



CIBA



A review of transmission channeling experiments in thin crystals

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Part I

Transmission Channeling Imaging of Crystal Defects

Part II

Channeling Patterns with ultra-thin silicon membranes

Part I

Transmission Channeling Imaging of Crystal Defects

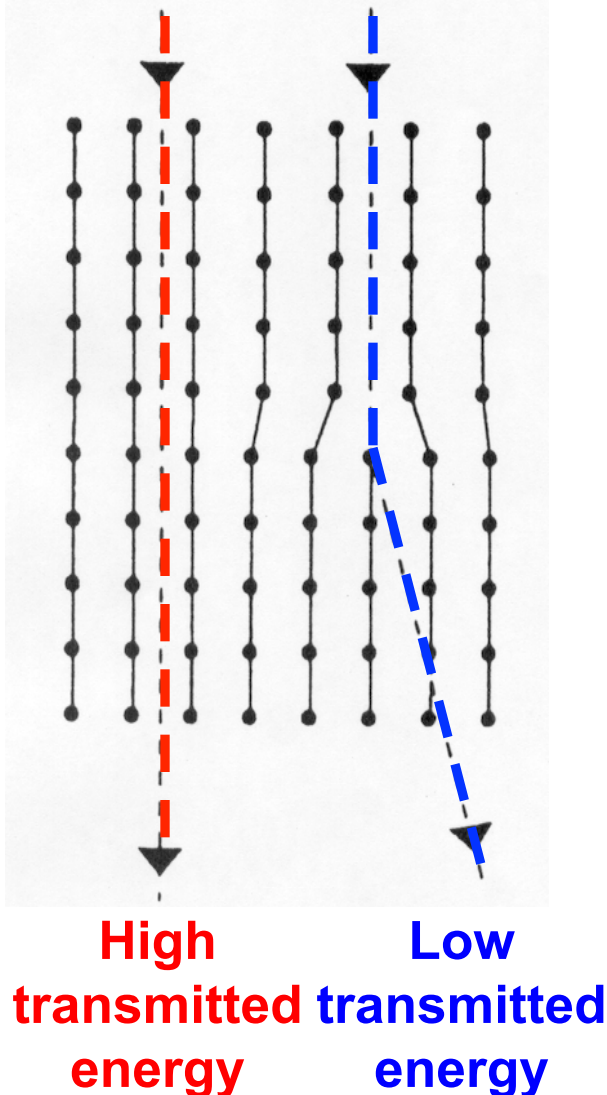
Use focused MeV ion beams to image crystal defects in crystals 20 to 40 microns thick in a nuclear microprobe

EXPERIMENTAL ADVANCES IN CAPABILITIES:

- I. Spatial resolution for channeling of 60 nm (now 10 nm) in high demagnification microprobe.
- II. New methods of altering beam/crystal tilt – channeling analysis without a goniometer
- III. Greatly increased counting statistics.

Basis of transmission channeling

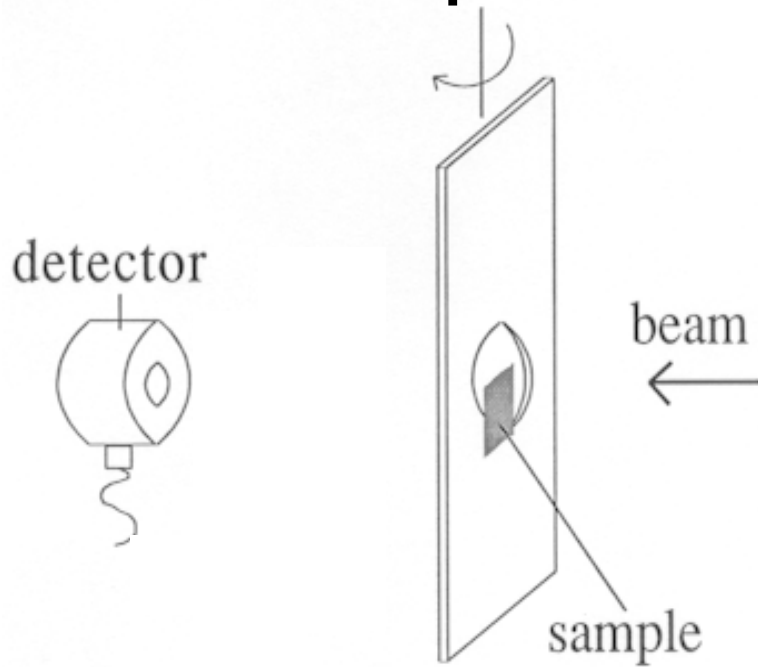
Focused MeV proton beam



- **Well-channeled protons** pass through crystal close to the middle of the lattice planes. Here the electron density is low, so they lose energy at half the random rate **[?] High transmitted energy**
- If channeled ions hit a **lattice defect** they dechannel and pass through remaining crystal thickness with the higher, random rate of energy loss **[?] Low transmitted energy**
- Measure transmitted proton energy-intensity in scanned area to image crystal defects

Basis of transmission channeling

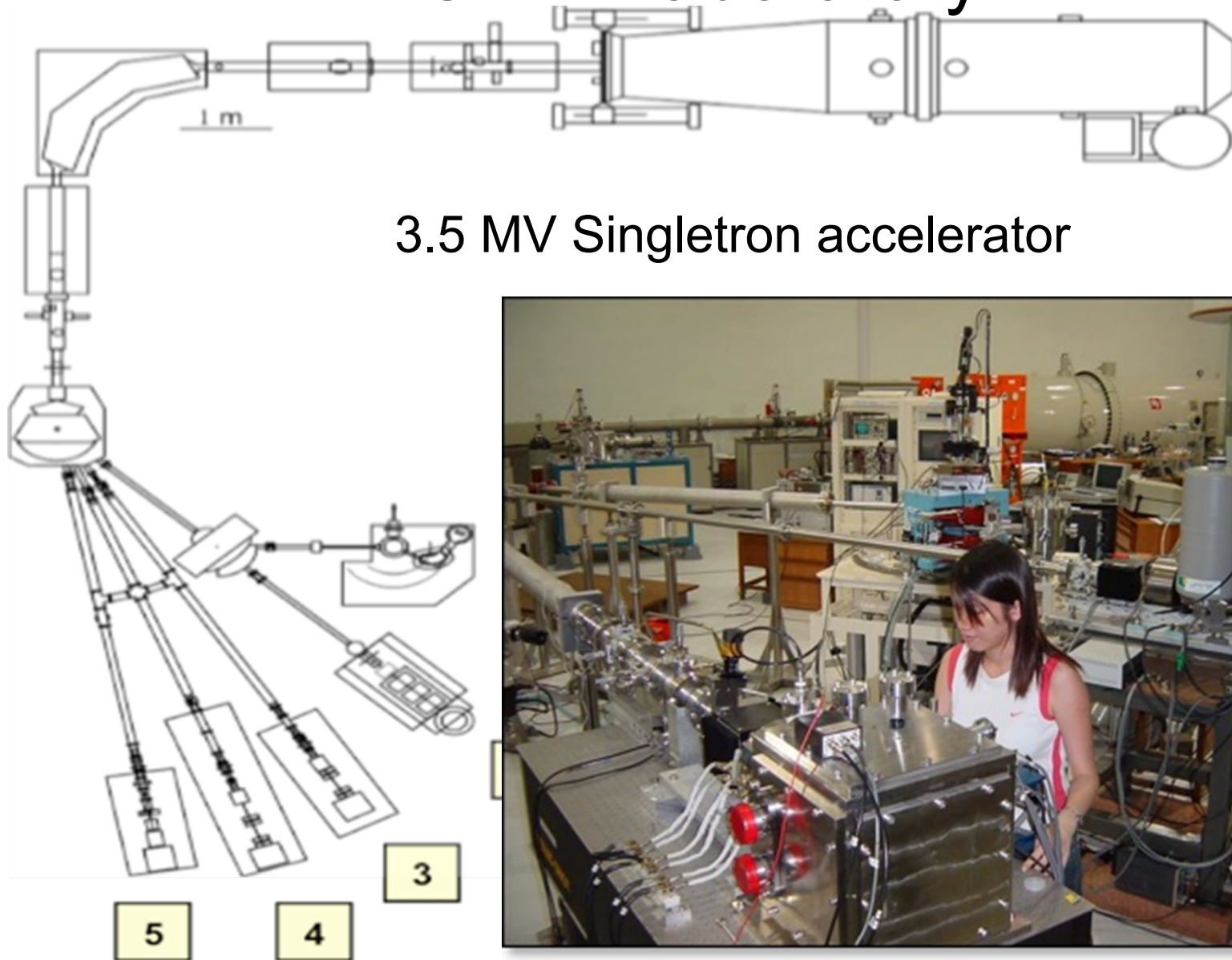
Basic set-up



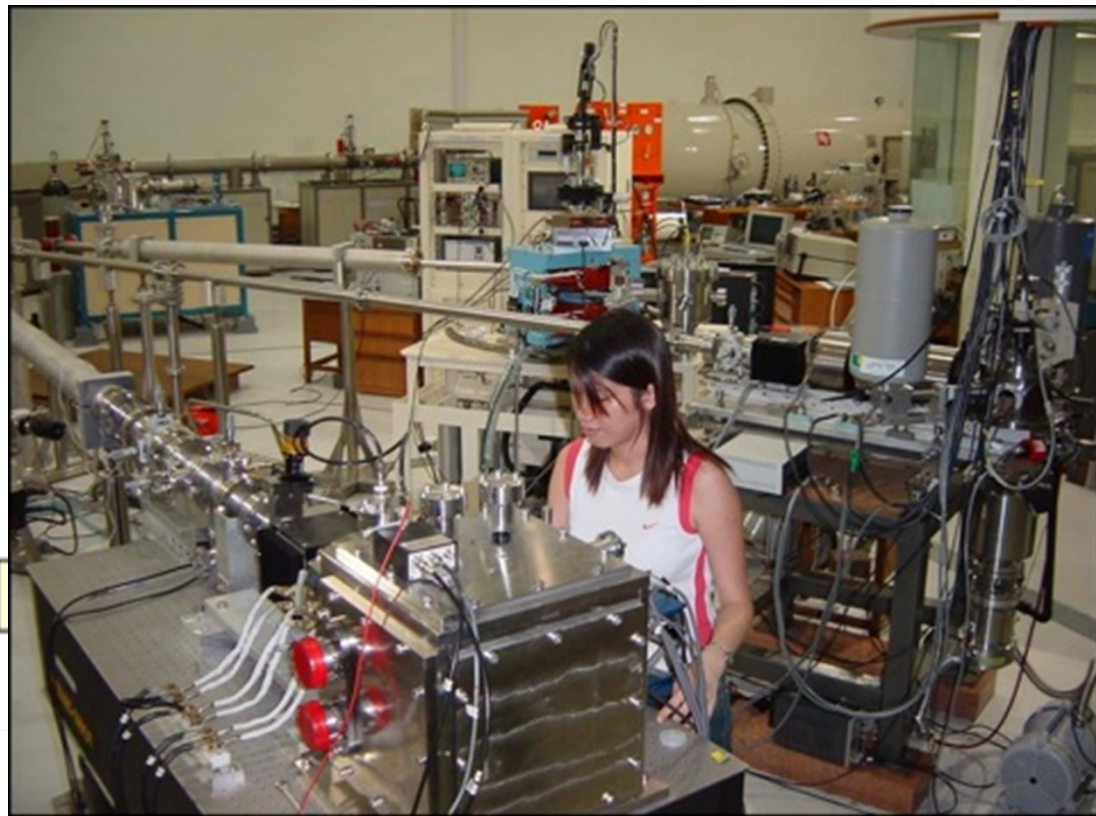
- Thin crystal to $\approx 50 \text{ nm}$, (less than range of a 2-3 MeV proton)
- Align thinned crystal with proton beam focused to a small spot in a nuclear microprobe.
- Measure energies of transmitted protons in an array of pixels as focused beam scans over surface.

- Crystal defects can be point defects, dislocations, stacking faults, precipitates, twins, voids, bubbles etc
- Want to identify what defects are present, geometry, alignment, density.

