Step Free Surface Heteroepitaxy of 3C-SiC Layers on Patterned 4H/6H-SiC Mesas and Cantilevers

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Most SiC devices are implemented in homoepitaxial films grown on 4H/6H-SiC wafers with surfaces 3° to 8° off-axis from the (0001) basal plane. This approach has not prevented many substrate crystal defects from propagating into SiC epilayers, and does not permit the realization of SiC heteropolytype devices.

This presentation describes recent advancements in SiC epitaxial growth that begin to overcome the above shortcomings for arrays of mesas patterned into on-axis 4H/6H-SiC wafers. First, we demonstrated that atomic-scale surface steps can be completely eliminated from 4H/6H-SiC mesas via on-axis homoepitaxial step-flow growth, forming (0001) basal plane surfaces (up to 0.4 mm x 0.4 mm) far larger than previously thought possible [1]. Step-free surface areas were then extended by growth of thin lateral cantilevers from the mesa tops [2]. These lateral cantilevers enabled substrate defects to be reduced and relocated in homoepitaxial films in a manner not possible with off-axis SiC growth [3]. Finally, growth of vastly improved 3C-SiC heterofilms was achieved on 4H/6H-SiC mesas using the recently developed step-free surface heteroepitaxy process [4,5]. These epitaxial growth developments should enable improved homojunction and heterojunction silicon carbide prototype devices.

[1] J. A. Powell et al.: Appl. Phys. Lett. Vol. 77 (2000), p. 1449
[2] P. G. Neudeck et al.: J. Appl. Phys. Vol. 92 (2002), p. 2391
[3] P. G. Neudeck et al.: Mat. Res. Soc. Symp. Proc. Vol. 742 (2003), p. K5.2.1
[4] P. G. Neudeck, et al.: et al.: Mater. Sci. Forum Vol. 389-393 (2002), p. 311
[5] P. G. Neudeck et al.: Mater. Sci. Forum Vol. 433-436 (2003), p. 213

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CWRU Materials Science and Engineering Colloquium
November 25, 2003

Acknowledgements

NASA
G. Beheim
M. Mrdenovich
B. Osborn
D. Androjna
G. Hunter
L. Matus
D. Larkin
J. Heisler
S. Elder
R. Meredith
T. Ferrier
C. Blaha
P. Lampard
M. Artale
B. Rodriguez

SUNY Stonybrook
M. Dudley
X. Huang
W. Vetter

Carnegie Mellon University
M. Skowronski
J. Liu
T. Kuhr

Work funded by NASA Glenn Research Center
Ultra Efficient Engine Technology Program
Revolutionary Aeropropulsion Concepts Program
Aerospace Power and Propulsion Technology Program

Outline

Experimental studies of on-axis SiC epitaxial growth on arrays of mesas patterned into 4H/6H-SiC wafers.

Homoepitaxial growth of large (hundreds of μm dimension) (0001) 4H/6H basal plane surfaces free of steps.

Heteroepitaxial nucleation and growth of 3C-SiC on top of step-free 4H/6H-SiC mesas.

Fundamental insights and potential benefits.

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International Conference on Silicon Carbide and Related Materials
October 5-10, 2003
Lyon, France

Acknowledgements

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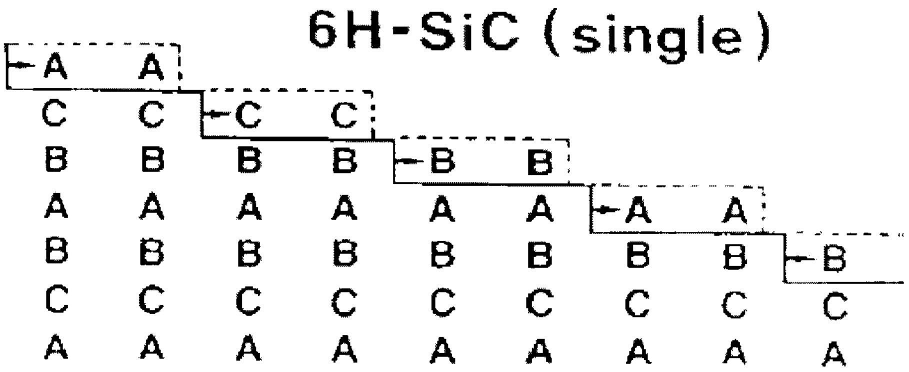
Conventional SiC Epitaxy on 4H/6H Substrates

(After T. Kimoto, A. Itoh, and H. Matsunami, Phys. Stat. Sol. (b) **202** p. 247, (1997))

