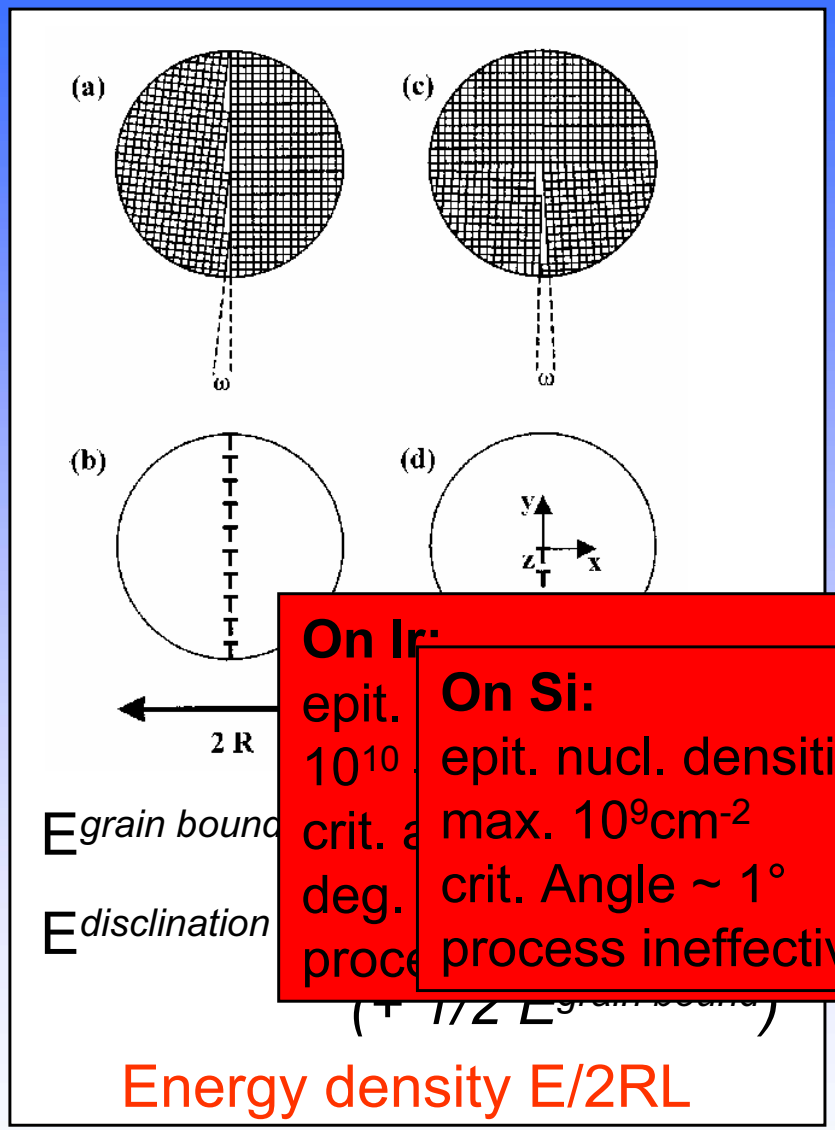
Disclinations energetically favoured

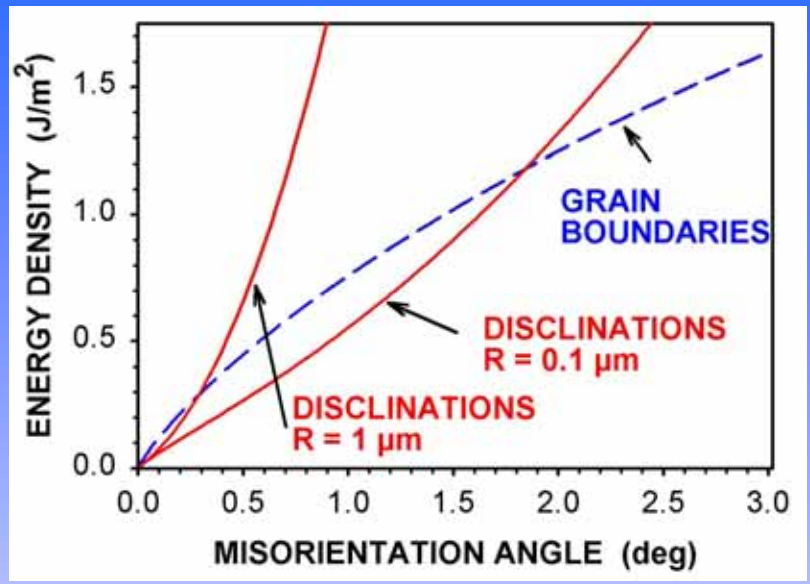
Grain boundaries energetically favoured

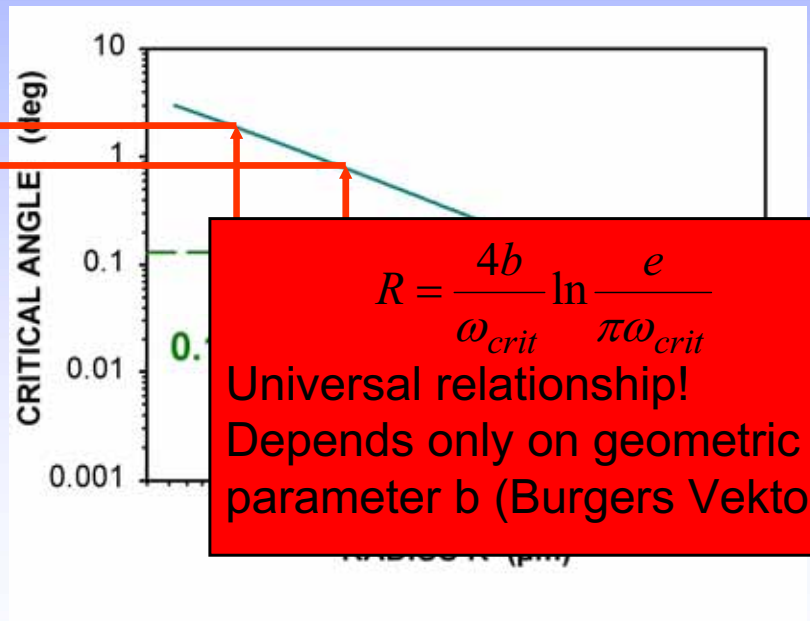
Critical angle: ~ 1.8°

Critical angle: ~ 0.3°

COMPARISON: GRAIN BOUNDARY ⇔ DISCLINATION