CIBA Laboratory



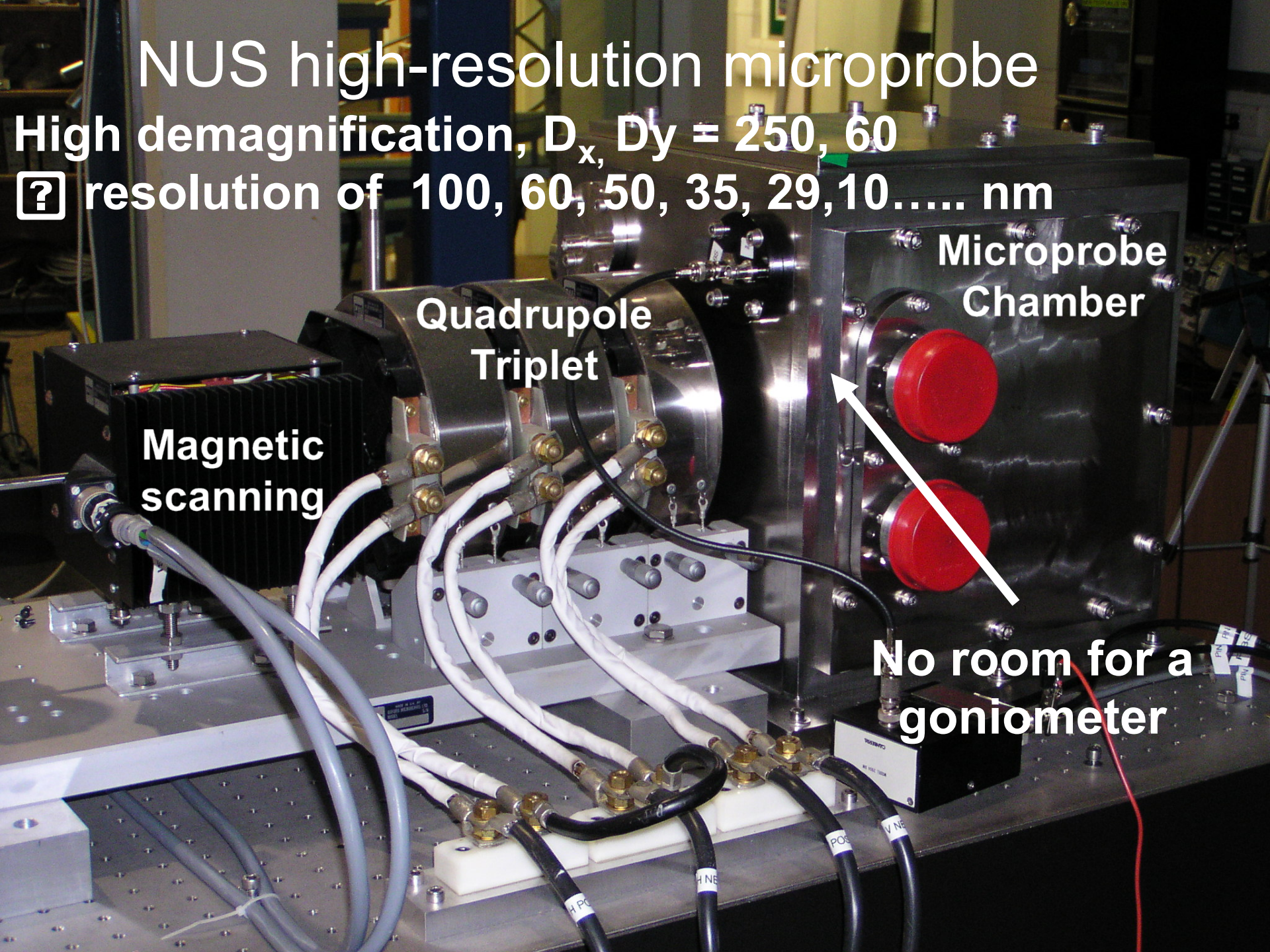
3.5 MV Singletron accelerator



NUS high-resolution microprobe

High demagnification, $D_x, D_y = 250, 60$

Resolution of 100, 60, 50, 35, 29, 10..... nm



Quadrupole
Triplet

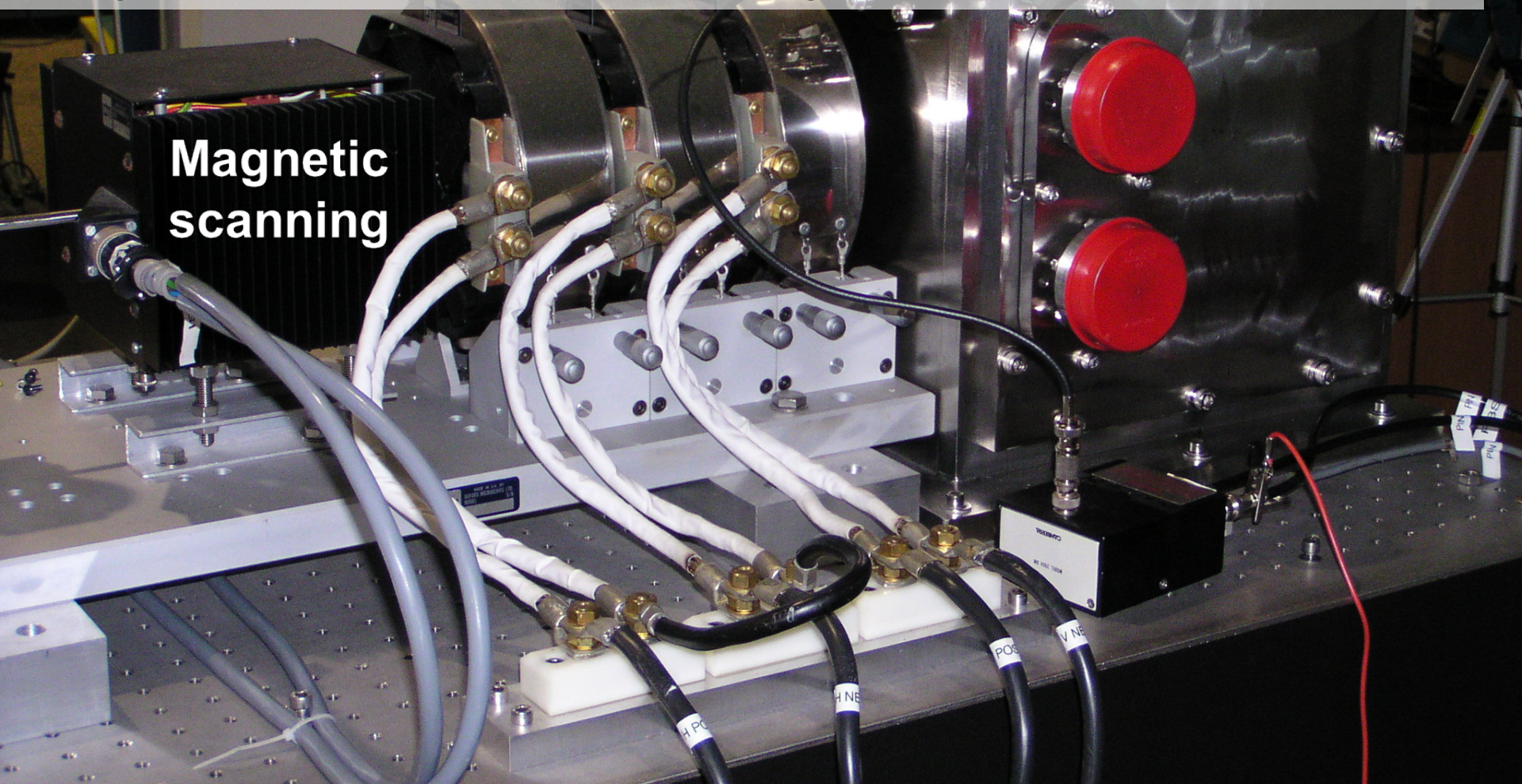
Magnetic
scanning

Microprobe
Chamber

No room for a
goniometer

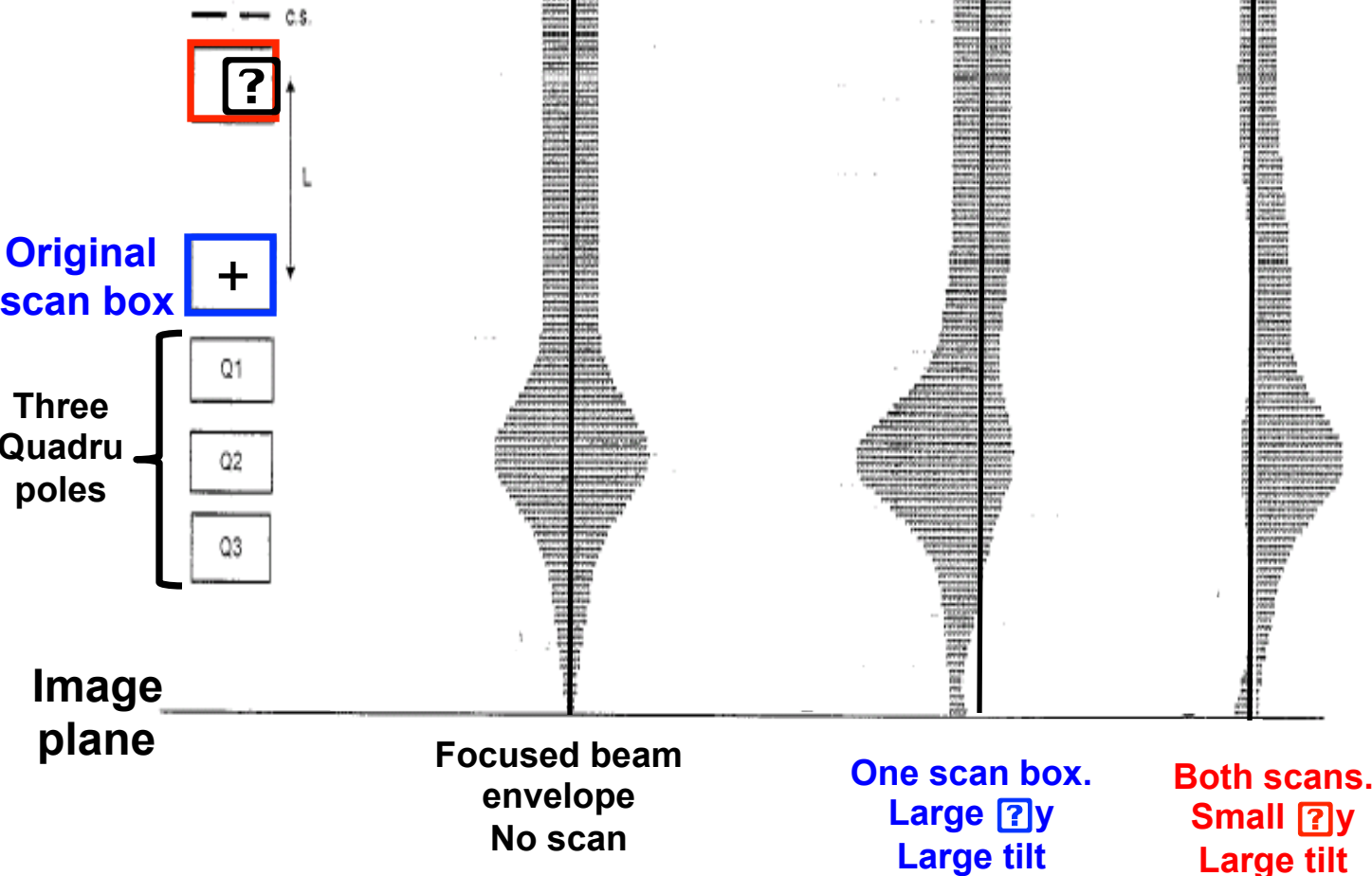
Channelling without a Goniometer

- Little space for a goniometer in a high demagnification microprobe.
- How to perform channelling measurements ?
- Beam rocking system developed – add second magnetic scanning system operated in opposite polarity

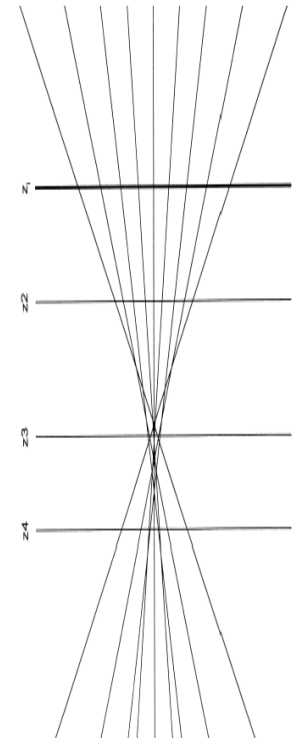


Beam rocking

Add another scan
box, opposite
polarity



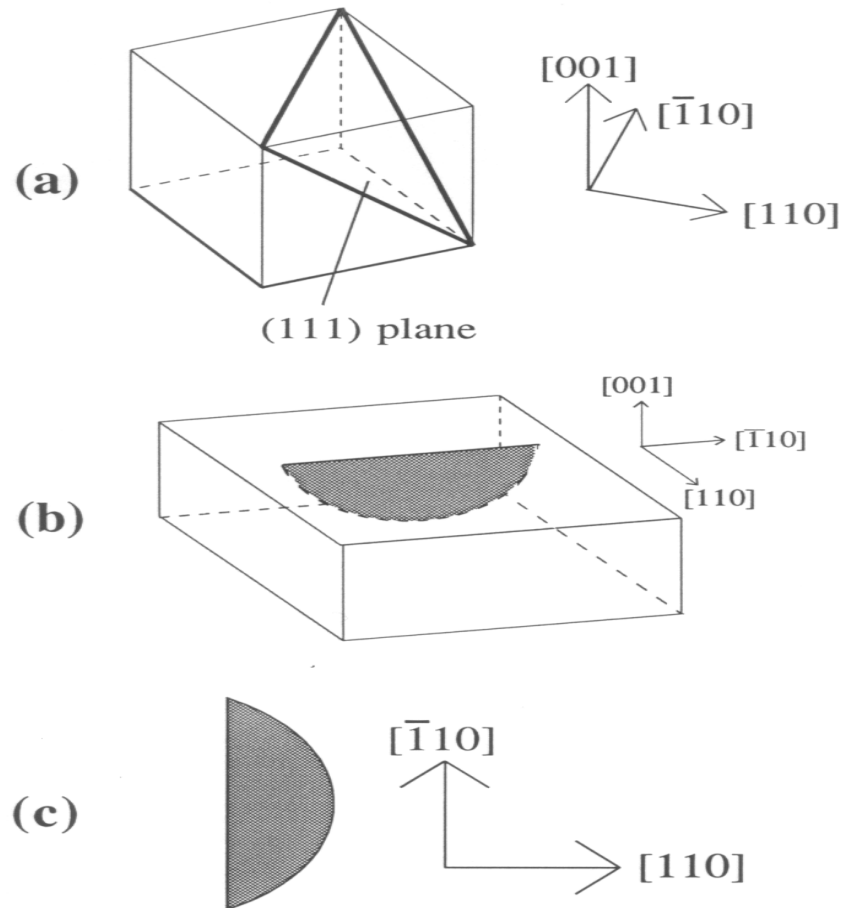
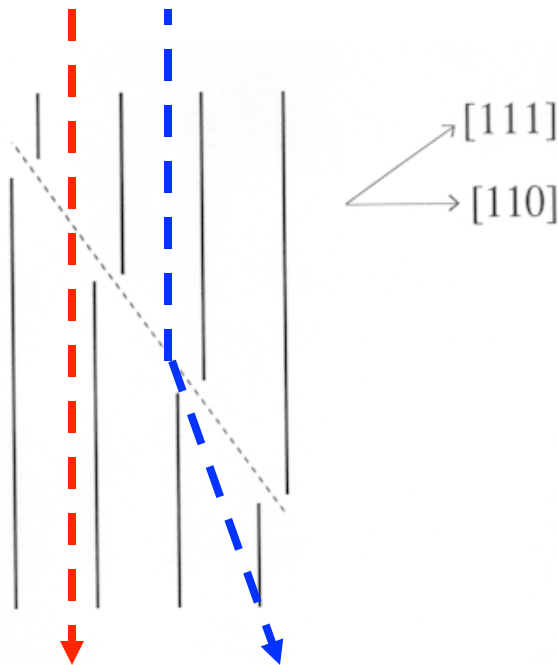
Beam
continuously
rocks through
range of tilts



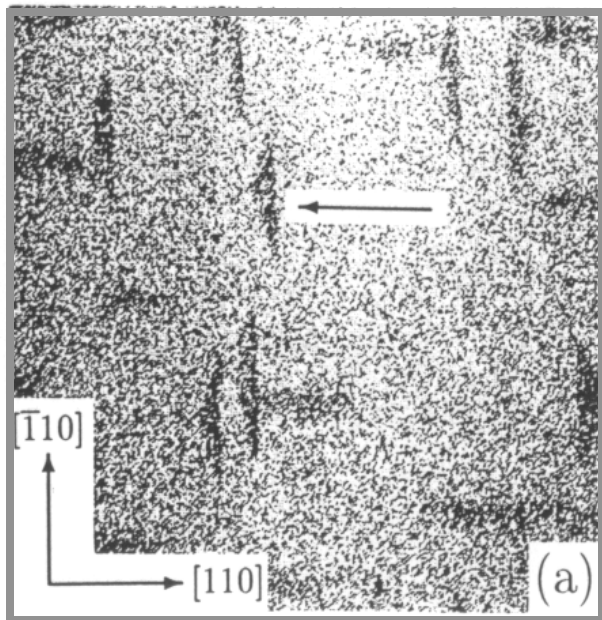
Can achieve tilt of $3\Delta\theta$ with
 $\Delta y = 2\Delta y_m$

Stacking Faults

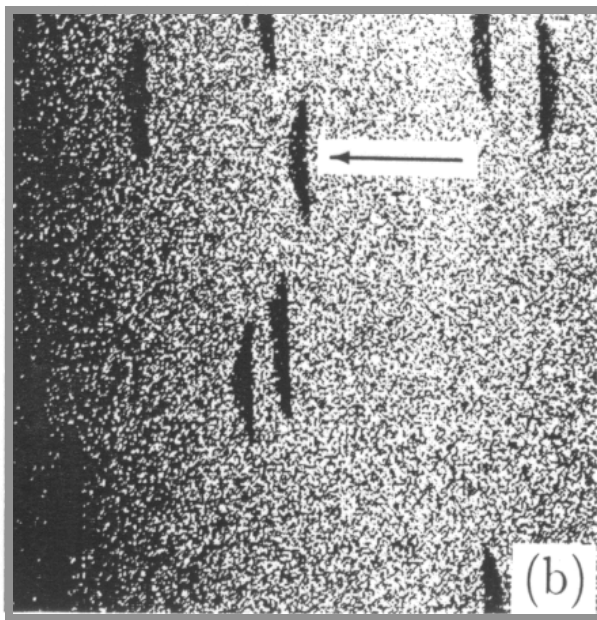
Inclined “D” shaped faults, running along $\{111\}$ planes at angle of $55\frac{1}{2}^\circ$ to the surface



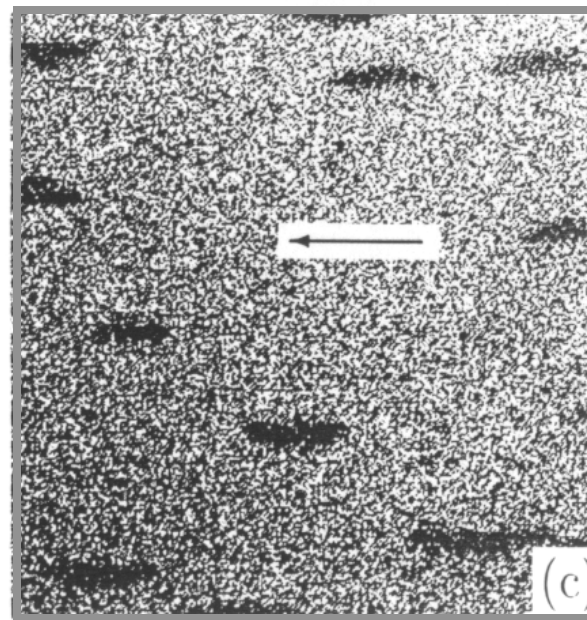
Transmission Channelling Images of Stacking Faults



[100]



(110)

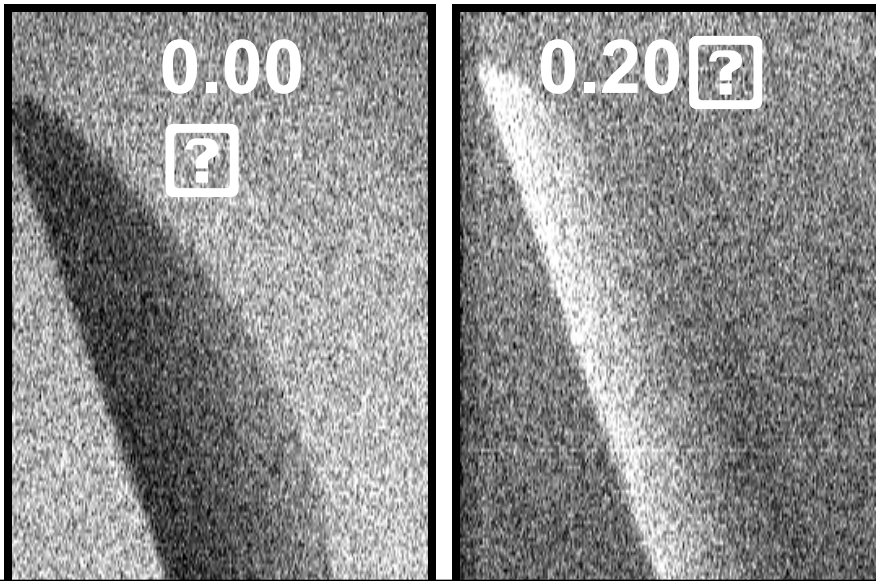


(011)

*Faults appear when aligned with different channeling planes,
allowing their translation vector to be found
(Dark [?] high energy loss)*

**100 x 100 [?]m² crystal area, aligned with different
channeling planes**

New, High-Magnification Transmission Channeling Images

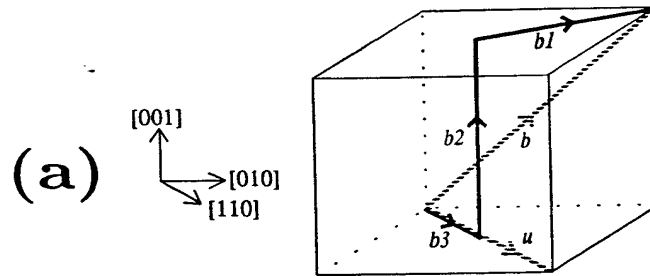


Published as

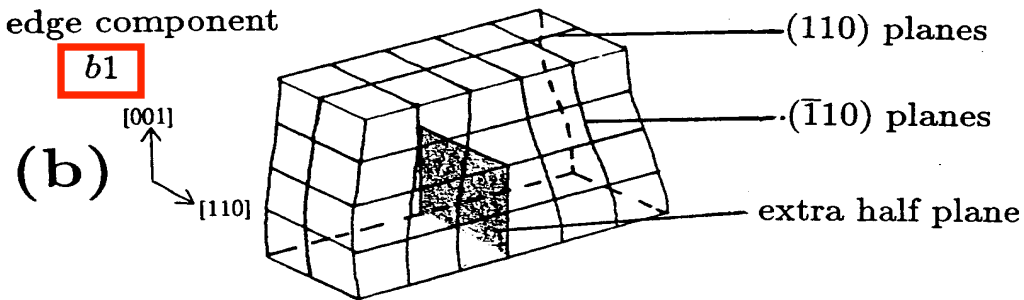
- *Equivalent effects of a lattice translation and rotation on planar channeled protons*, M B H Breese and P J M Smulders, *Physical Review Letters* 81 (1998) 51
- *Enhanced Planar Channelling of MeV Protons Through Thin Crystals*, M. B. H. Breese, M. A. Rana, T. Osipowicz, *Physical Review Letters* 93 (2004) 105505
- *Observation of Many Coherent Oscillations for MeV Protons Transmitted Through Stacking Faults*, M. B. H. Breese, E. J. Teo, M. A. Rana, L. Huang, J. van Kan, F. Watt, P. J. C. King, *Physical Review Letters* **92(4)** (2004) 045503