Conventional SiC Homoepitaxy

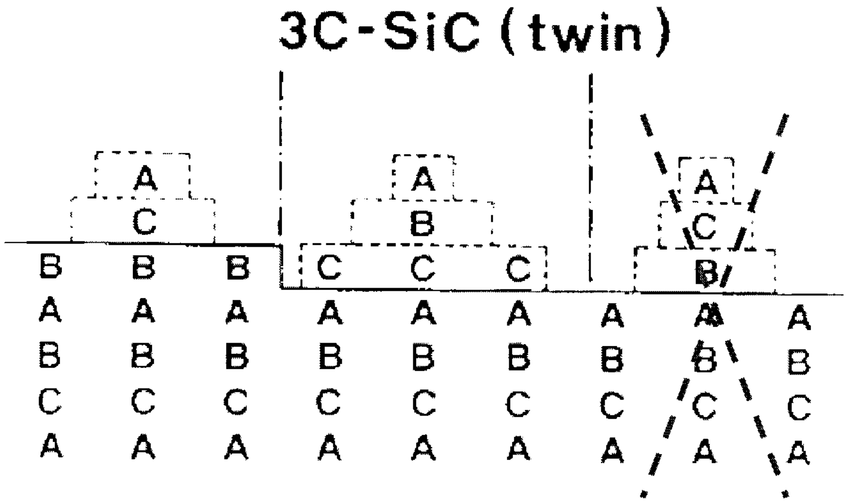
(Off-axis 4H/6H-SiC wafers)



High (> 3°) surface miscut angle causes small terrace width that promotes reactant incorporation at step edges.

6H polytype of epilayer is “step-controlled” via lateral stepflow growth of existing surface steps.
(**Kinetic** mechanism).

Previous “on-axis” SiC Epitaxy



Low (< 0.5 °) miscut angle makes 2D terrace nucleation of 3C-SiC more probable.

Nucleation on terraces results in 3C-SiC (**Thermodynamic** mechanism).

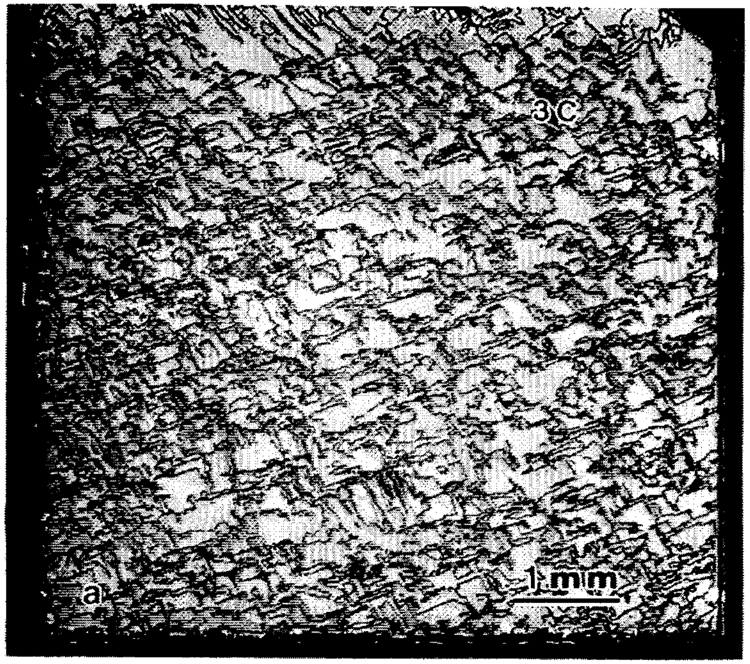
Poor 3C film quality - high extended defect density.

On-Axis Homoepitaxial SiC Growth

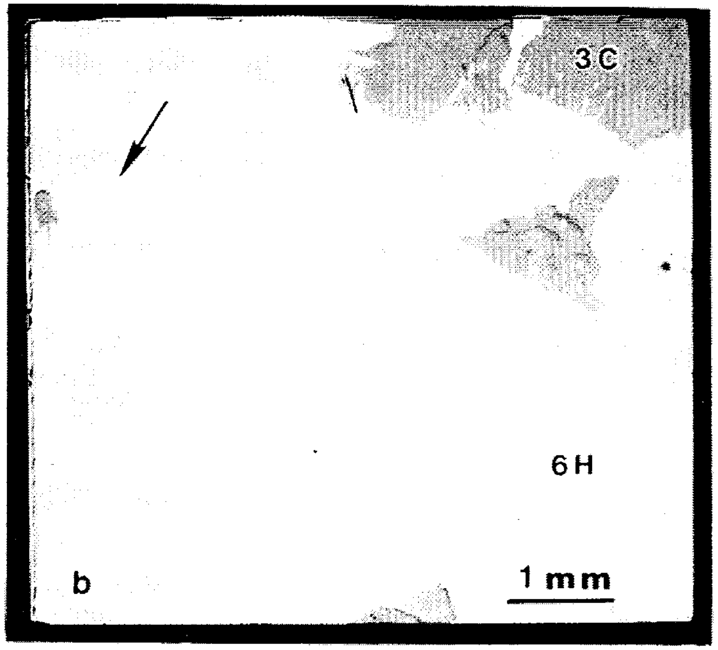
J. A. Powell et al., Appl. Phys. Lett. **59** p. 333 (1991).

On-axis (~ 0.2° miscut) stepflow homoepitaxy of 6H-SiC was realized at 1450 °C over large areas when suitable in-situ pre-growth etching was employed.

Inadequate Pre-Growth Etch
(HCl, 2 min. @ 1175 °C)



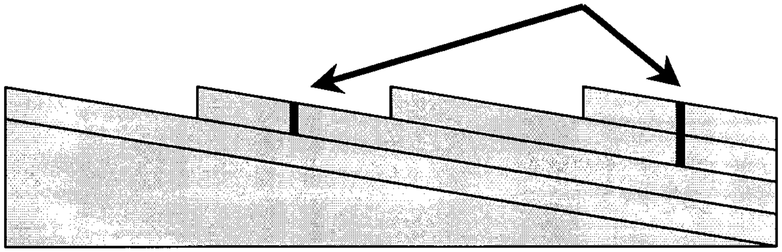
Sufficient Pre-Growth Etch
(HCl , 20 min. @ 1375 °C)



Critically important pre-growth etch removes surface damage that greatly assists terrace nucleation of 3C-SiC.