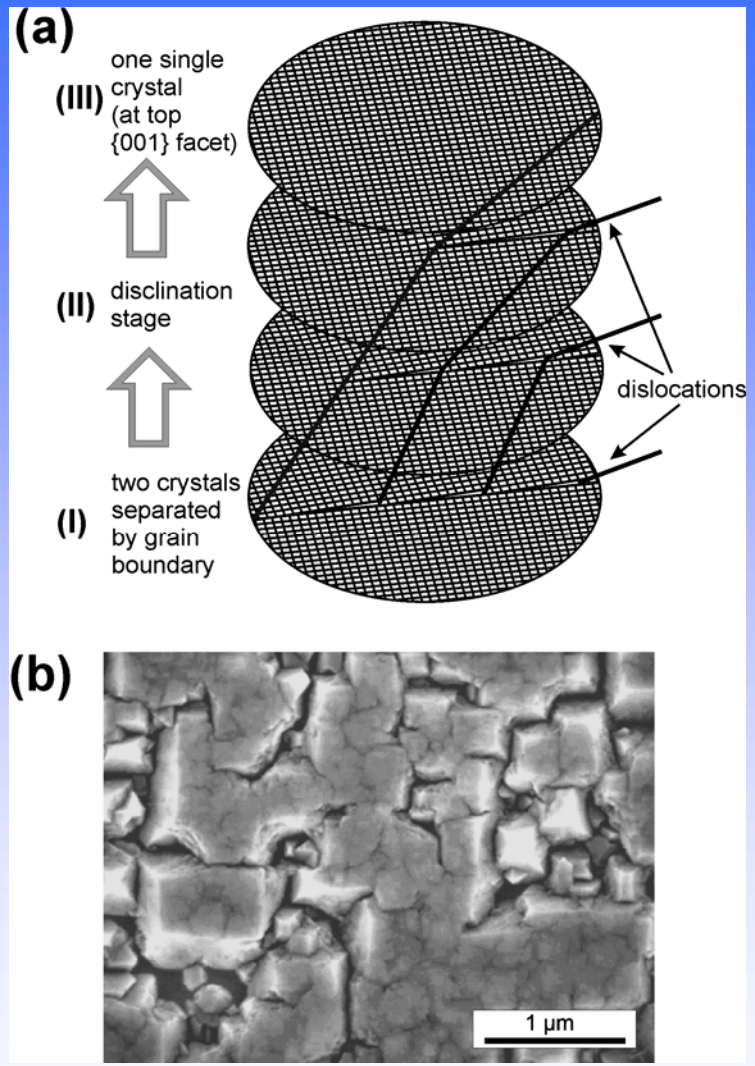
On Ir:
epit.
10^{10}
crit. a
deg.
proce

On Si:
epit. nucl. densities
max. $10^9 cm^{-2}$
crit. Angle ~ 1°
process ineffective

$$R = \frac{4b}{\omega_{crit}} \ln \frac{e}{\pi \omega_{crit}}$$

Universal relationship!
Depends only on geometric parameter b (Burgers Vektor)