? ^{24}MeV protons, (110) planes

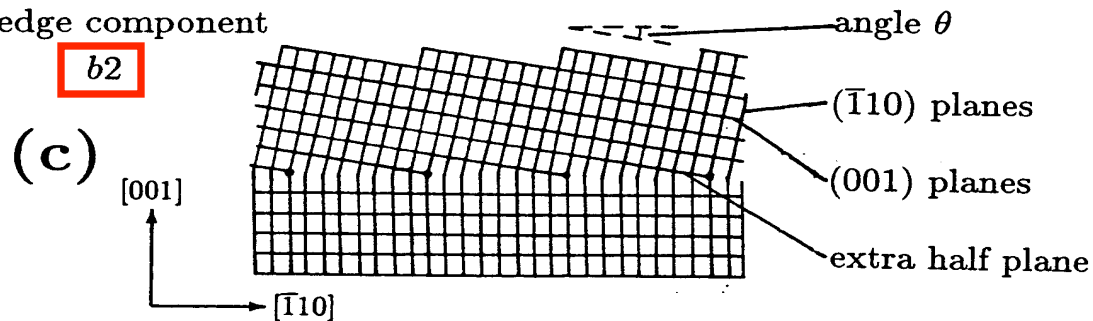
60° Dislocations



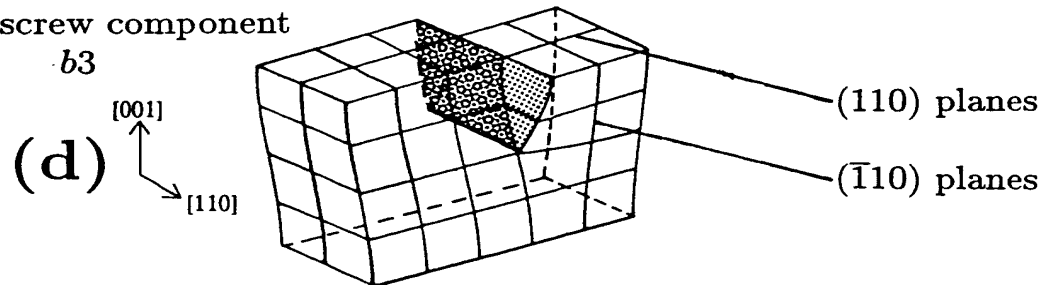
edge component



edge component

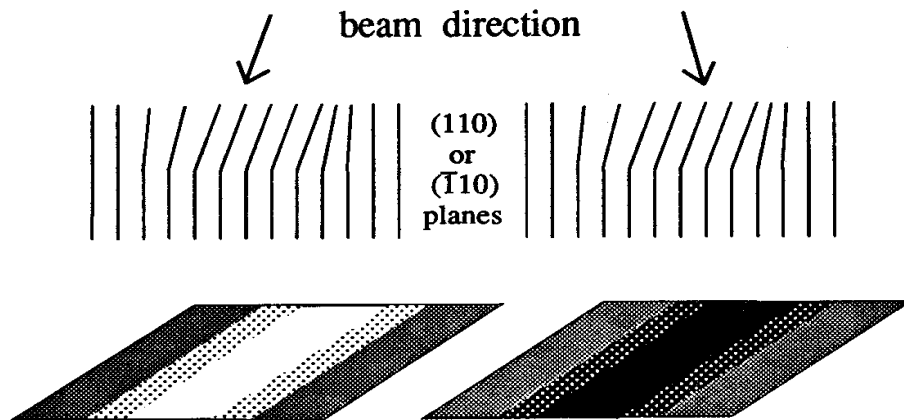
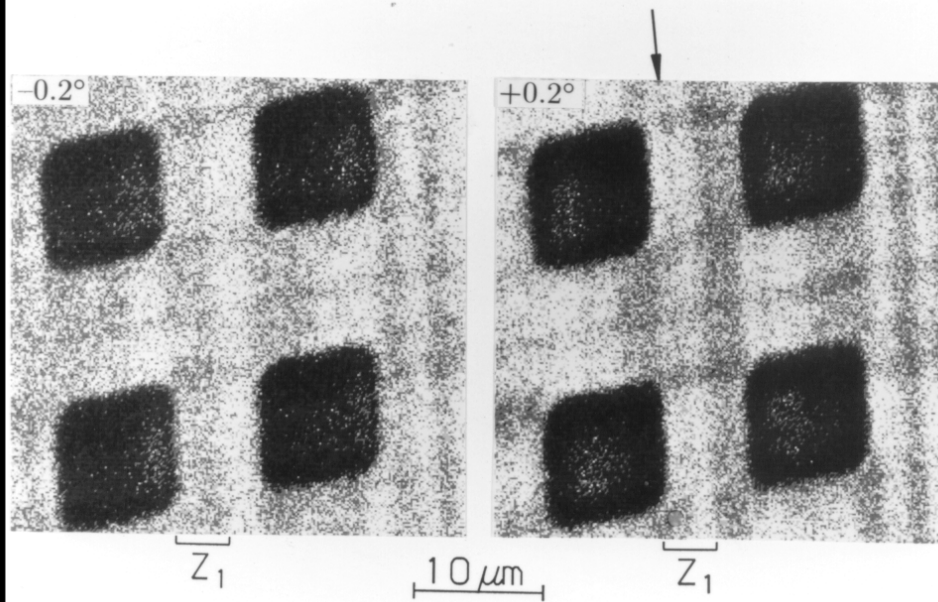


screw component

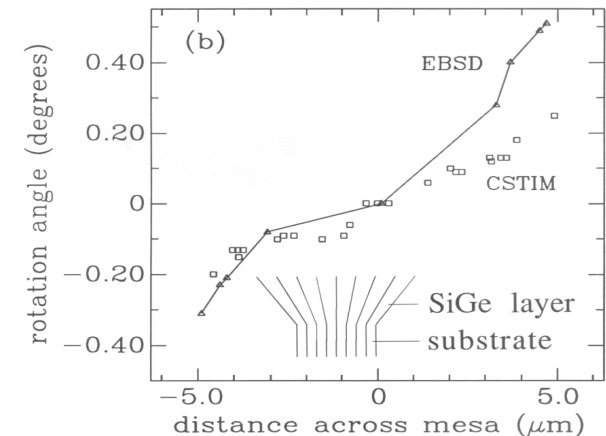
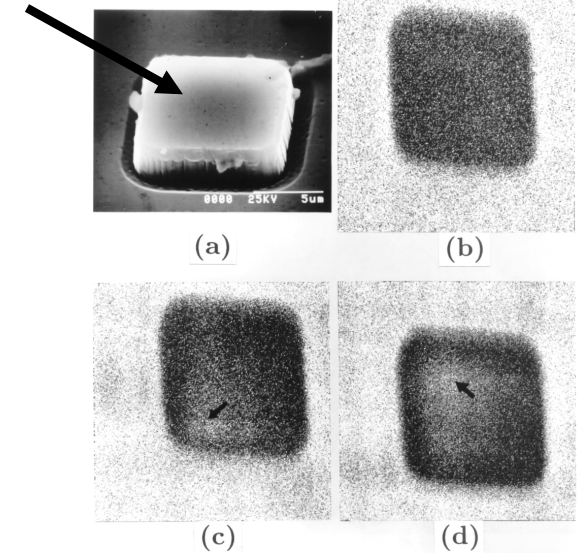


Misfit dislocations, SiGe Mesas

Patterned Si substrate with $\text{Si}_{0.85}\text{Ge}_{0.15}$ layer

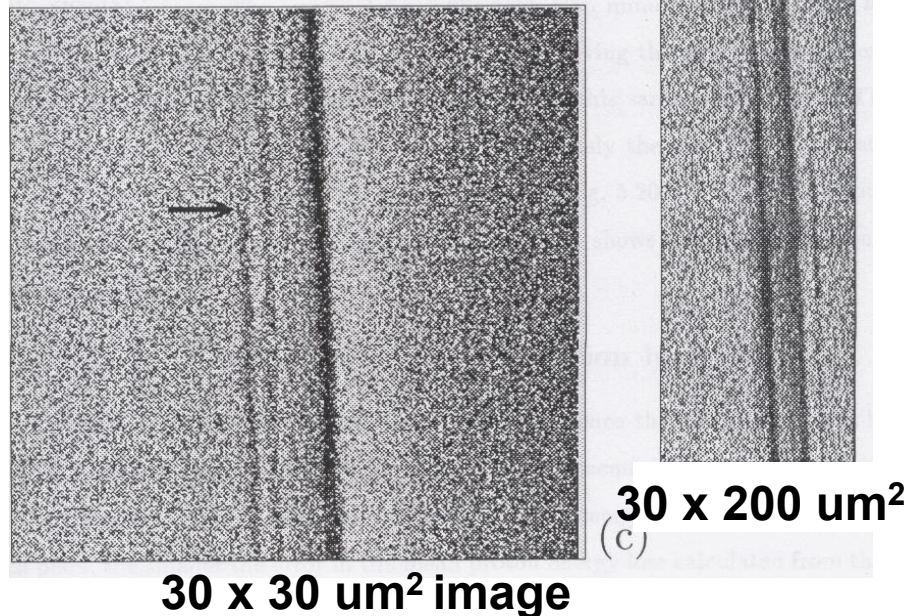
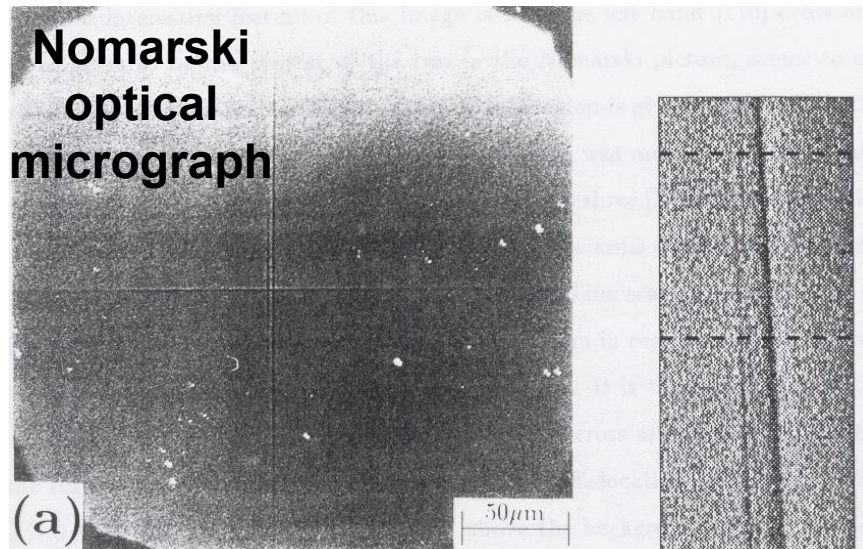


Elastic Strain, SiGe Mesas
No dislocations ?



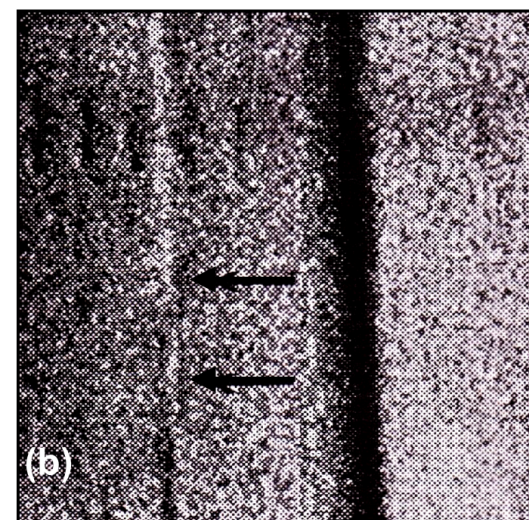
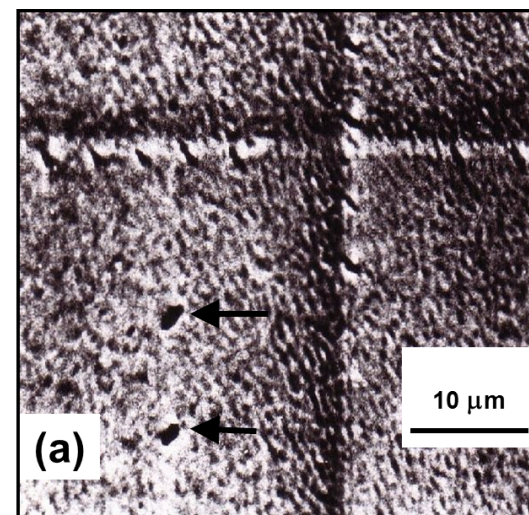
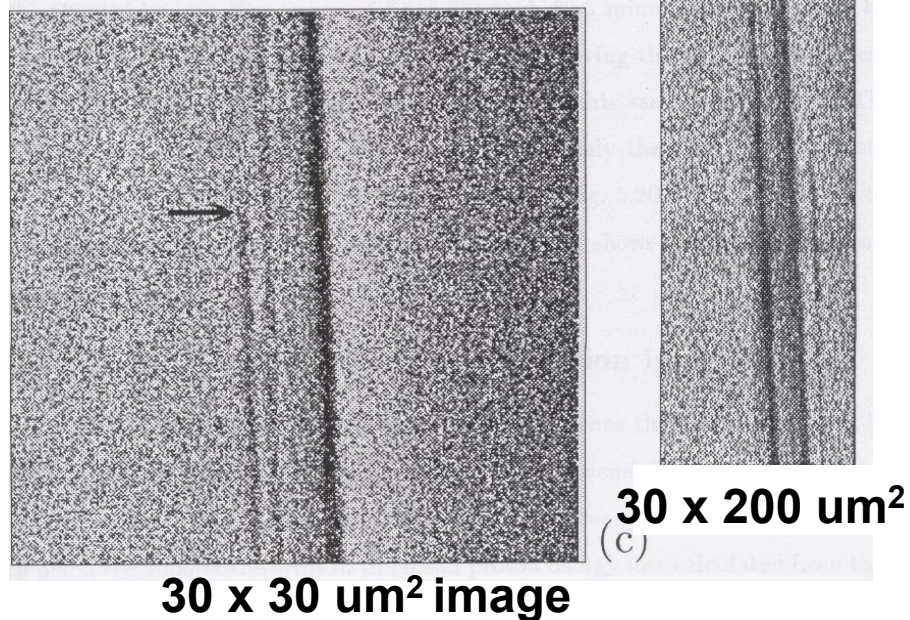
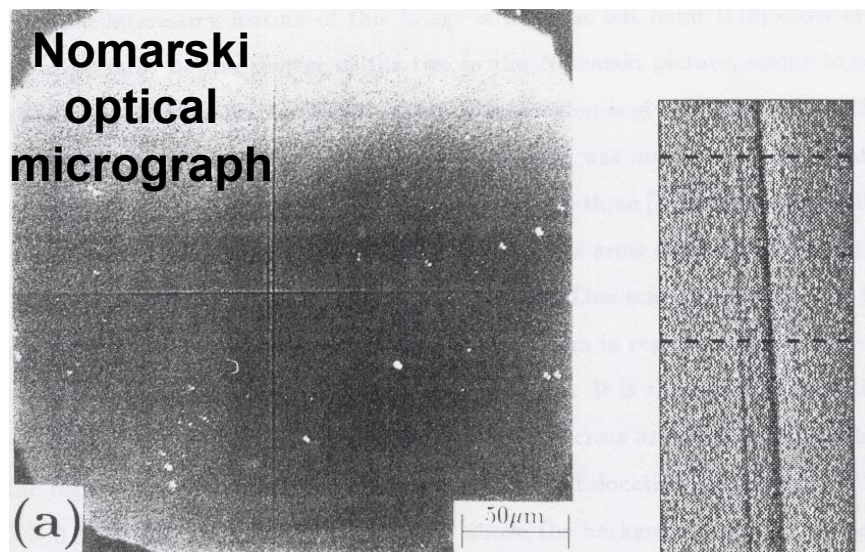
Variations of Elastic strain

Single 60° Dislocations



- Slightly relaxed 5% Ge 1.8 μm epilayer
- Misfits nucleate and spread out along orthogonal $\{110\}$ directions, forming a “cross” structure
- Transmission channelling images show more dislocation lines than Nomarski.

Single 60° Dislocations



**Changing Burgers vector
along dislocation line**

Part II:

Channeling Patterns with Ultra-thin silicon membranes

- Axial Channeling in silicon membranes
- Planar channeling in $\{111\}$ planes
- Superfocusing
- Angular distributions through thin crystals
- Proof of superfocusing effect

Thin membranes: old method

Review of Scientific Instruments / Volume 51 / Issue 9

Rev. Sci. Instrum. **51**, 1212 (1980); <http://dx.doi.org.libproxy1.nus.edu.sg/10.1063/1.1136404>
(5 pages)

Preparation of large-area monocrystalline silicon thin windows

N. W. Cheung

California Institute of Technology, Pasadena, California 91125

Abstract

References (15)

Citing Articles (29)

Related Content

A procedure suitable for laboratory practice is described to prepare large-area self-supporting silicon thin crystals with (100), (110) and (111) orientations. Windows up to 2 cm in diameter and 2000 Å in thickness can be produced. Variation in the thickness across the sample can be as low as ± 60 Å as measured by $^4\text{He}^+$ backscattering spectrometry.