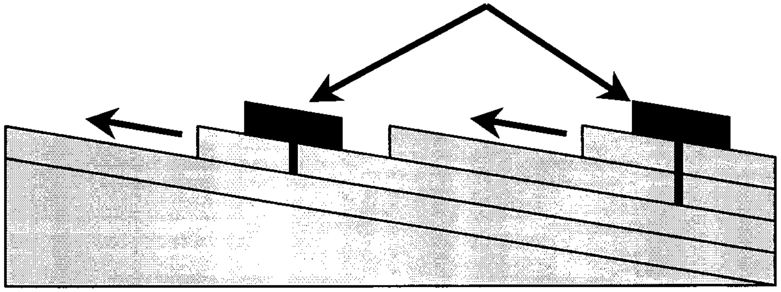
Defect-Assisted Terrace Nucleation Model

Original Wafer Surface with Defects/Contamination



4H- or 6H-SiC
(0001) Surface

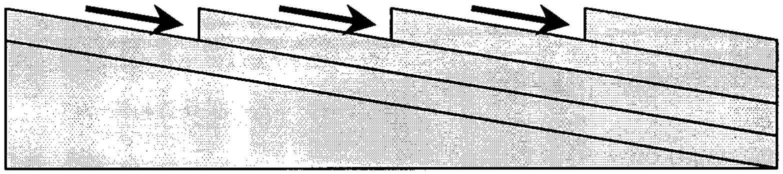
Epitaxial Growth Initiated With Surface Defects Present
(2D Terrace Nucleation at Defects)



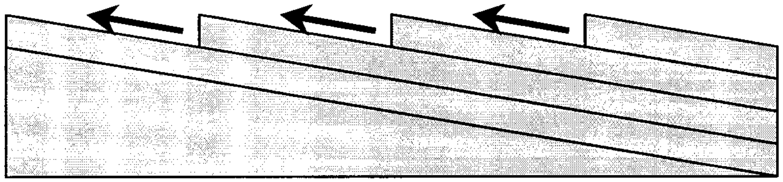
Results in Film With 3C Inclusions

No 2D Nucleation of 3C-SiC on the Defect-Free Terraces

In-Situ Pre-Growth Etch Removes Surface Defects/Contamination



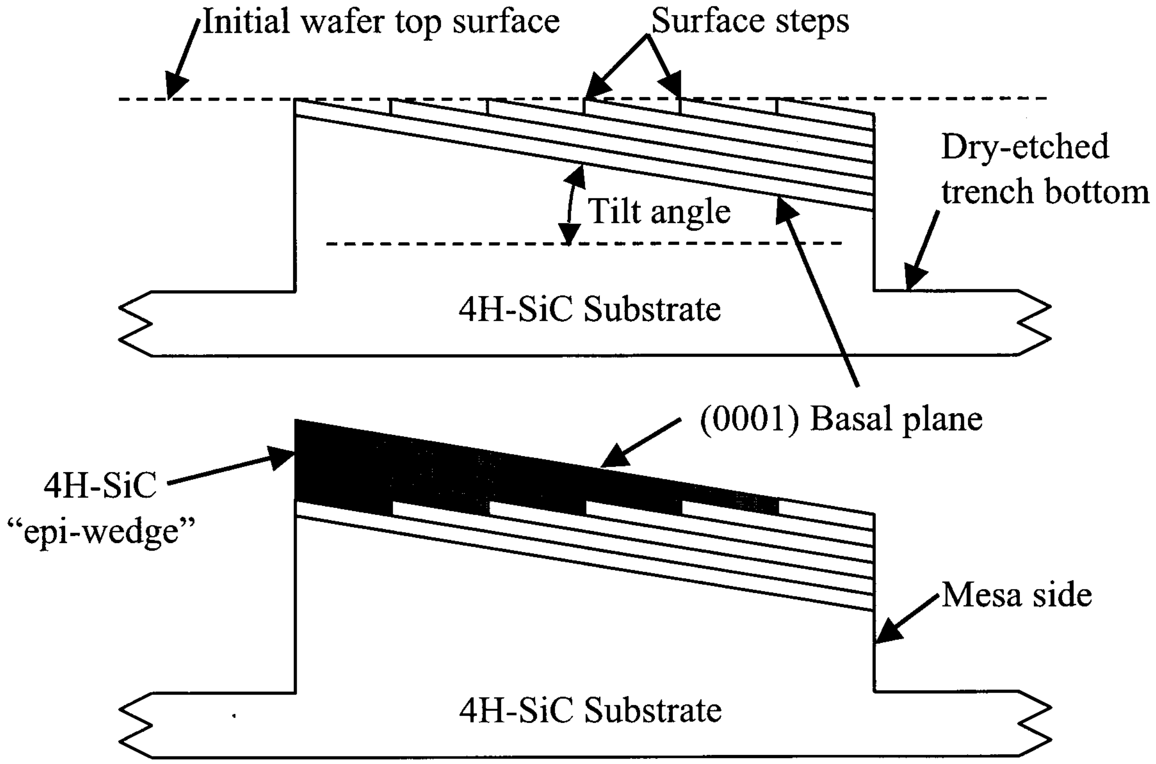
Epitaxial Growth Initiated Without Surface Defects Present