REDUCTION OF DISLOCATION DENSITY

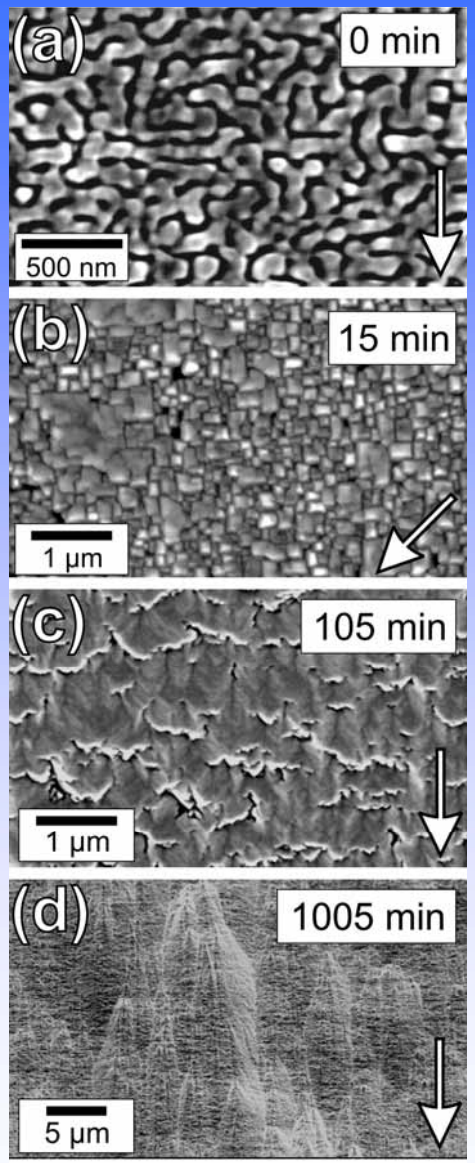


Dislocation densities in current state of the art layers?

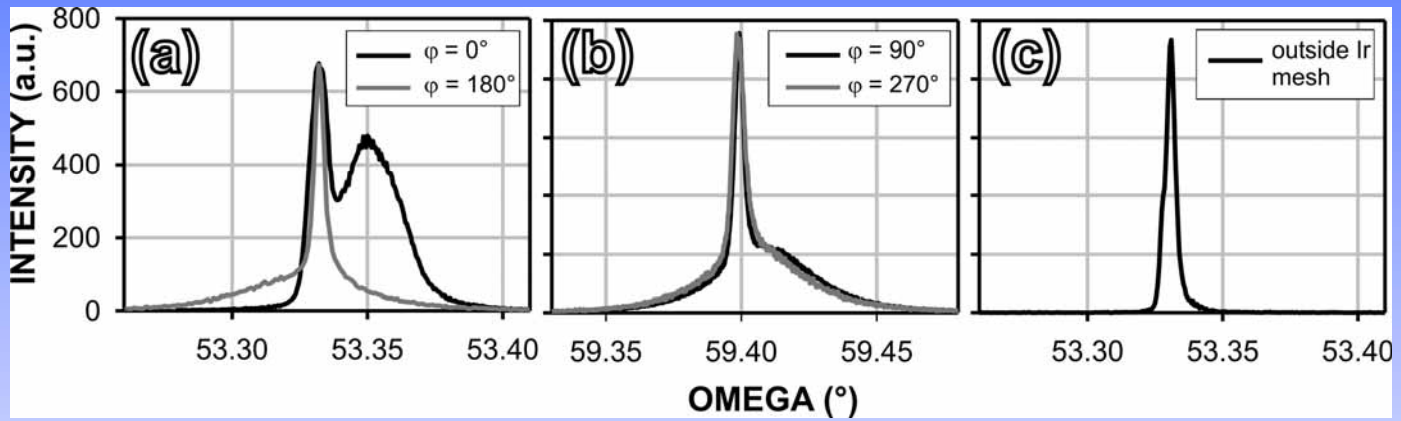
How far can the concept be extended by simple growth of thick layers?