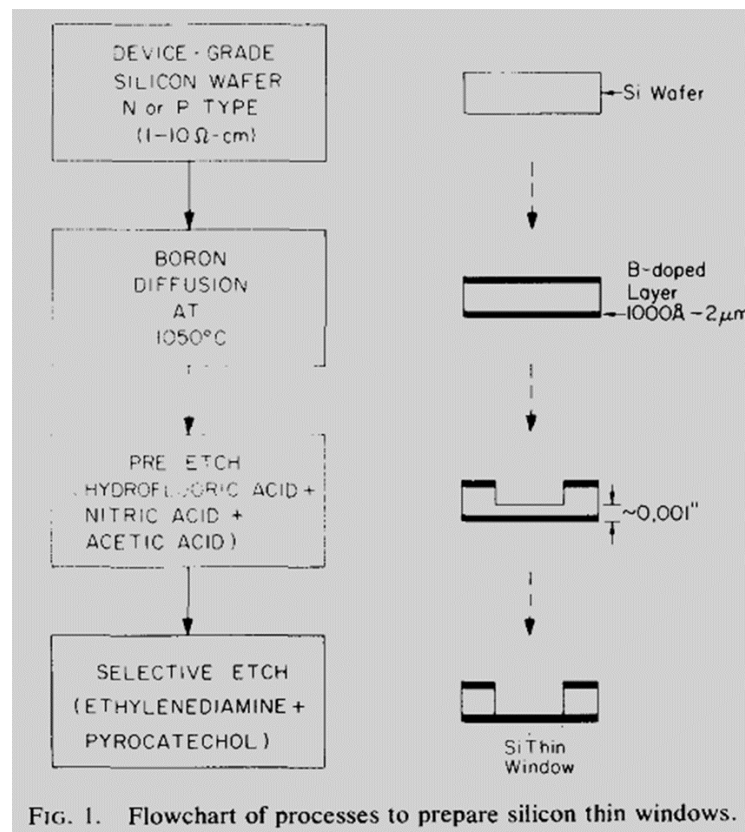


FIG. 1. Flowchart of processes to prepare silicon thin windows.

Ultra-thin membranes

APPLIED PHYSICS LETTERS **99**, 223105 (2011)

Fabrication of large-area ultra-thin single crystal silicon membranes

Z. Y. Dang,¹ M. Motapothula,¹ Y. S. Ow,¹ T. Venkatesan,² M. B. H. Breese,^{1,3,a)} M. A. Rana,⁴ and A. Osman⁵

¹*Center for Ion Beam Applications, Physics Department, National University of Singapore, Lower Kent Ridge Road, Singapore 117542, Singapore*

²*NanoCore, National University of Singapore, Singapore 117576, Singapore*

³*Singapore Synchrotron Light Source (SSLS), National University of Singapore, 5 Research Link, Singapore 117603, Singapore*

⁴*Physics Division, Directorate of Science, PINSTECH, P.O. Nilore, Islamabad, Pakistan*

⁵*National Centre for Physics (NCP), Shahdara Valley Road, Islamabad, Pakistan*

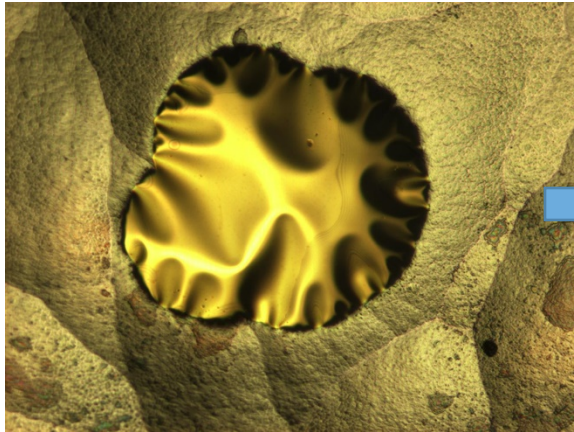
(Received 3 November 2011; accepted 14 November 2011; published online 1 December 2011)

Perfectly, crystalline, 55 nm thick silicon membranes have been fabricated over several square millimeters and used to observe transmission ion channeling patterns showing the early evolution of the axially channeled beam angular distribution for small tilts away from the [011] axis. The reduced multiple scattering through such thin layers allows fine angular structure produced by the highly non-equilibrium transverse momentum distribution of the channeled beam during its initial propagation in the crystal to be resolved. The membrane crystallinity and flatness were measured by using proton channeling measurements and the surface roughness of 0.4 nm using atomic force microscopy. © 2011 American Institute of Physics. [doi:[10.1063/1.3665620](https://doi.org/10.1063/1.3665620)]

Ultra-thin membranes

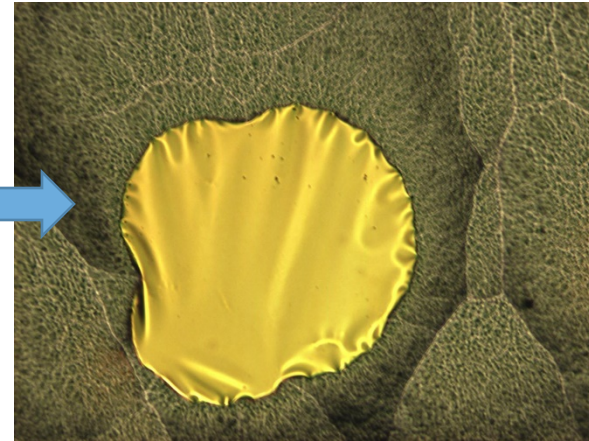
Optical Micrographs

145 nm SiO₂ on 55 nm Si



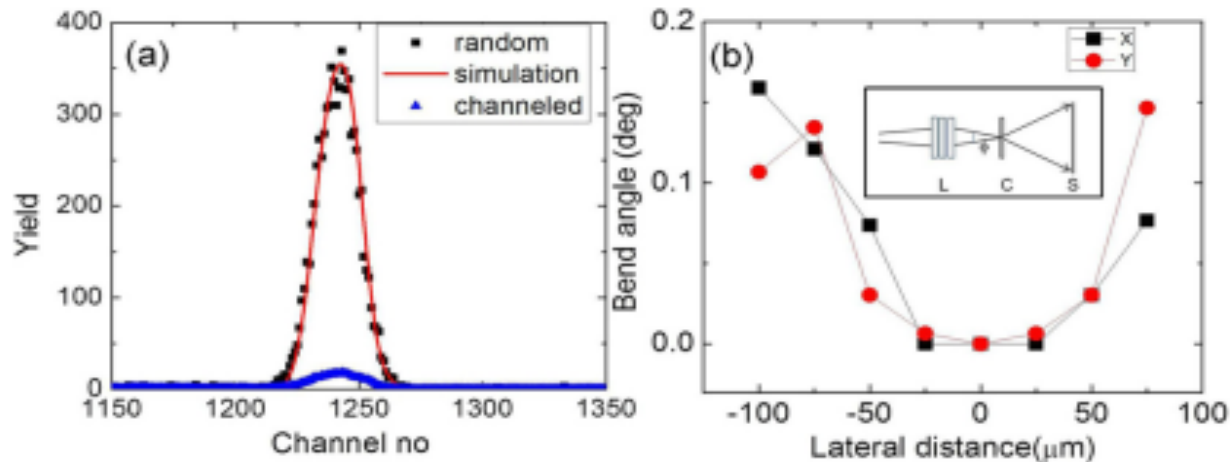
(500 μm)

55nm Si



(500 μm)

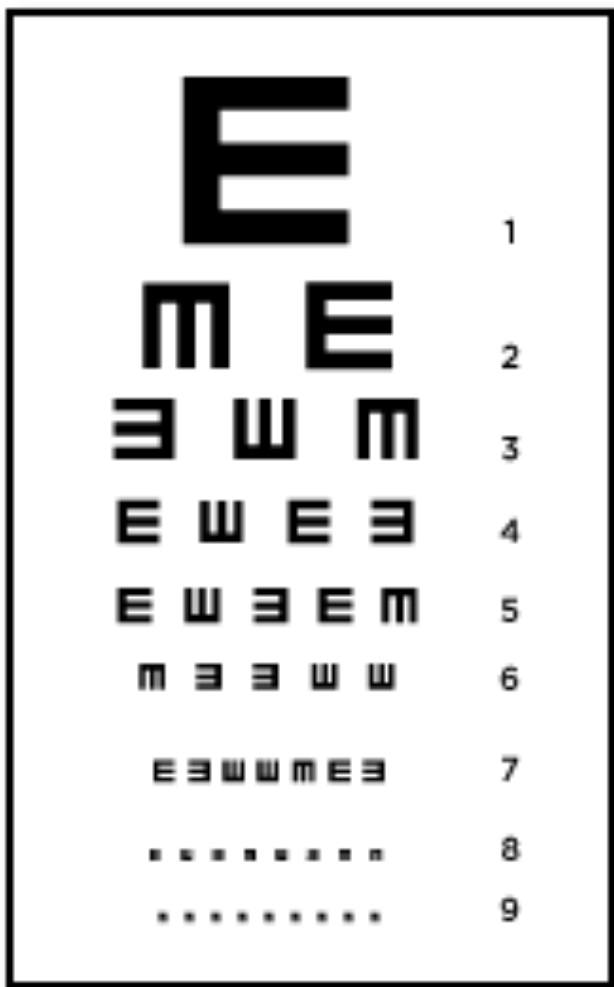
Membrane is flatter after removing SiO₂



- Makes use of enormous efforts in SOI technology
- Membranes are not flat, but of very high quality
- Curvature not a problem when using a microprobe

Thick crystals Feel like....





Thick vs. Thin Crystals

Rosner et. al, Phys
 Rev B 18 1978 1066

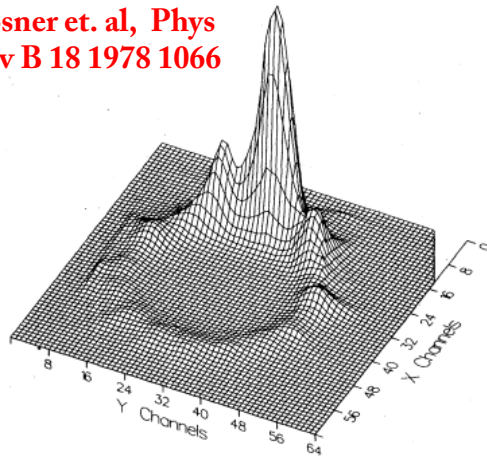
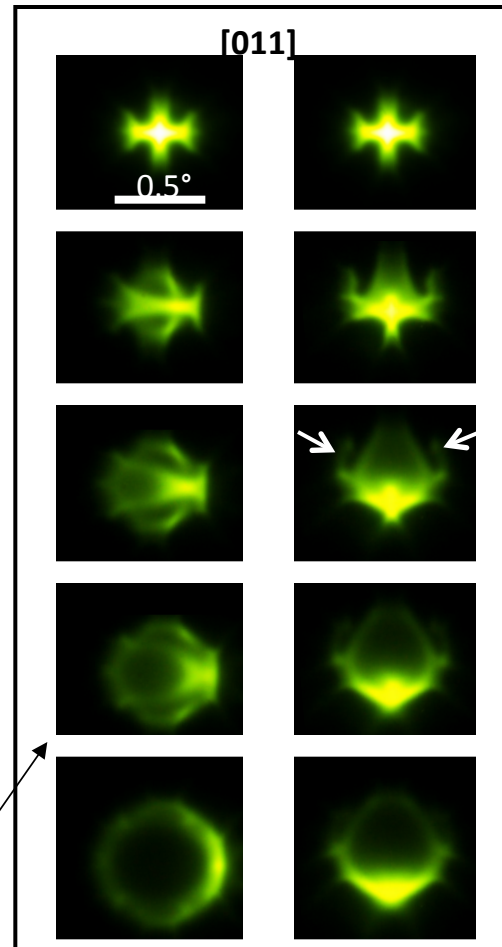


FIG. 5. (a) Doughnut formed with $\psi = 0.327^\circ$ from
 $\langle 110 \rangle$ axis. Crystal thickness, 1900 Å.

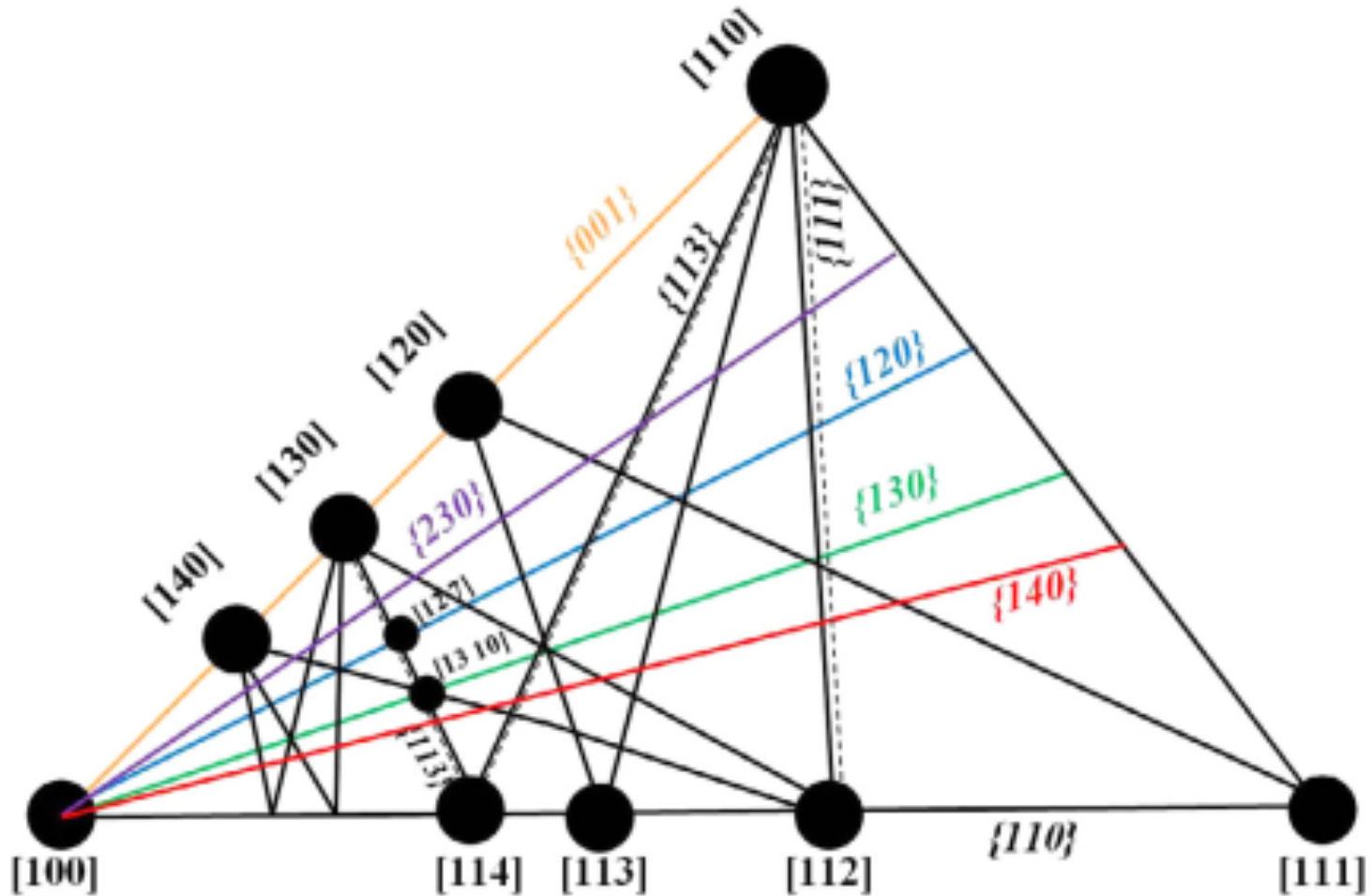
2D PSD, 100 ions/sec.
 Long data collection

Fluorescent screen.
 Recorded in 100 ms



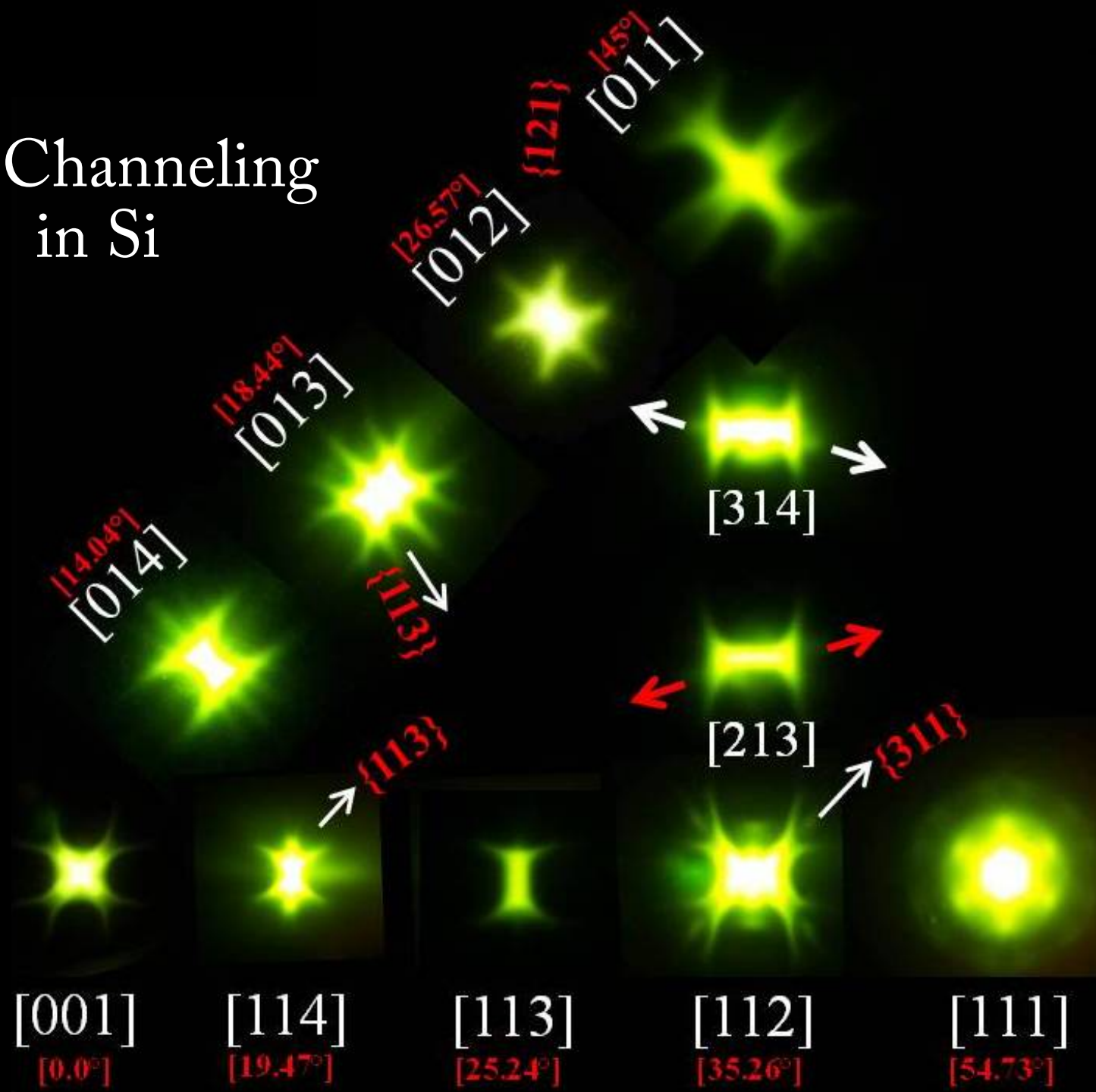
2 MeV protons, 55 nm
 [001] Si membrane,