Adatom Mobility Sufficient for Stepflow Growth Without 3C Inclusions

Growth of Step-Free 4H/6H-SiC Mesa Surfaces

J. A. Powell et al., Appl. Phys. Lett. **77** p. 1449 (2000).



1. Dry etch mesas into on-axis wafer (strip etch mask).
2. In-situ pre-growth etch (HCl or H_2).
3. Stepflow growth at 1600-1700 °C <u>without any 2D nucleation of 3C-SiC.</u>

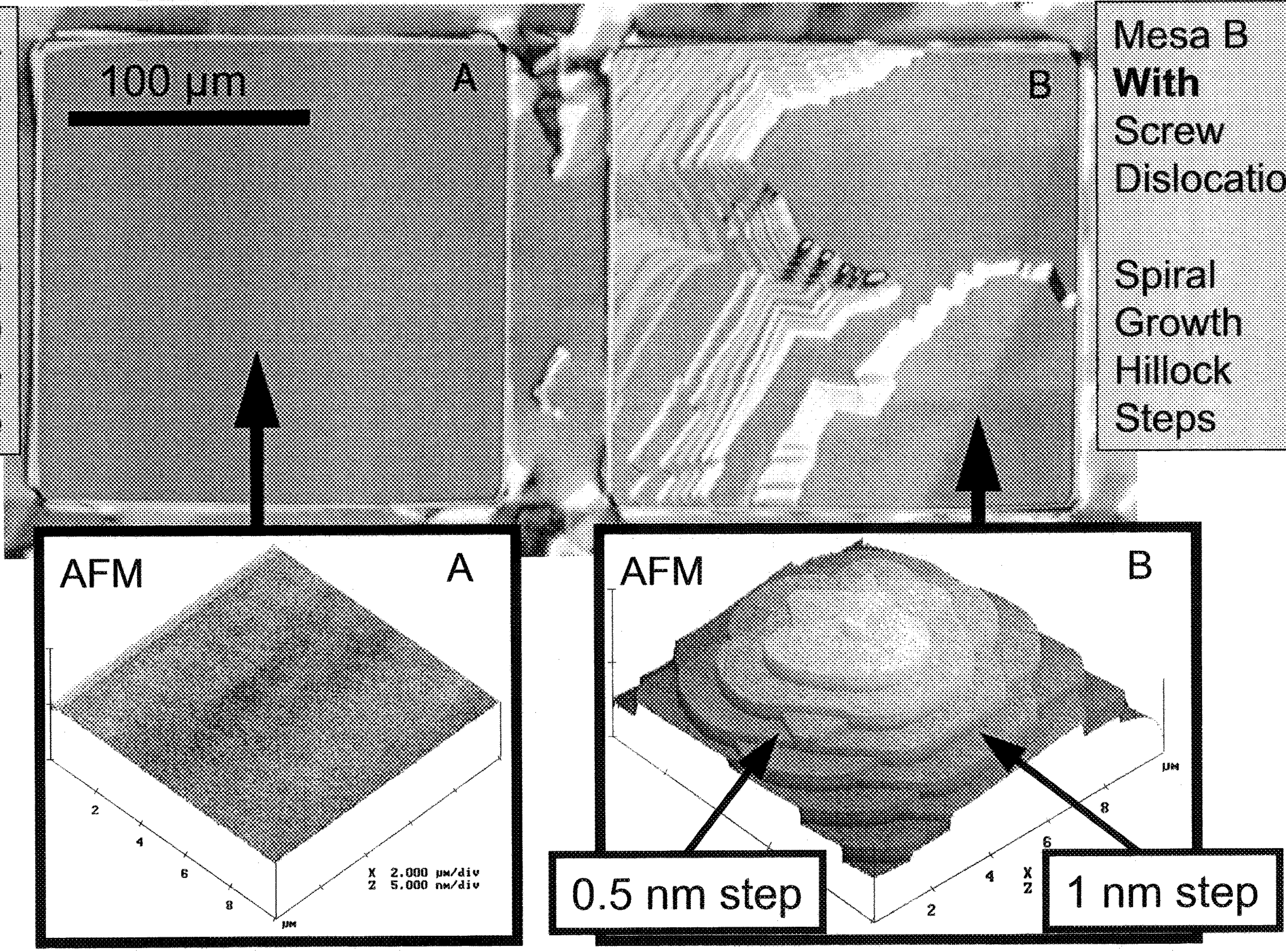
No growth (i.e., no new bilayers) in <0001> direction.

Process does not work if screw dislocation (kinetic step source) threads mesa.