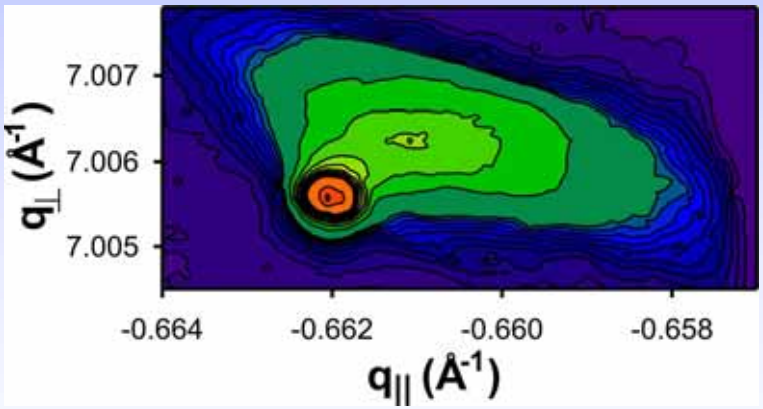
Controlled Epitaxial Lateral Overgrowth (ELO)

EXPLORATORY EXPERIMENTS WITH ELO ON HPHT SINGLE CRYSTALS



13 nm Ir on Ib single crystal





T. Bauer et al., DRM 16 (2007) 711

PATTERNING OF THE NUCLEATION LAYER

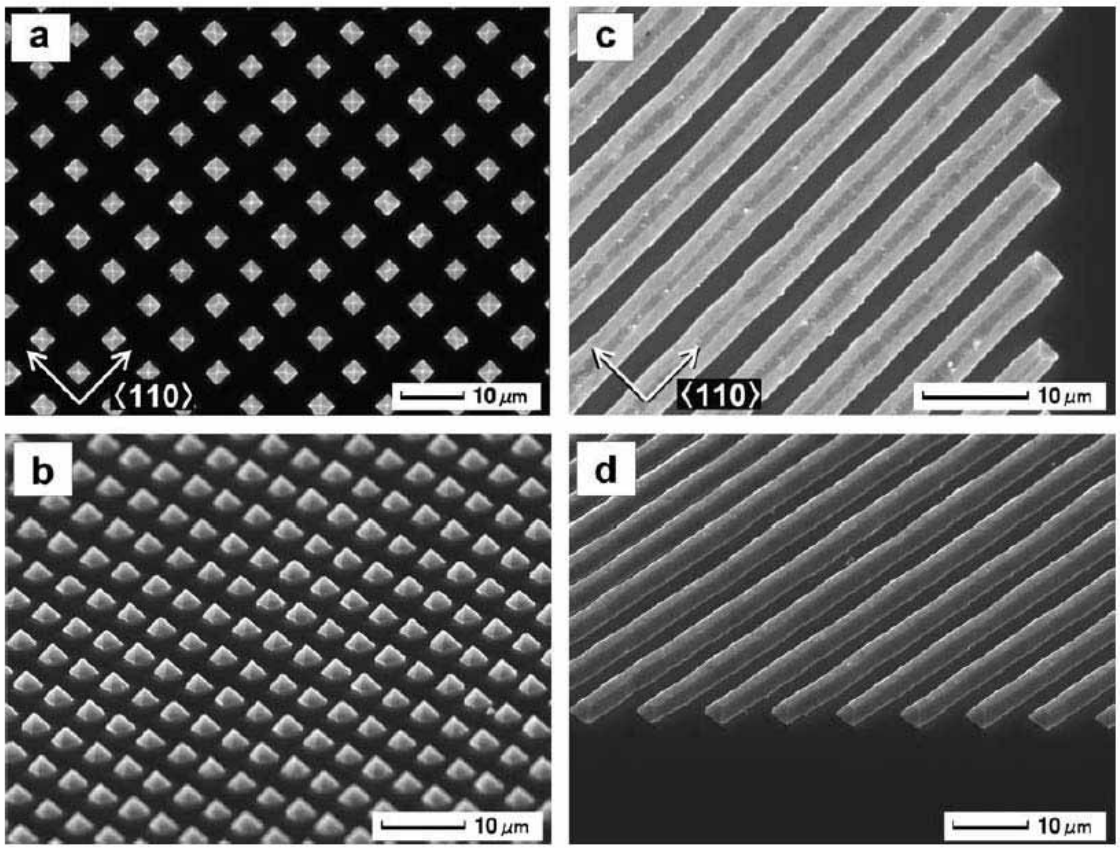
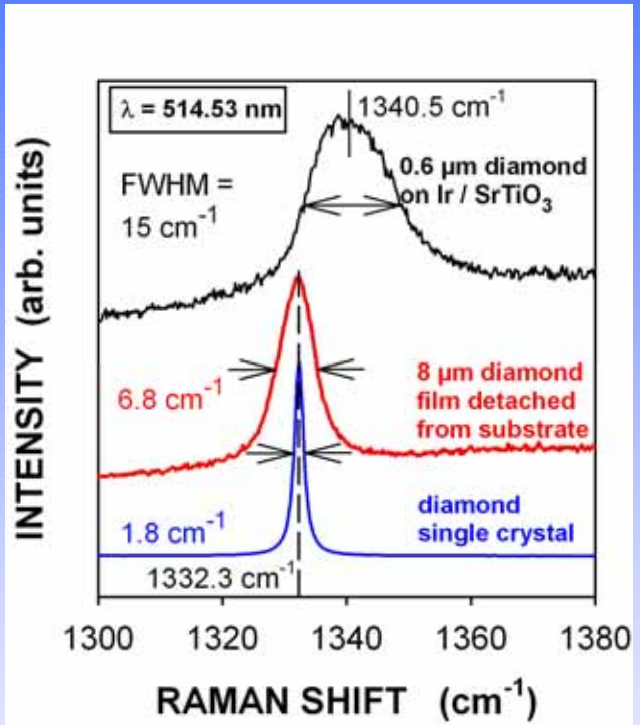