Axial Channeling in silicon

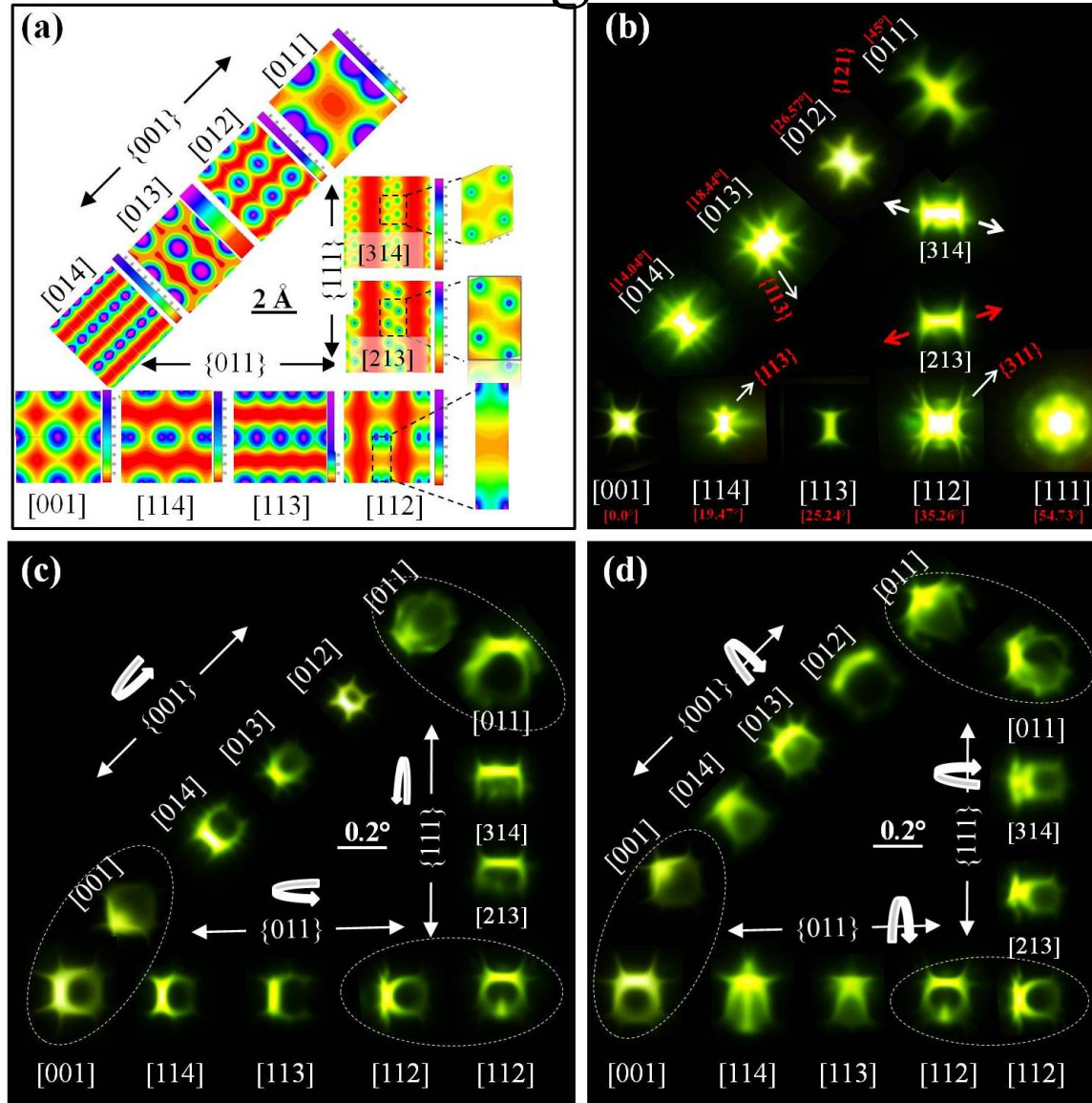


Stereographic projection of axes and planes in Si lattice along $\langle 001 \rangle$, showing the major and minor axes and planes considered

Axial Channeling in Si



Axial Channeling in Si



2 MeV protons, 55 nm
[001] Si membrane,

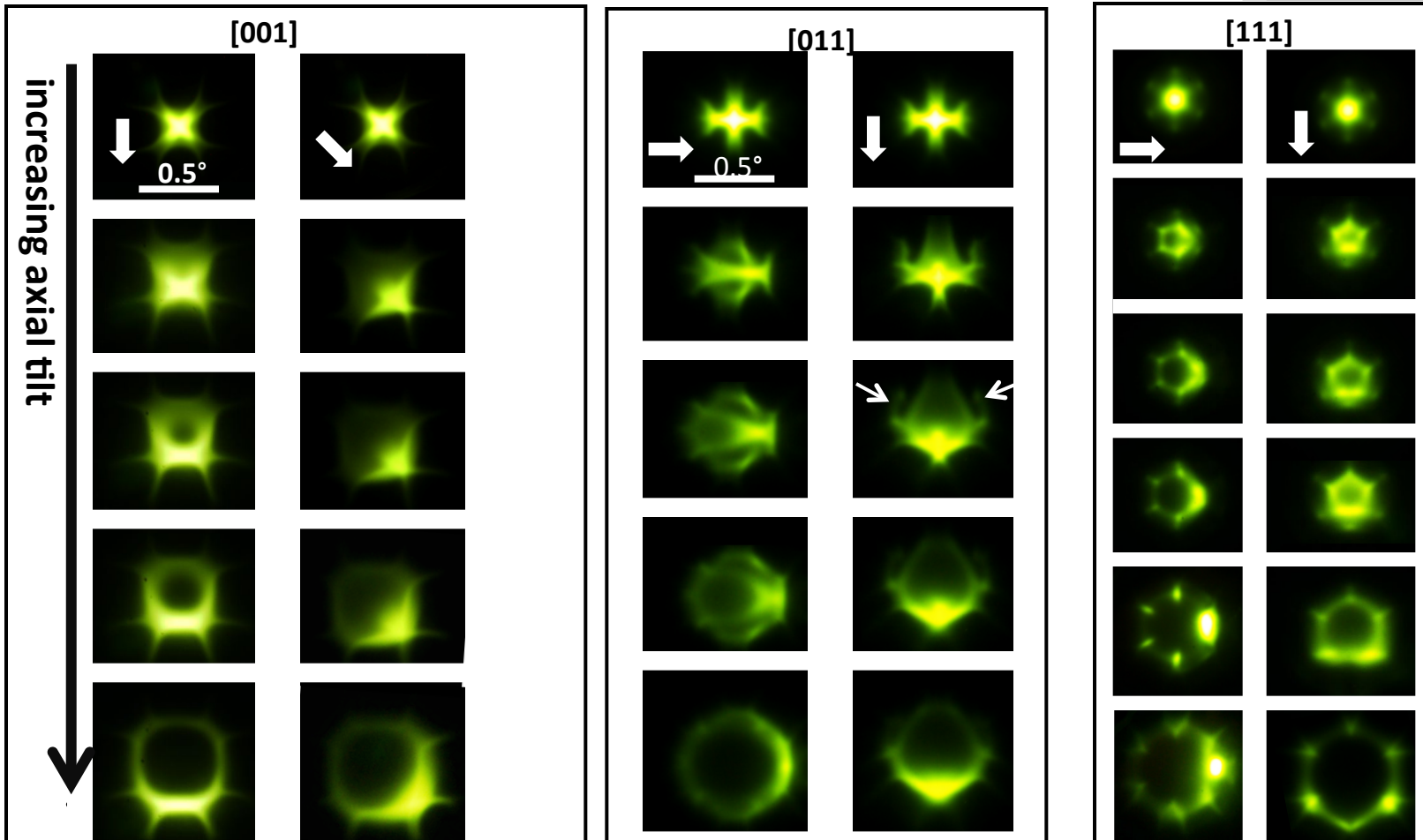
(a) Static atomic potentials at axes away from the [100] axis, along (001), (011), (111) planar directions.

(b) Experimental channeled angular distributions at aligned case

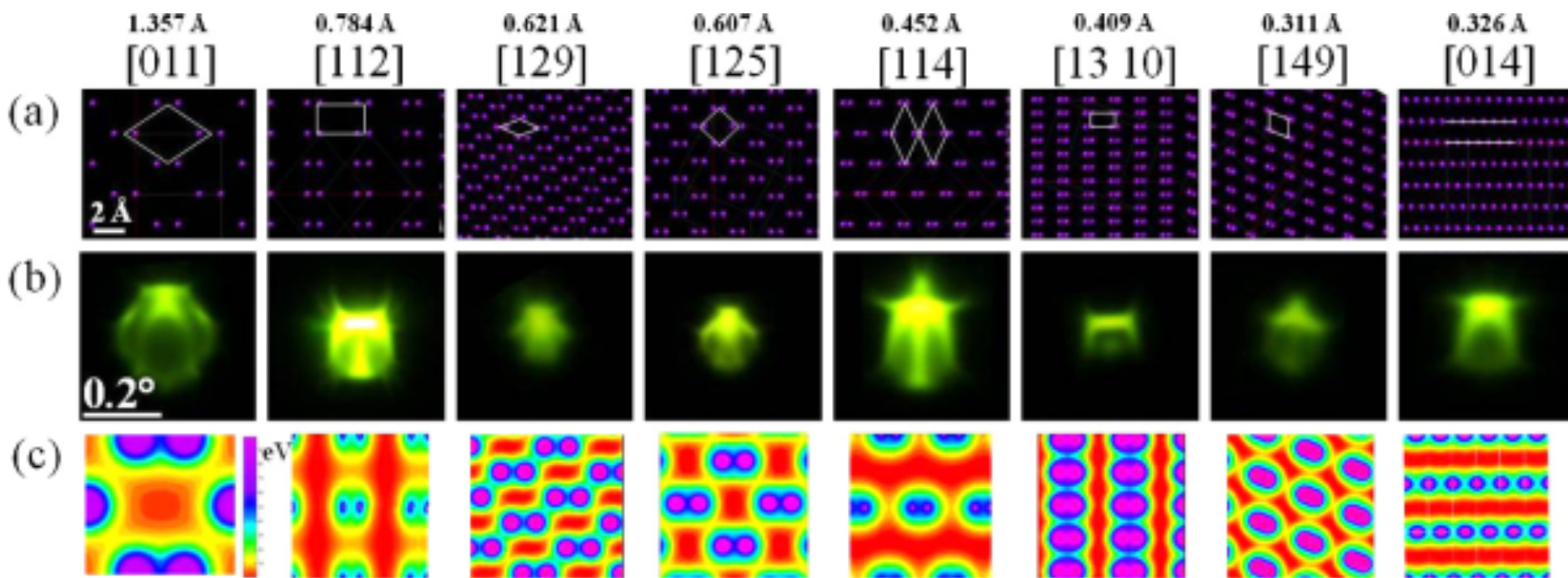
Doughnut channeling patterns for (c) tilting along the planes and (d) perpendicular to the planes.

Axial Channeling: small tilts

2 MeV protons, 55 nm
[001] Si membrane,



Axial Channeling in Si



Compendium of channeling patterns when tilting the crystal perpendicular to close-spaced atoms, in order of decreasing spacing between the two atoms.

Channeling in wide and narrow {111} planes

PRL **108**, 195502 (2012)

PHYSICAL REVIEW LETTERS

week ending
11 MAY 2012

Influence of the Narrow {111} Planes on Axial and Planar Ion Channeling

M. Motapothula,^{1,2} Z. Y. Dang,¹ T. Venkatesan,² M. B. H. Breese,^{1,3,*,†} M. A. Rana,⁴ and A. Osman⁵

¹*Center for Ion Beam Applications, Physics Department, National University of Singapore,
Lower Kent Ridge Road, Singapore 117542*

²*NUSNNI, National University of Singapore, Singapore 117576*

³*Singapore Synchrotron Light Source (SSLS), National University of Singapore, 5 Research Link, Singapore 117603*

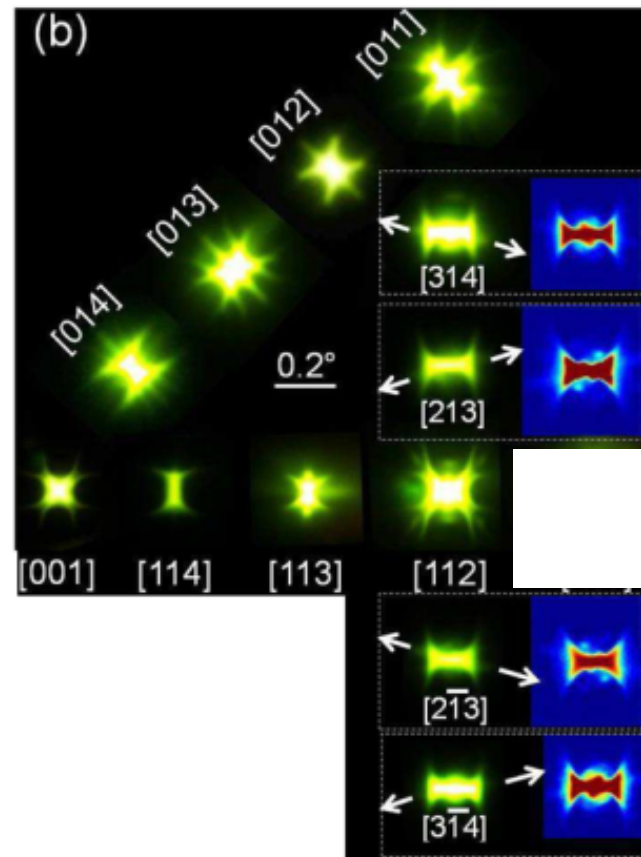
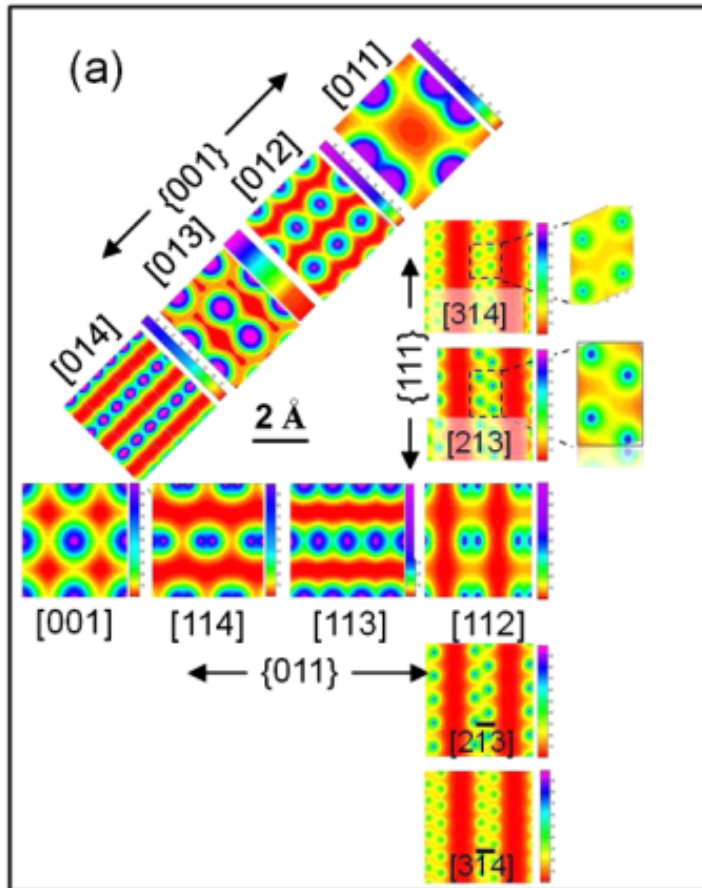
⁴*Physics Division, Directorate of Science, PINSTECH, P. O. Nilore, Islamabad, Pakistan*