Growth of Step-Free 4H-SiC Mesa Surfaces

Mesa A
Without
Screw
Dislocation

200x200 µm^2
(0001)
Step-Free
Surface



Mesa B
With
Screw
Dislocation

Spiral
Growth
Hillock
Steps

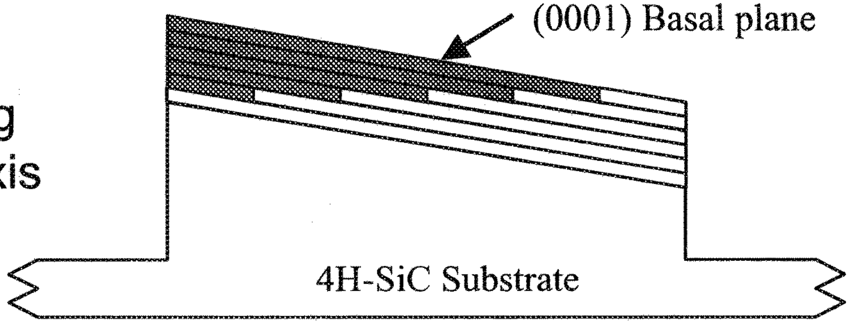
J. A. Powell et al., Appl. Phys. Lett. **77** p. 1449 (2000).

Result Summary

Step-free yield primarily limited by substrate screw dislocations (SD's).

- 400 µm x 400 µm step-free mesas realized on SD-free mesas.

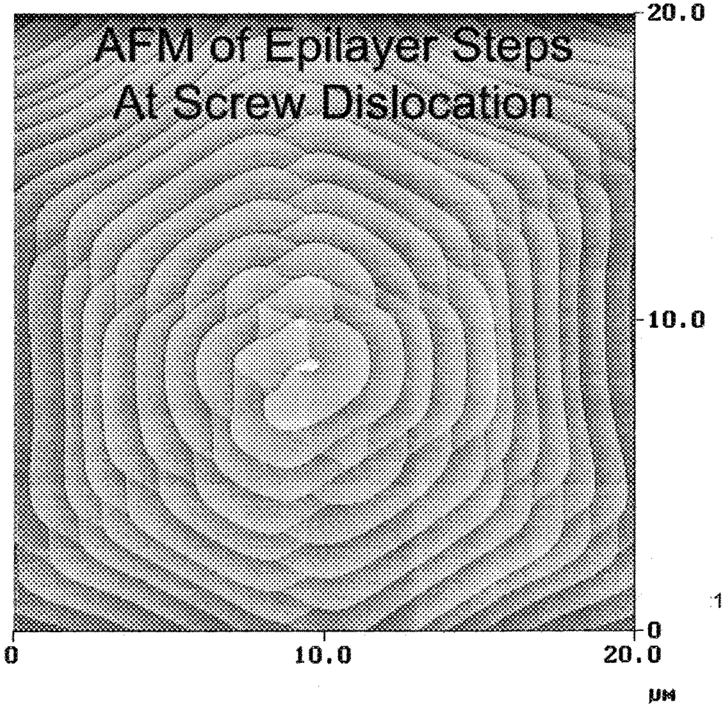
Demonstrates **complete suppression of 2D terrace nucleation of 3C-SiC** during on-axis growth (corresponding off-axis growth rate ~2 µm/hour).



Demonstrates **very long adatom terrace diffusion lengths**

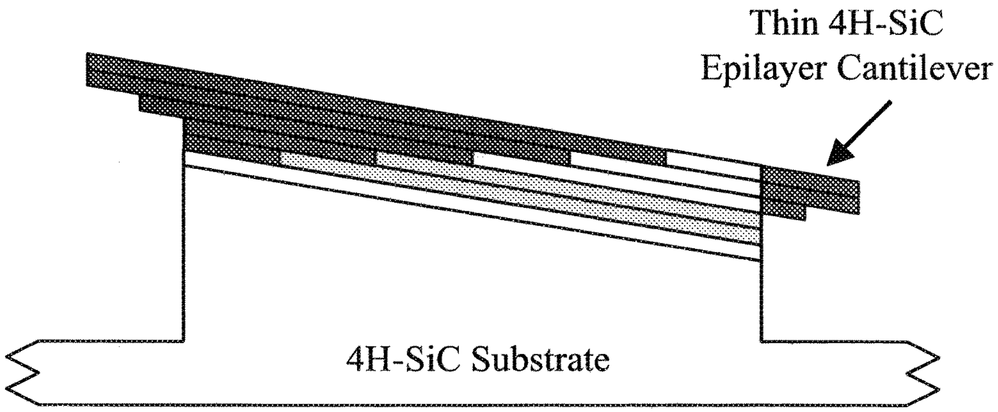
Surface miscut angle and tilt direction is local, especially following epitaxial growth!

Step-free mesas provide a zero-miscut-angle growth surface.