Fig. 6. Pyramidal array (a, b) and line/space (c, d) of heteroepitaxial diamond grown on Ir(001) surface.

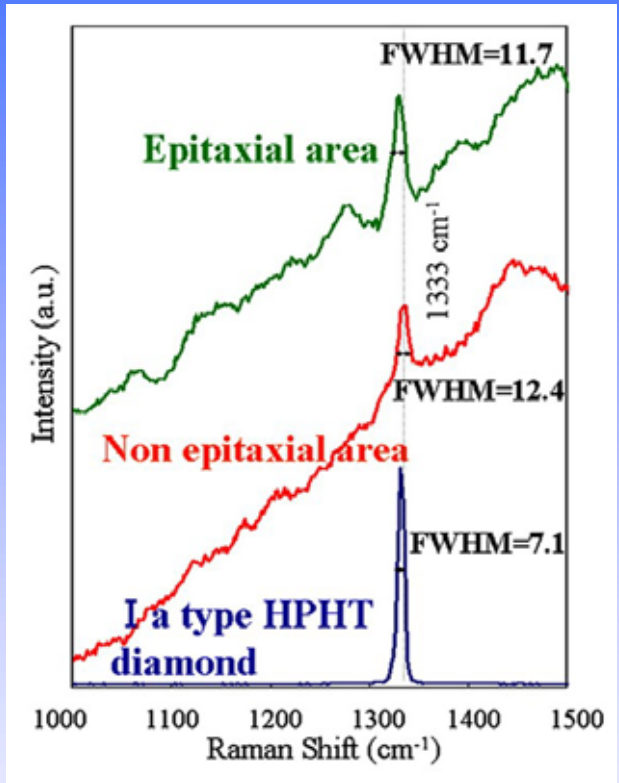
Y. Ando, J. Kuwabara, K. Suzuki, A. Sawabe, DRM 13 (2004) 1975.

RAMAN SPECTRA OF HETEROEPITAXIAL DIAMOND LAYERS ON Ir

Diamond on Ir/$SrTiO_3$(001)