⁵*National Centre for Physics (NCP), Shahdara Valley Road, Islamabad, Pakistan*

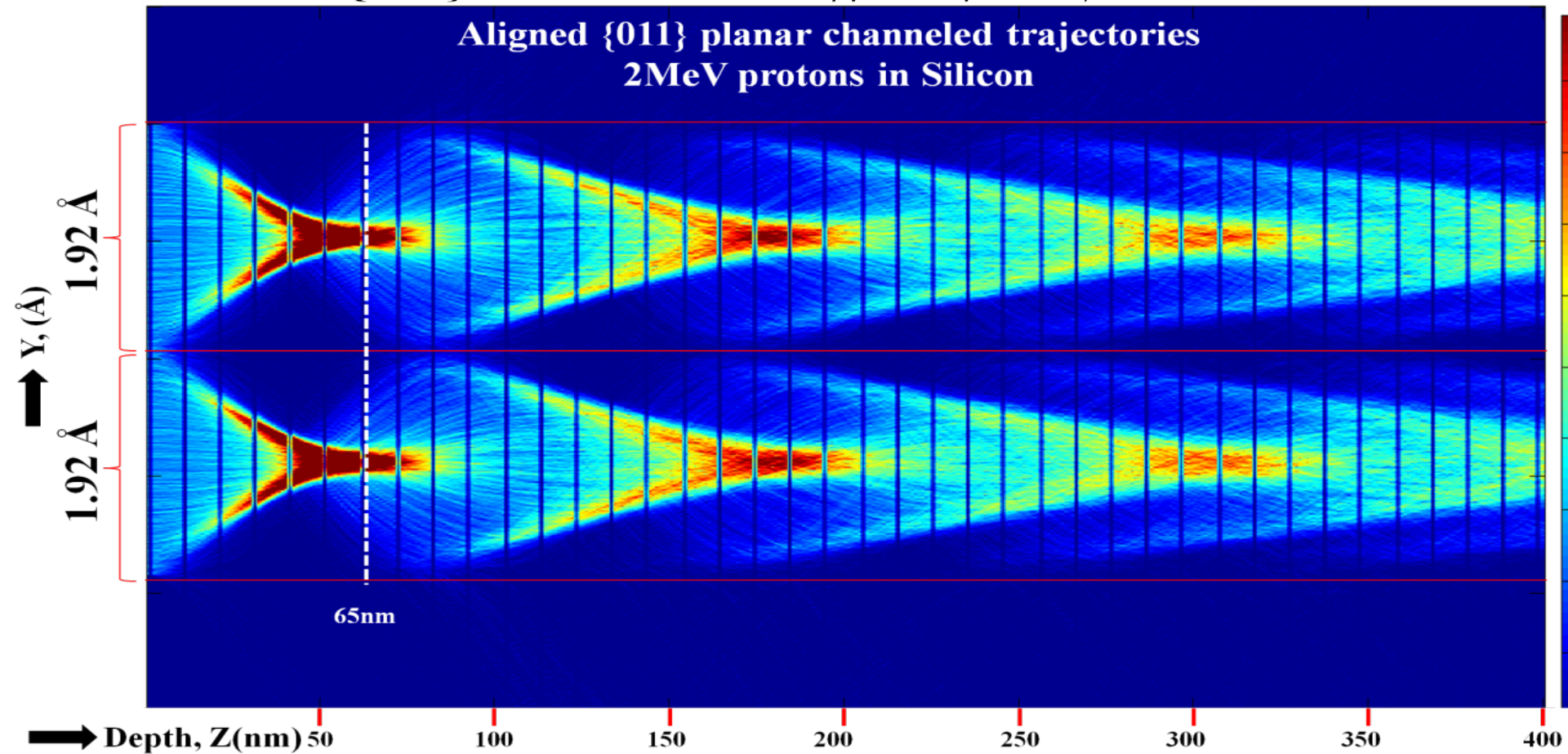
(Received 16 January 2012; published 7 May 2012)

We report channeling patterns where clearly resolved effects of the narrow {111} planes are observed in axial and planar alignments for 2 MeV protons passing through a 55 nm [001] silicon membrane. At certain axes, such as $\langle 213 \rangle$ and $\langle 314 \rangle$, the offset in atomic rows forming the narrow {111} planes results in shielding from the large potential at the wide {111} planes, producing a region of shallow, asymmetric potential from which axial channeling patterns have no plane of symmetry. At small tilts from such axes, different behavior is observed from the wide and narrow {111} planes. At planar alignment, distinctive channeling effects due to the narrow planes are observed. As a consequence of the shallow potential well at the narrow planes, incident protons suffer dechanneled trajectories which are excluded from channeling within the wide planes, resulting in an anomalously large scattered beam at {111} alignment.

$\{111\}$ planar channeling

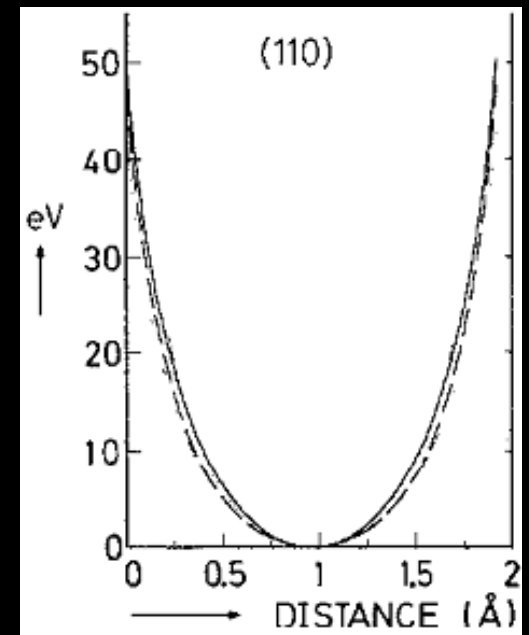
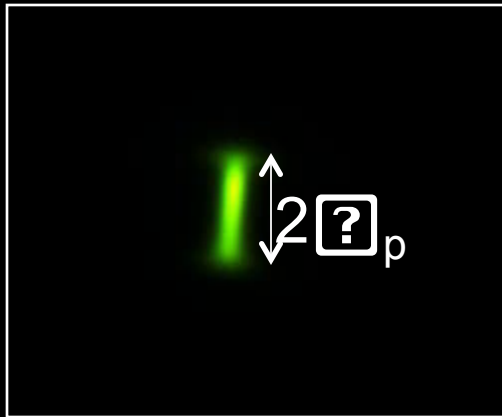


{110} Planar channeling: Trajectory Plots

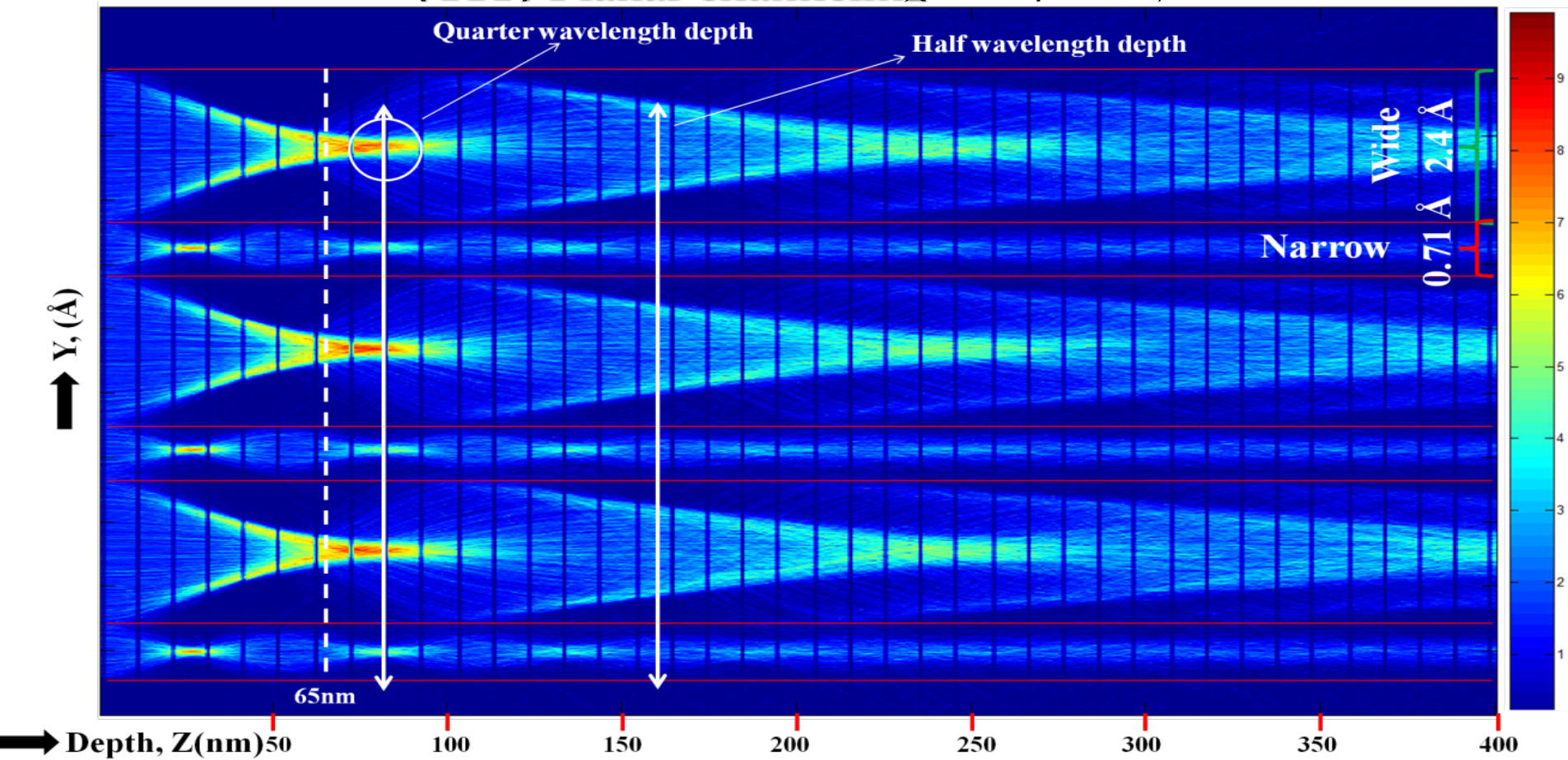


{110} Planar channeling

2MeV Protons; 55nm thick [001] Silicon

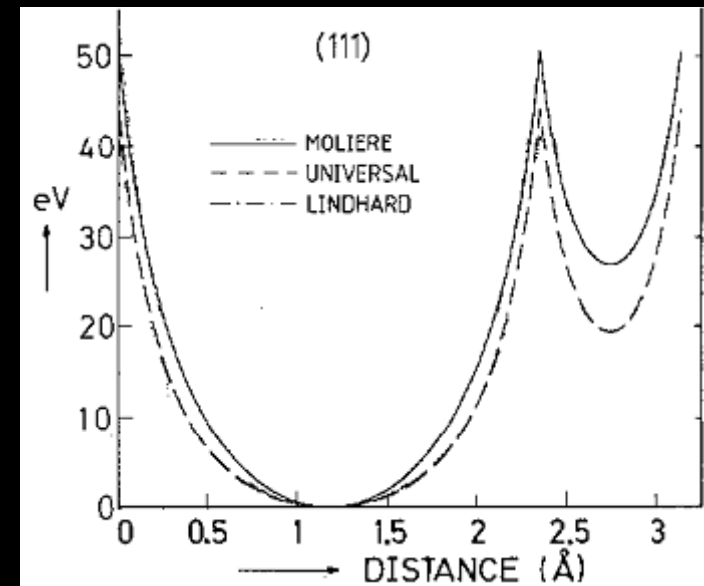


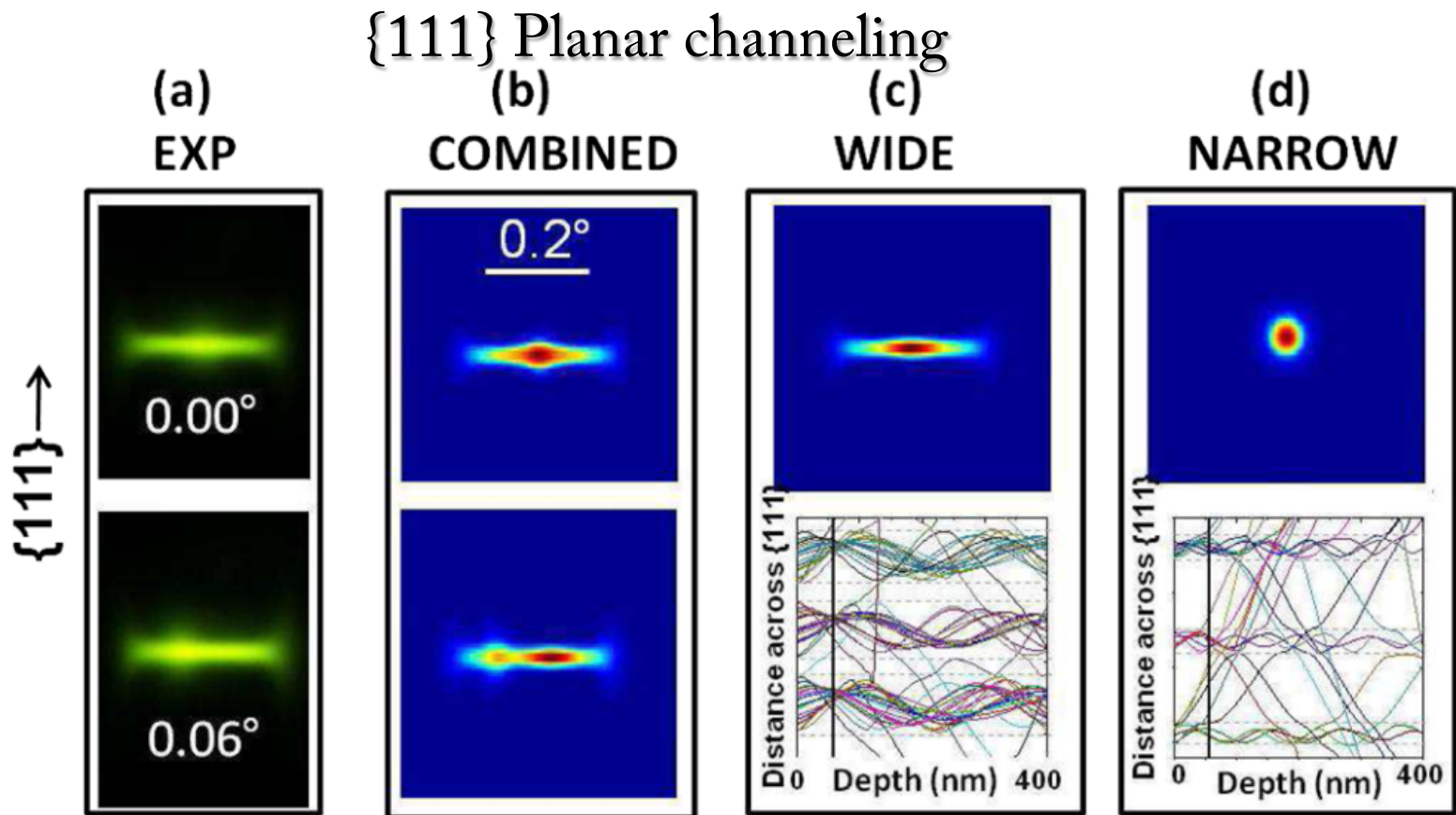
{111} Planar channeling: Trajectory Plots



{111} Planar channeling

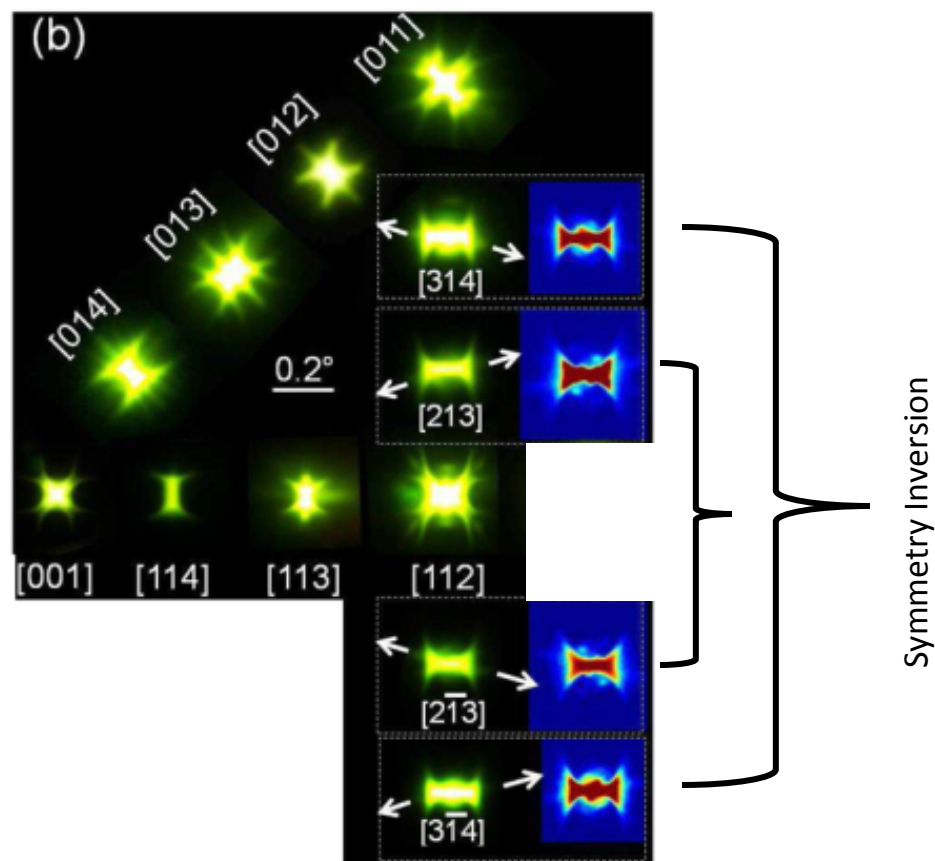
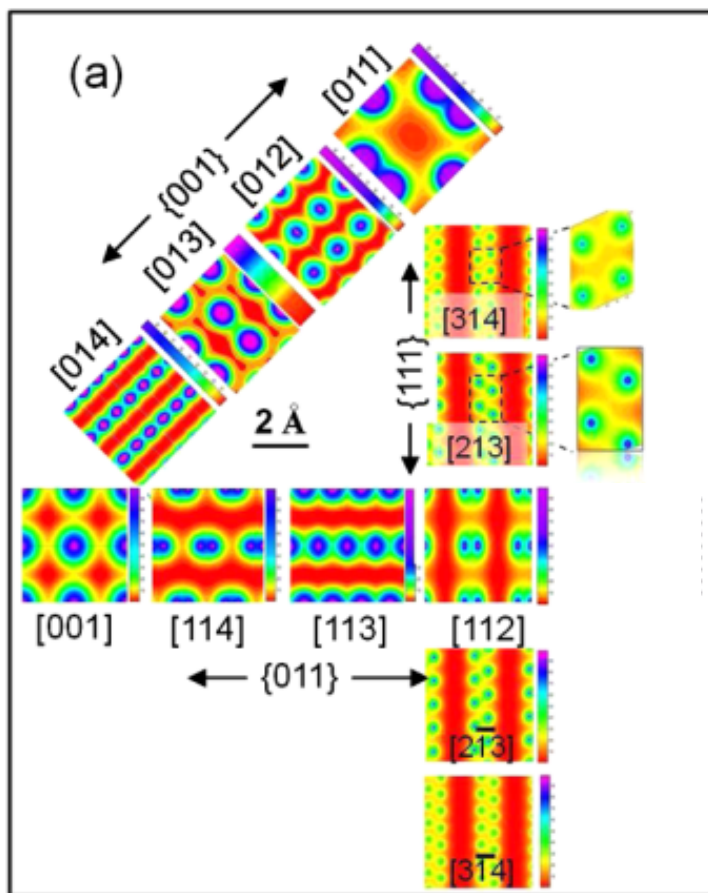
2MeV Protons; 55nm thick [001] Silicon