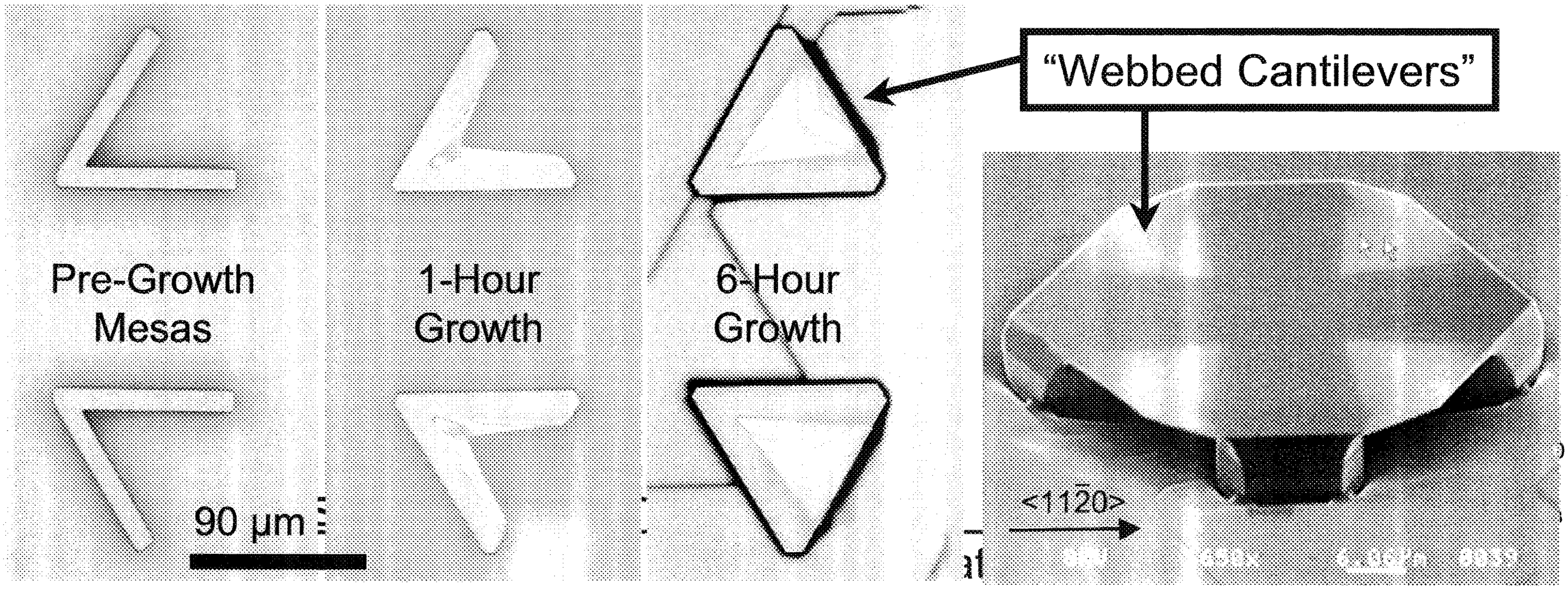
Homoepitaxial Growth of Thin Cantilevers

P. G. Neudeck et al., J. Appl. Phys. 92 p. 2391 (2002).

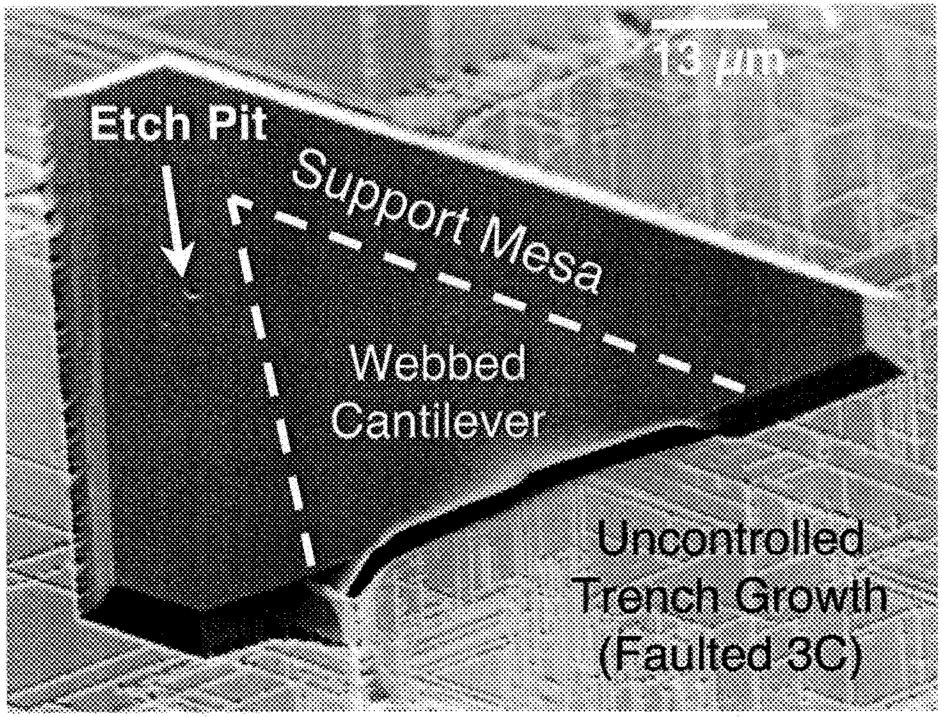


Continued growth with 2D nucleation completely suppressed leads to thin lateral cantilever formation that extends the step-free mesa surface.