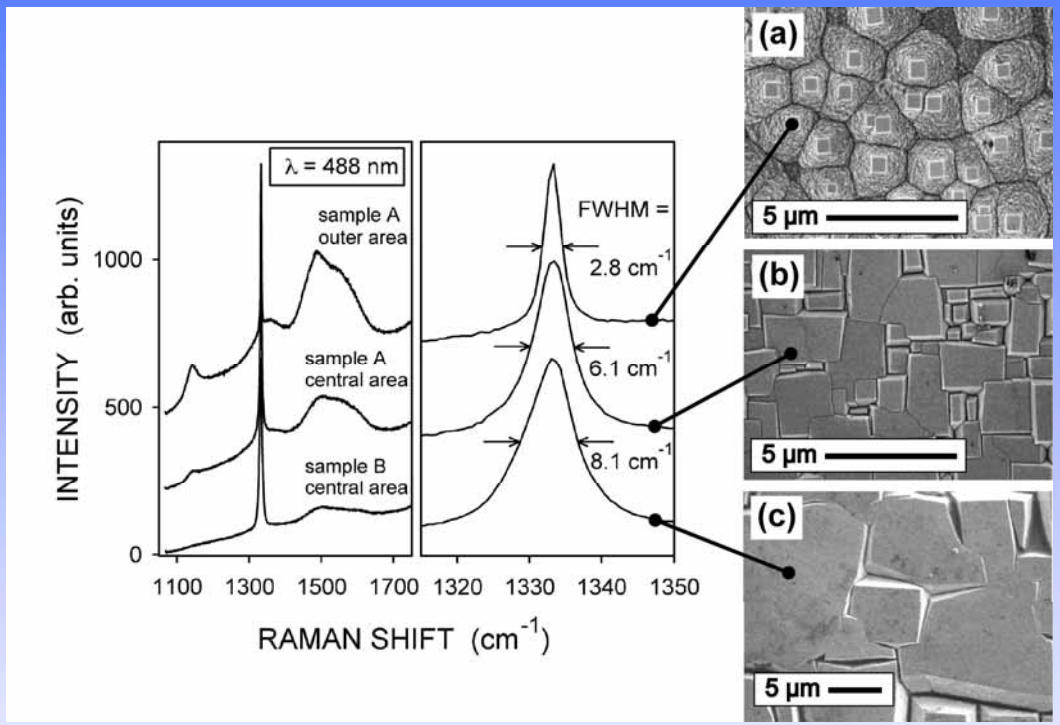
Diamond on Ir/Al_2O_3



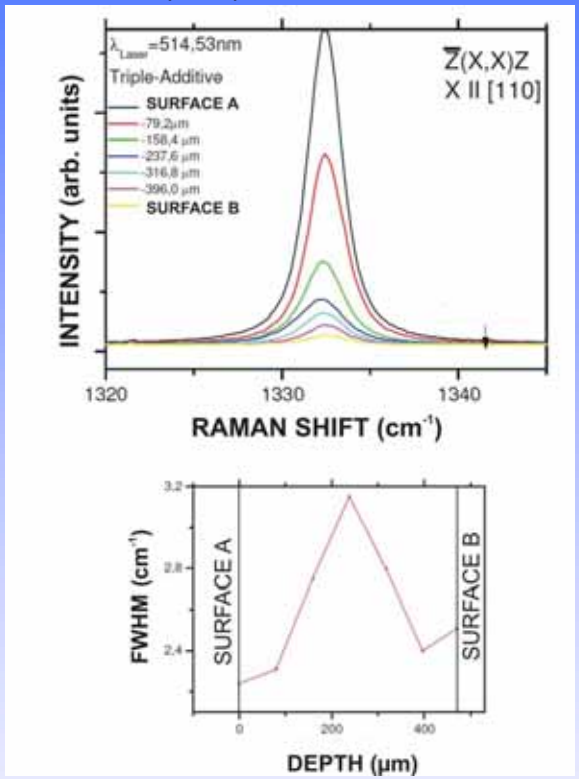
A. Samato, …. A. Sawabe, T. Suzuki, Diamond Relat. Mater. 17 (2008) 1039.

RAMAN SPECTRA OF HETEROEPITAXIAL DIAMOND LAYERS

Raman spectra of epitaxial diamond layers on silicon



Raman spectra of a 475 µm thick diamond layer grown on Ir/YSZ/Si(001)



Schreck, Hörmann, Roll, Bauer, Stritzker:
NDFCT 11 (2001) 189.

SUMMARY

- Mosaic spread of heteroepitaxial diamond on Ir/YSZ/Si(001) outperform former layers on Ir/$SrTiO_3$(001)
- Mosaic spread (Lowest values ~0.1-0.3°) is in the range of standard IIa layers but still 2 orders of magnitude higher than for high quality IIa's
- Raman line width: lowest values ~ 2.5 cm^{-1}
- Upscaling on Ir/YSZ/Si(001): 4 inch size for the Ir films and more than 10 cm^2 for the diamond (critical: homogeneity)
- First measurements of transient currents (TC) to test detector performance ➔ **see contribution Elèni Berdermann**
- Concepts for further reduction of dislocation densities: ELO