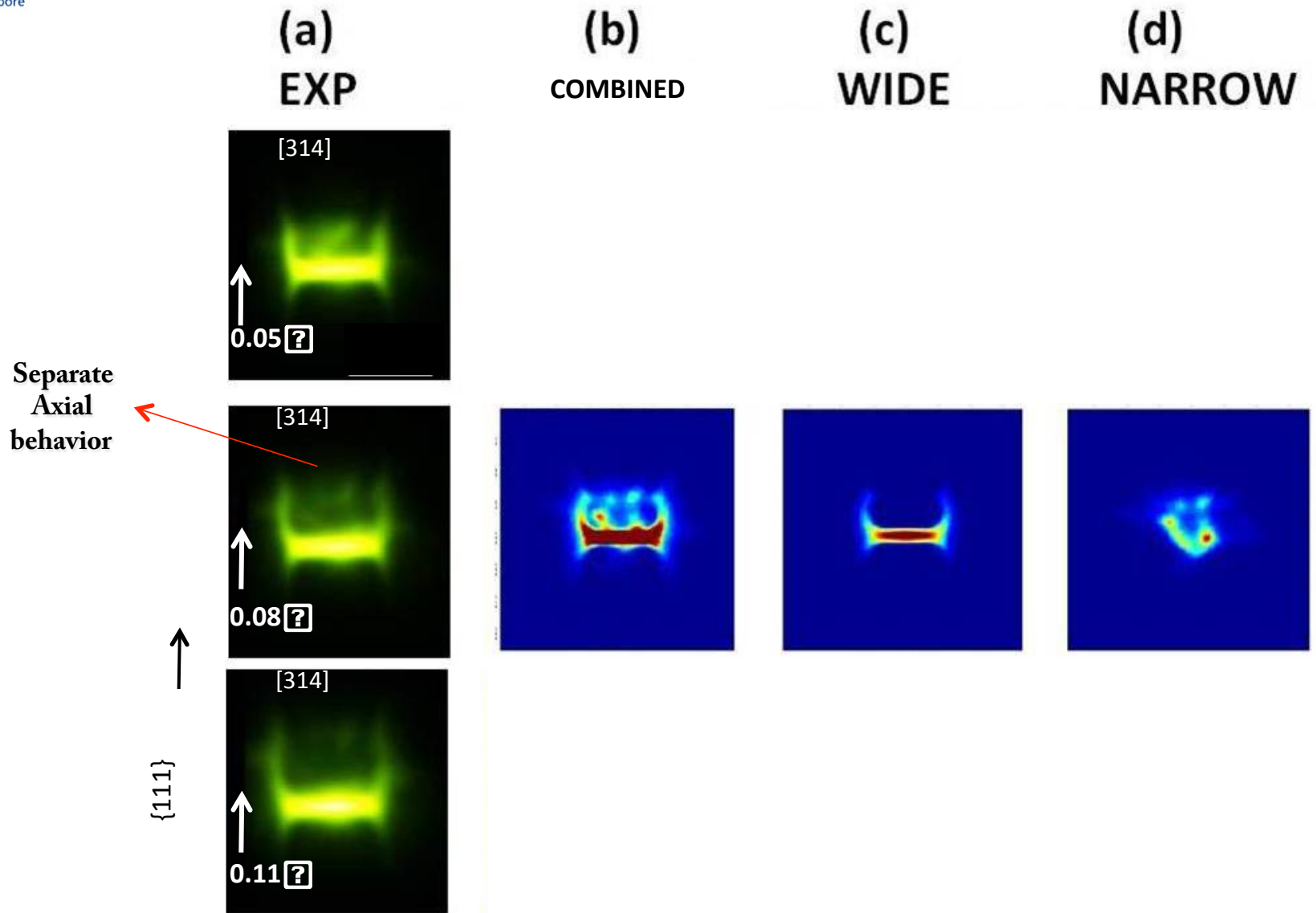


- (a) Experimental channeling patterns for 2 MeV protons transmitted at tilts of 0.00° and 0.06° .
- (b) Simulated FLUX channeling patterns for the angular distribution in the combined planes
- (c,d) in the wide / narrow planes, showing angular distributions at a tilt of 0.00° and channeling trajectories for a tilt of 0.06° over three adjacent wide or narrow planes. A depth of 55 nm is indicated by a line

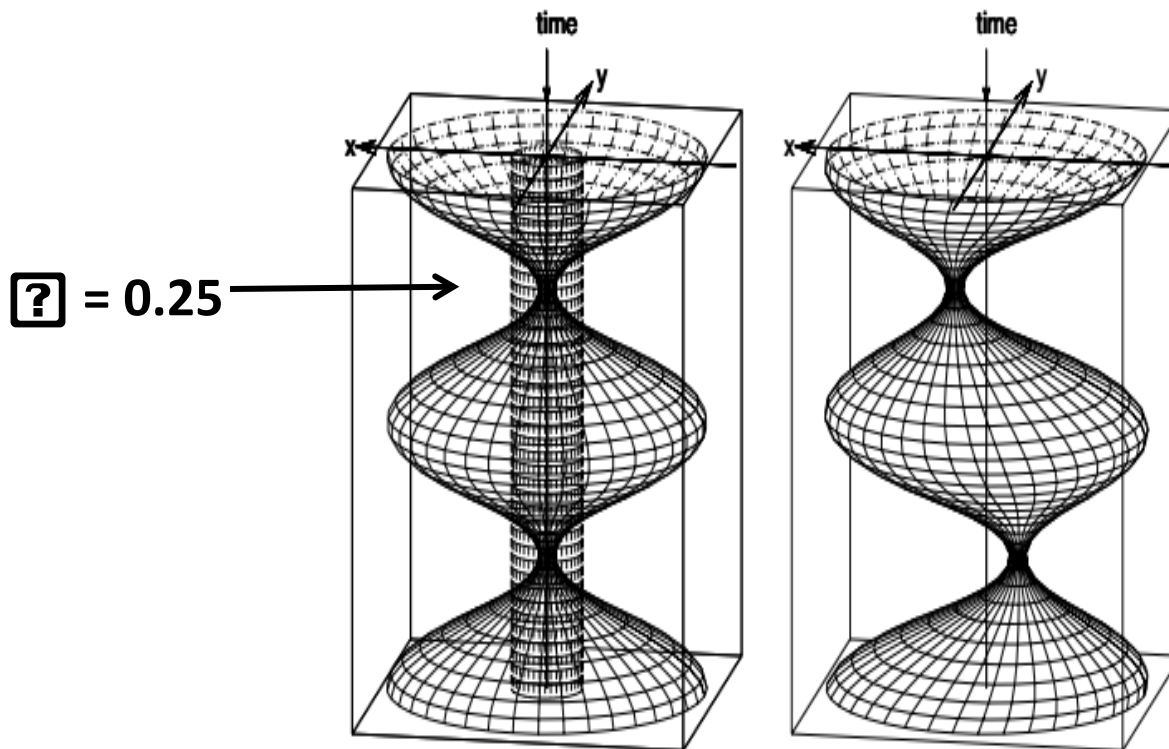
$\{111\}$ planar channeling: $[213]$ and $[314]$ axes



Doughnuts at $[314]$ axes



Superfocusing

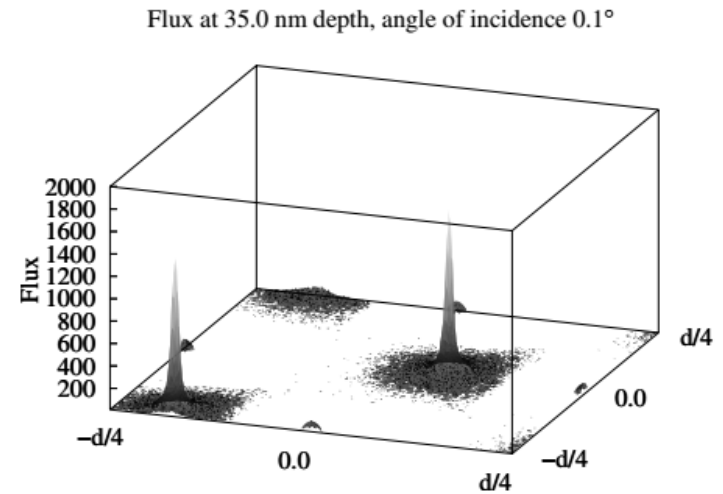
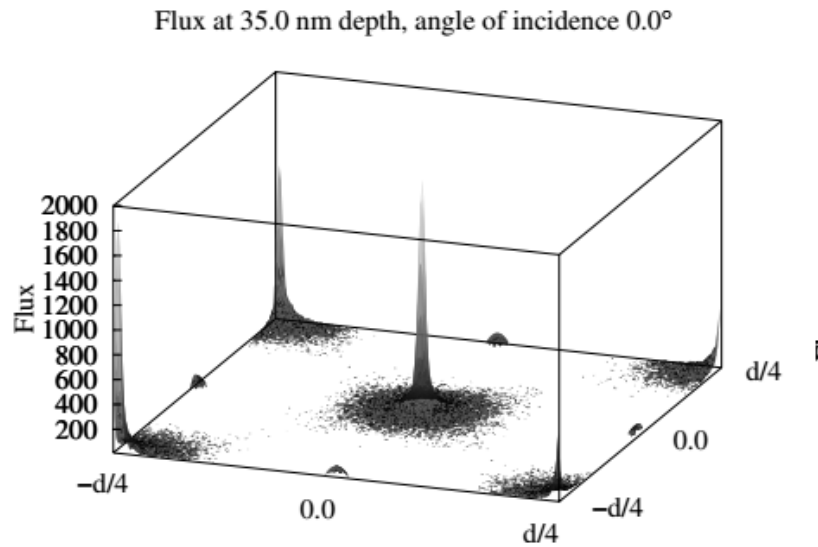


Yu.N. Demkov and J.D. Meyer

THE EUROPEAN PHYSICAL JOURNAL B B 42, 361–365 (2004)

A sub-atomic microscope, superfocusing in channeling and close encounter atomic and nuclear reactions

Superfocusing



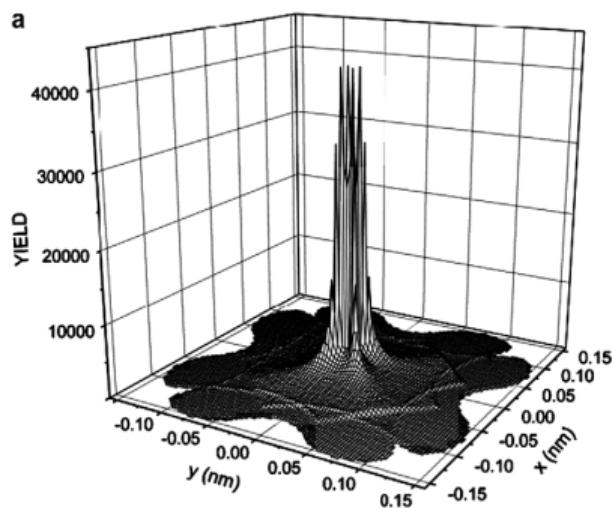
The same sharpness of the focus is observed, whereas the point of focus has moved by ≈ 0.05 nm out of the center.

Yu.N. Demkov and J.D. Meyer

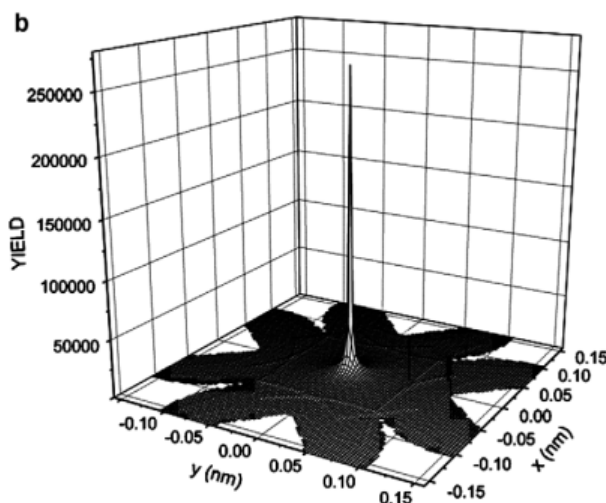
THE EUROPEAN PHYSICAL JOURNAL B B 42, 361–365 (2004)

A sub-atomic microscope, superfocusing in channeling and close encounter atomic and nuclear reactions

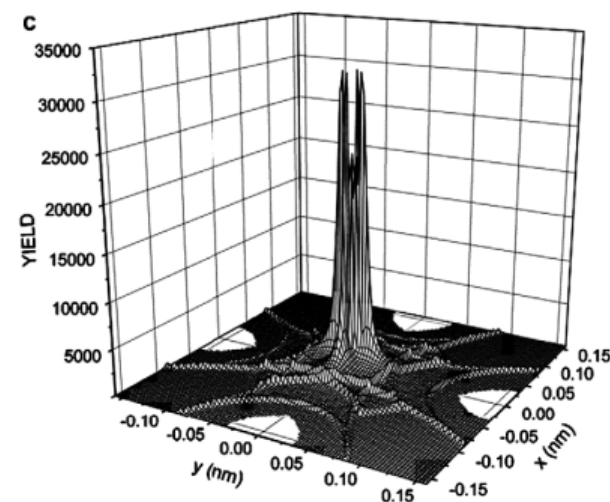
Superfocusing



(a) $\boxed{?} = 0.20$



(b) $\boxed{?} = 0.25$

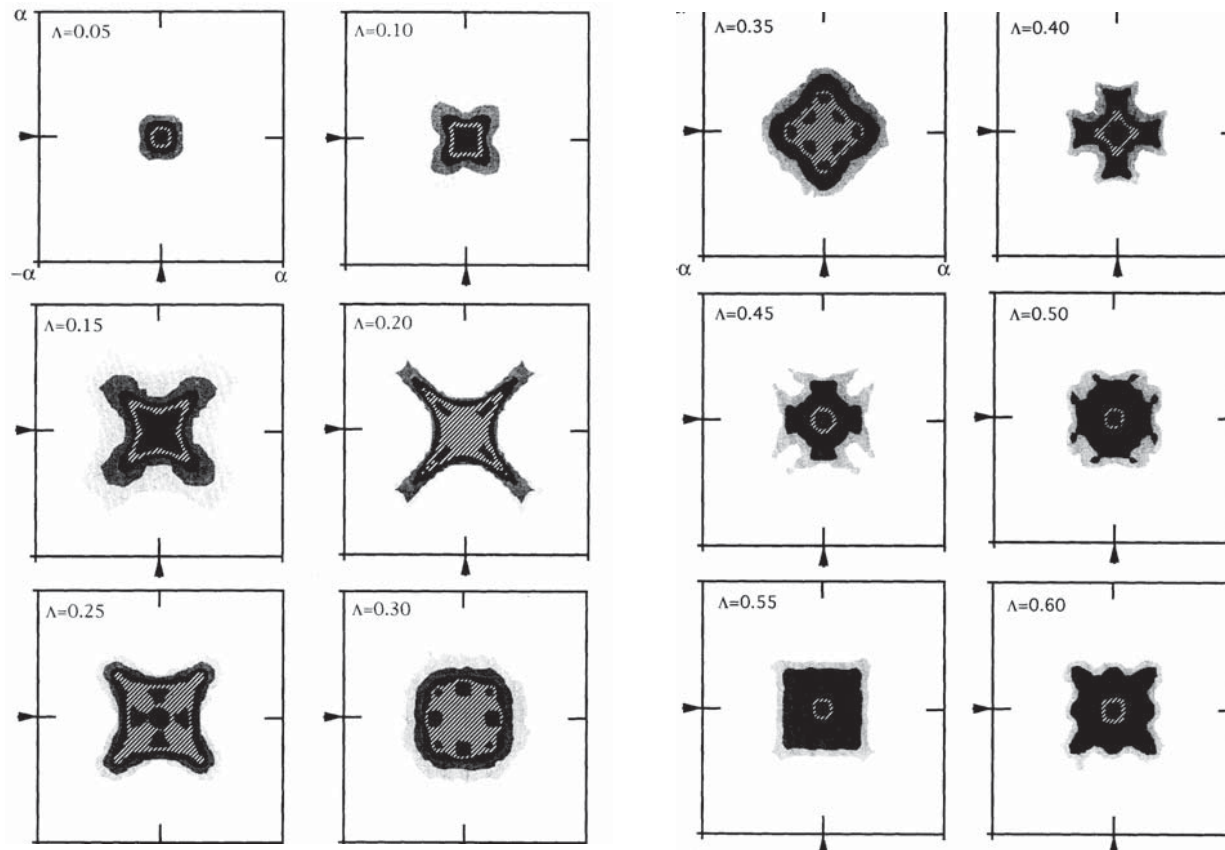


(c) $\boxed{?} = 0.30$

Spatial distributions of 2 MeV protons in a $\langle 100 \rangle$ channel of a Si crystal

N. Neškovic, S. Petrovic, D. Borka, NIM B 267 (2009) 2616–2620
Superfocusing of channeled protons and crystal rainbows

Angular distributions through thin crystals



H. F. Krause et al, Phys. Rev. A 49 283 (1994)

Angular distribution of ions axially channeled in a very thin crystal: Experimental and theoretical results

Angular Distributions through thin crystals

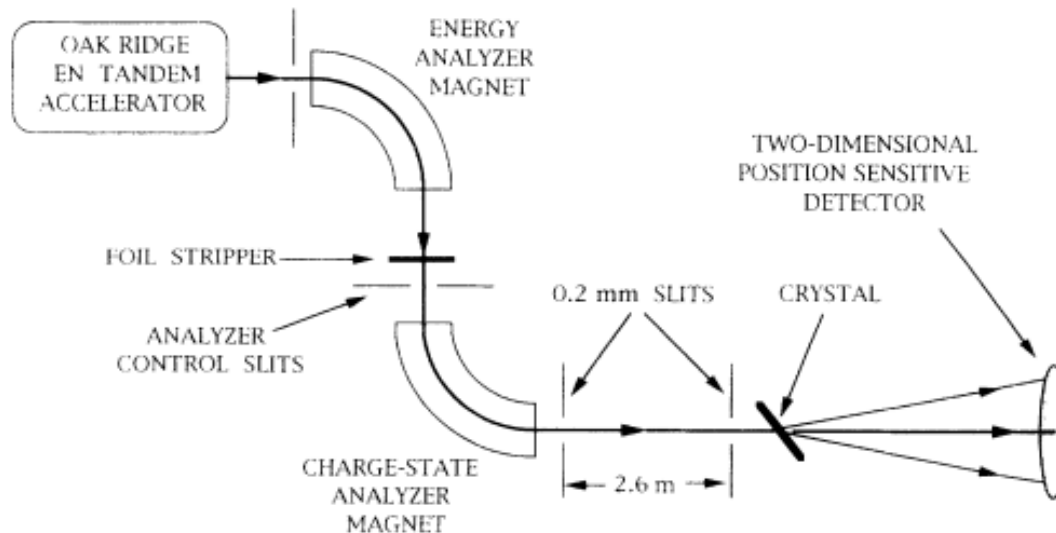


FIG. 1. Sketch of the experimental arrangement. A thin carbon foil, a calibration mask, and removable ZnS phosphors, situated between the crystal and the detector, that were used in the angular calibration and during crystal alignment, are not shown.