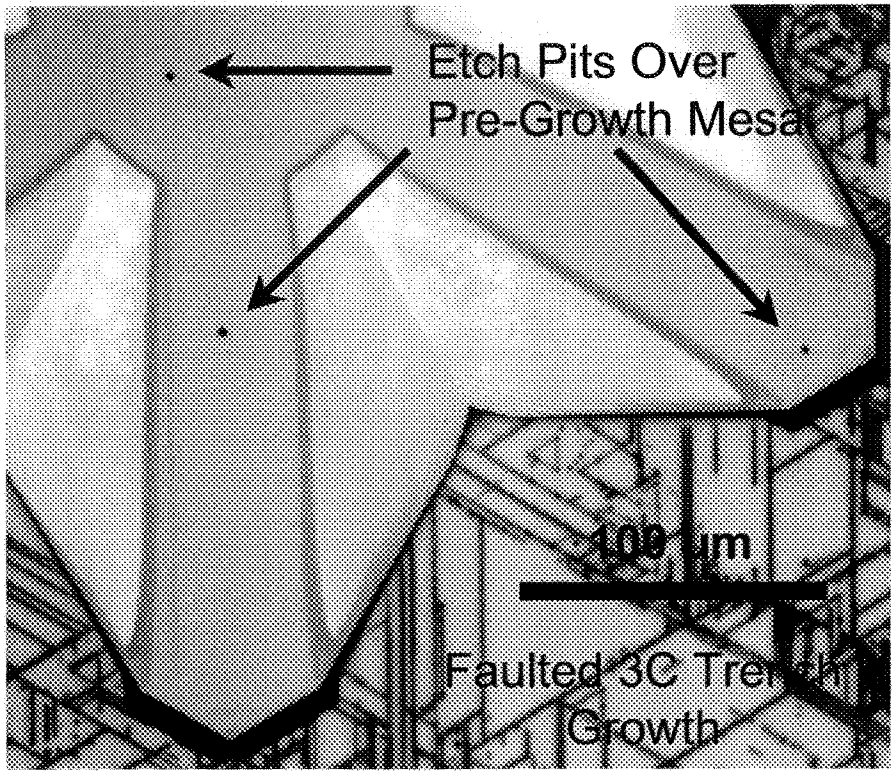
Cantilever evolution strong function of mesa shape and crystallographic orientation.



Defect-Preferential Etching of 4H-SiC Webbed Cantilevers



(Etch: ~ 50 sec. molten KOH @ ~ 500 °C)



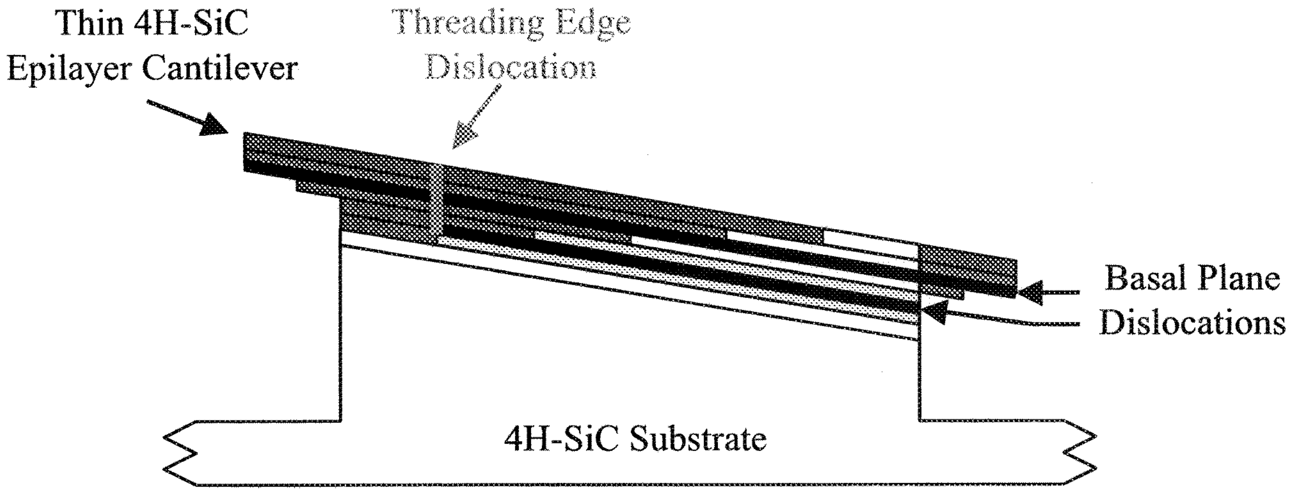
Large hexagonal etch pits (not shown above) observed at screw dislocations.

Small hexagonal etch pits (shown above) observed on pre-growth mesa regions.

No etch pits observed on outwardly-coalesced thin cantilevers, even when cantilevers reside over micropipes or closed-core screw dislocations.

Coalescence of cantilevers from separate mesas almost always defective.