H. F. Krause et al, Phys. Rev. A 49 283 (1994)

Angular distribution of ions axially channeled in a very thin crystal: Experimental and theoretical results

Angular Distributions through thin crystals

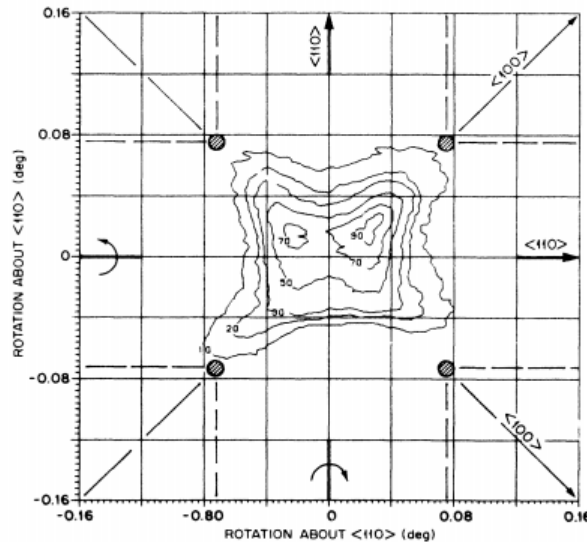


FIG. 6. The experimental angular distribution for 7-MeV protons axially channeled along the $\langle 100 \rangle$ direction of silicon where the crystal is 1400 Å thick (atomic string length, 260 atoms). Intensity cuts through the distribution at a fraction of the maximum count (percentage) are plotted as contours at the following levels: 10, 20, 30, 50, 70, 90. Atomic strings lying closest to the center of the channel are shown. The $\langle 100 \rangle$ and $\langle 110 \rangle$ axial directions are identified.

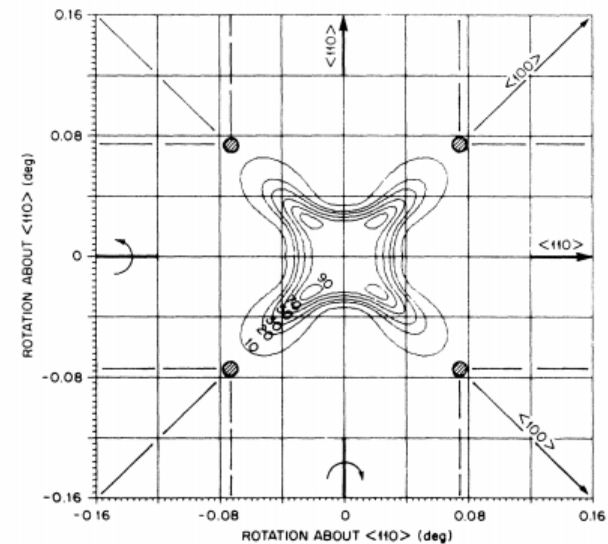


FIG. 7. The calculated angular distribution for 7-MeV protons axially channeled in the $\langle 100 \rangle$ direction of silicon. The Monte Carlo calculation assumed the Lindhard potential ($C=4.0$), measured angular resolution functions appropriate to the experiment [0.010° vertical; 0.015° horizontal (FWHM)], and the experimental crystal thickness. Cuts through the intensity distribution at a fraction of the peak intensity (percentage) are plotted as contours at the same levels as Fig. 6. The $\langle 100 \rangle$ and $\langle 110 \rangle$ axial directions are identified.

H. F. Krause et al, Phys. Rev. A 49 283 (1994)

Angular distribution of ions axially channeled in a very thin crystal: Experimental and theoretical results

Superfocusing

- No experimental evidence
- What is focused beam size ?
- What is flux enhancement ?

Angular Distributions through thin crystals

- Limited experimental evidence

Our Approach

- Compare FLUX simulations around $\pi/4$ with experimental patterns.
- If get good agreement with angular images then this supports the case for believing the spatial enhancement in the same simulations

PHYSICAL REVIEW B **94**, 075415 (2016)

Experimental evidence of the superfocusing effect for axially channeled MeV protons

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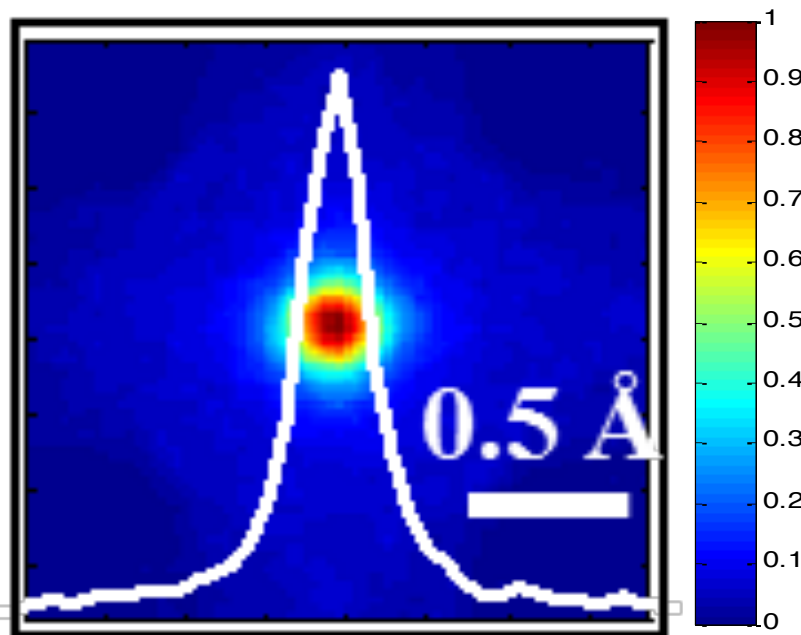
²*Laboratory of Physics, Vinča Institute of Nuclear Sciences, University of Belgrade, P.O. Box 522, 11001 Belgrade, Serbia*

(Received 16 February 2016; published 11 August 2016)

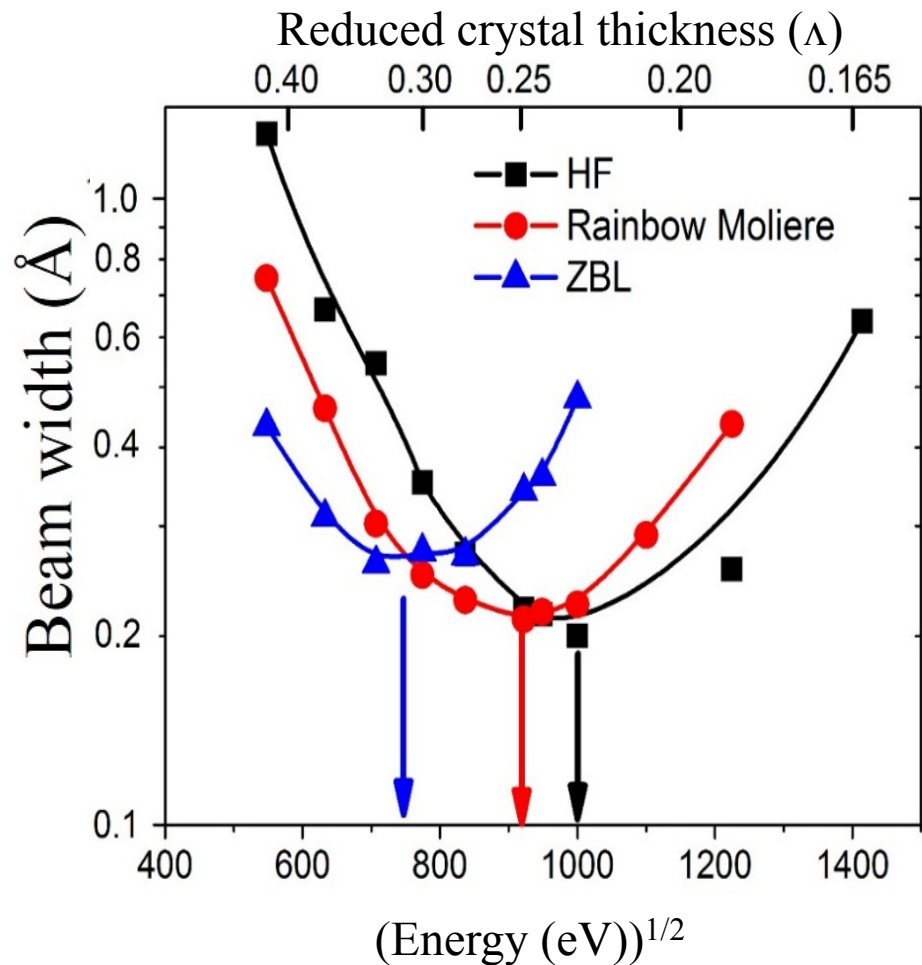
Sub-Ångström focusing of megaelectronvolt (MeV) ions within axial channels was predicted over 10 years ago, but evidence proved elusive. We present experimental angular distributions of axially channeled MeV protons in a 55-nm-thick (001) silicon membrane through which multiple scattering is negligible. Fine angular structure is in excellent agreement with Monte Carlo simulations based on three interaction potentials, providing indirect evidence of the existence of the superfocusing effect with flux enhancement of around 800 within a focused beam width of ~ 20 pm.

DOI: [10.1103/PhysRevB.94.075415](https://doi.org/10.1103/PhysRevB.94.075415)

FLUX Simulations



Overlap of simulated exit coordinate distribution and line profile under the superfocused condition for the Rainbow-Moliere potential at the exit face of a 55-nm-thick [001] Si layer.

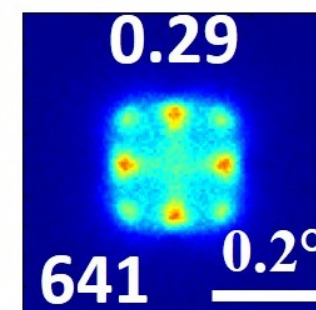
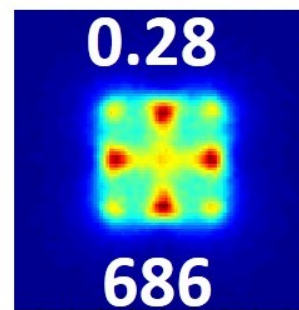
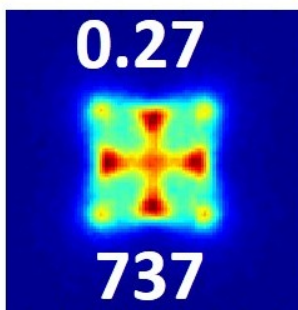
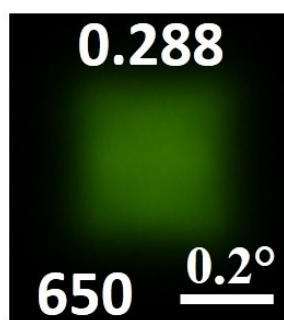
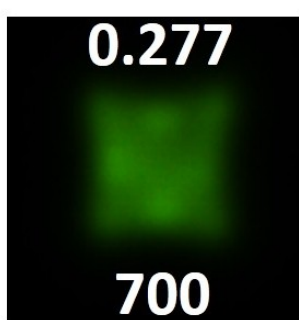
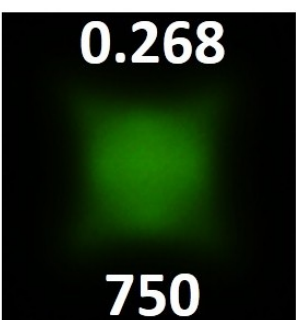
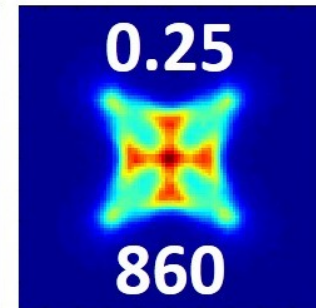
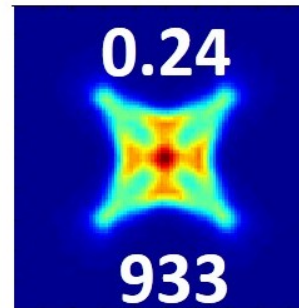
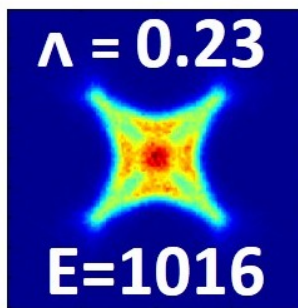
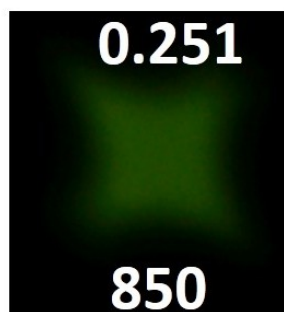
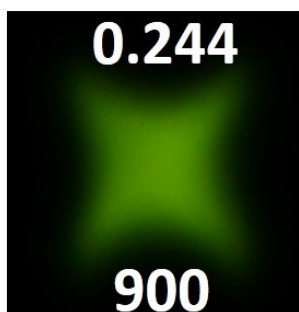
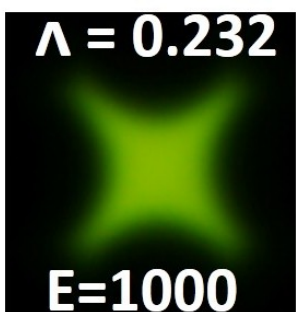


Simulated FWHM of superfocused proton distribution versus ion energy at the exit face of a 55-nm-thick [001] Si layer.

Experimental

and

Simulated Patterns



55-nm-thick [001] Si layer

- Indirect evidence of beam widths of ~ 20 pm from agreement between simulations and experimental patterns



Other work

Nuclear Instruments and Methods in Physics Research B 360 (2015) 23–29

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

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



Proton–silicon interaction potential extracted from high-resolution measurements of crystal rainbows

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ARTICLE INFO

Article history:

Received 20 May 2015

Received in revised form 21 July 2015

Accepted 21 July 2015

Keywords:

Ion channeling

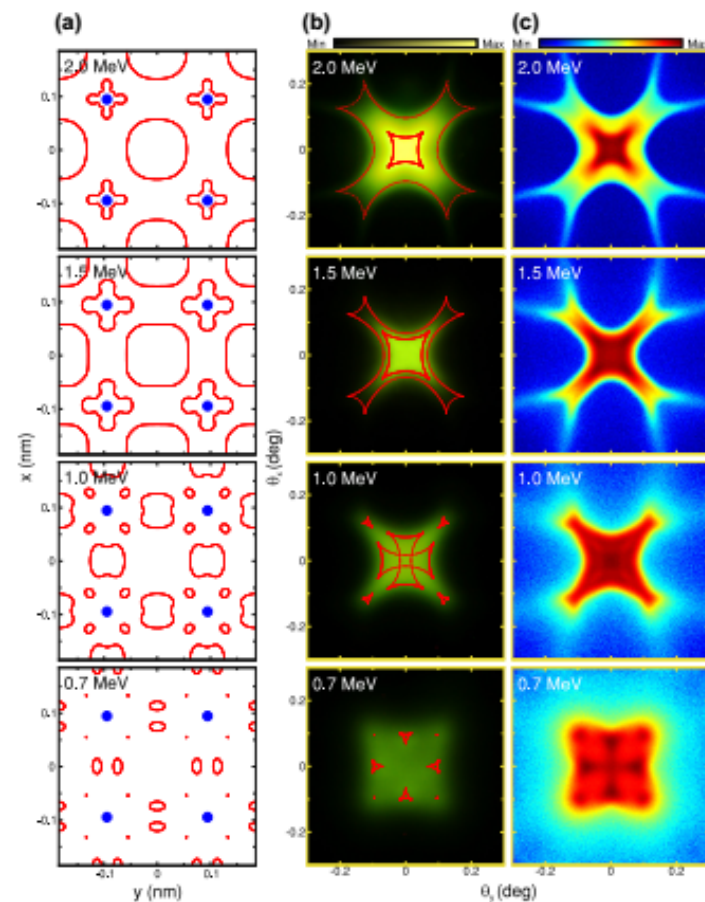
Rainbows

Interaction potential

ABSTRACT

This study provides a way to produce very accurate ion–atom interaction potentials. We present the high-resolution measurements of angular distributions of protons of energies between 2.0 and 0.7 MeV channelled in a 55 nm thick (0 0 1) silicon membrane. Analysis is performed using the theory of crystal rainbows in which the Molière's interaction potential is modified to make it accurate both close to the channel axis and close to the atomic strings defining the channel. This modification is based on adjusting the shapes of the rainbow lines appearing in the transmission angle plane, with the resulting theoretical angular distributions of transmitted protons being in excellent agreement with the corresponding experimental distributions.

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The End

Thank you !