Dislocation Model for 4H/6H-SiC Mesa Films

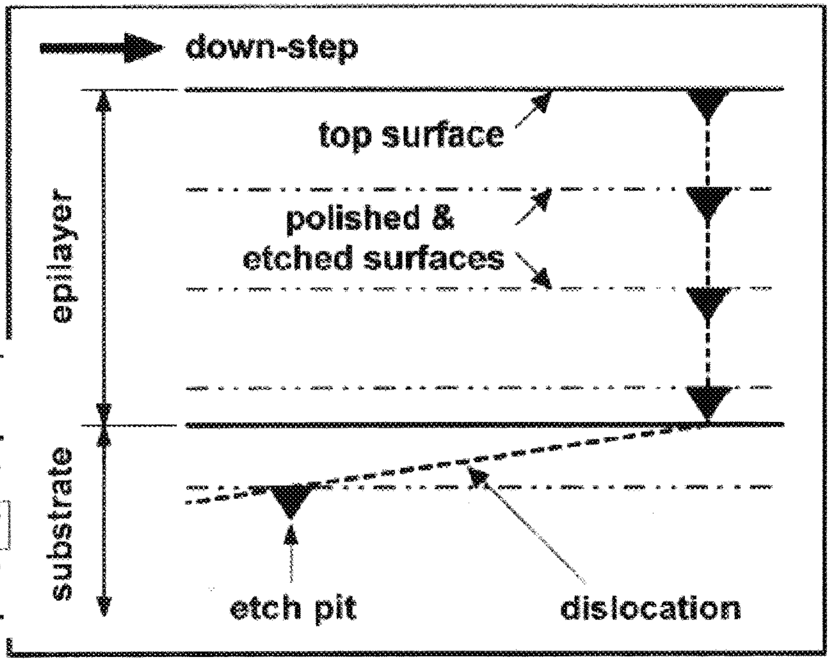


Small etch pit behavior is consistent with epilayer threading edge dislocation.

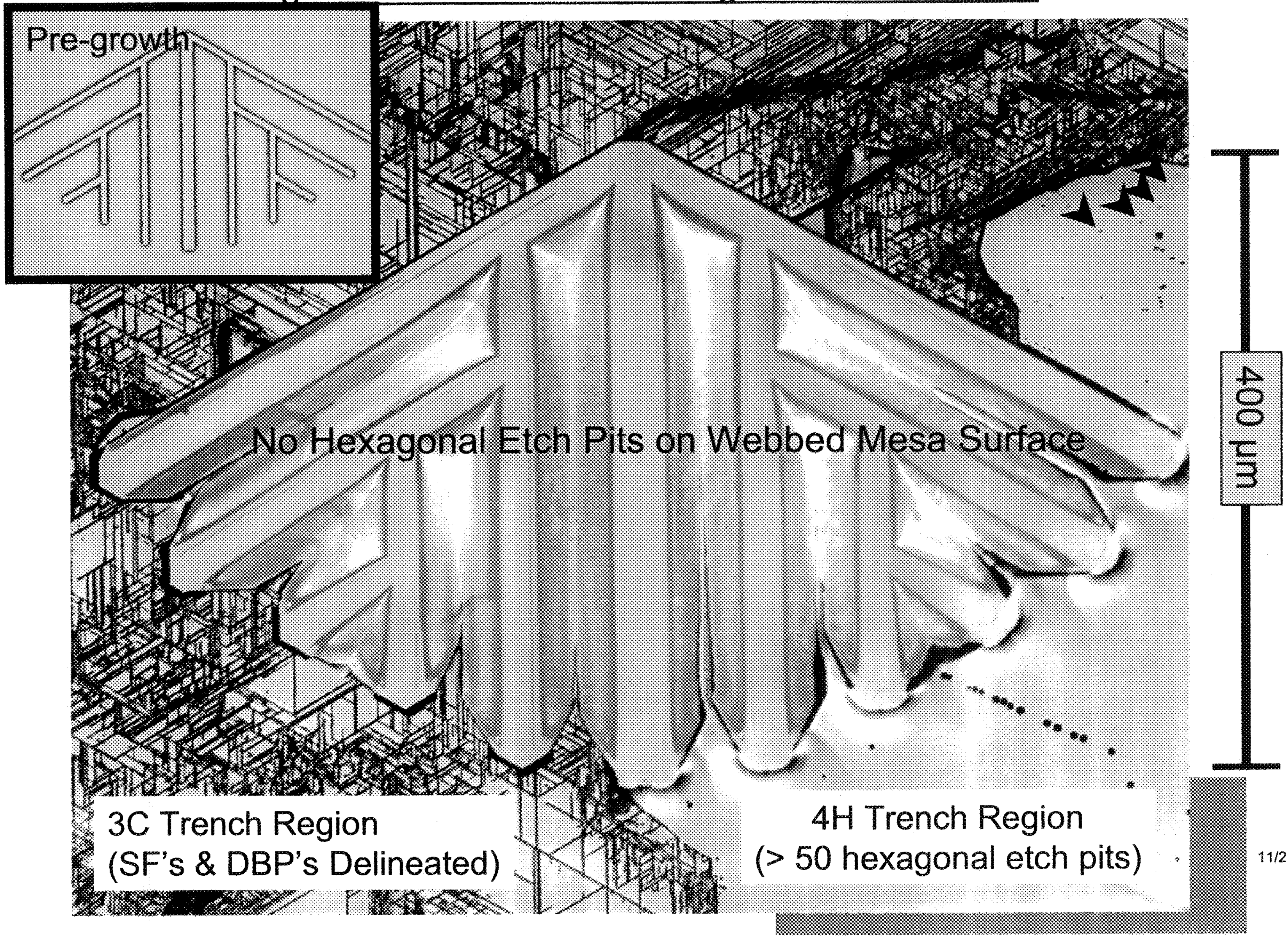
Etch pit results suggest that Ha et al's. model for off-axis dislocation conversion (from substrate basal plane to epilayer threading edge) also applies to on-axis homoepilayers.

S. Ha, et al., J. Cryst. Growth **244** p. 257 (2002).

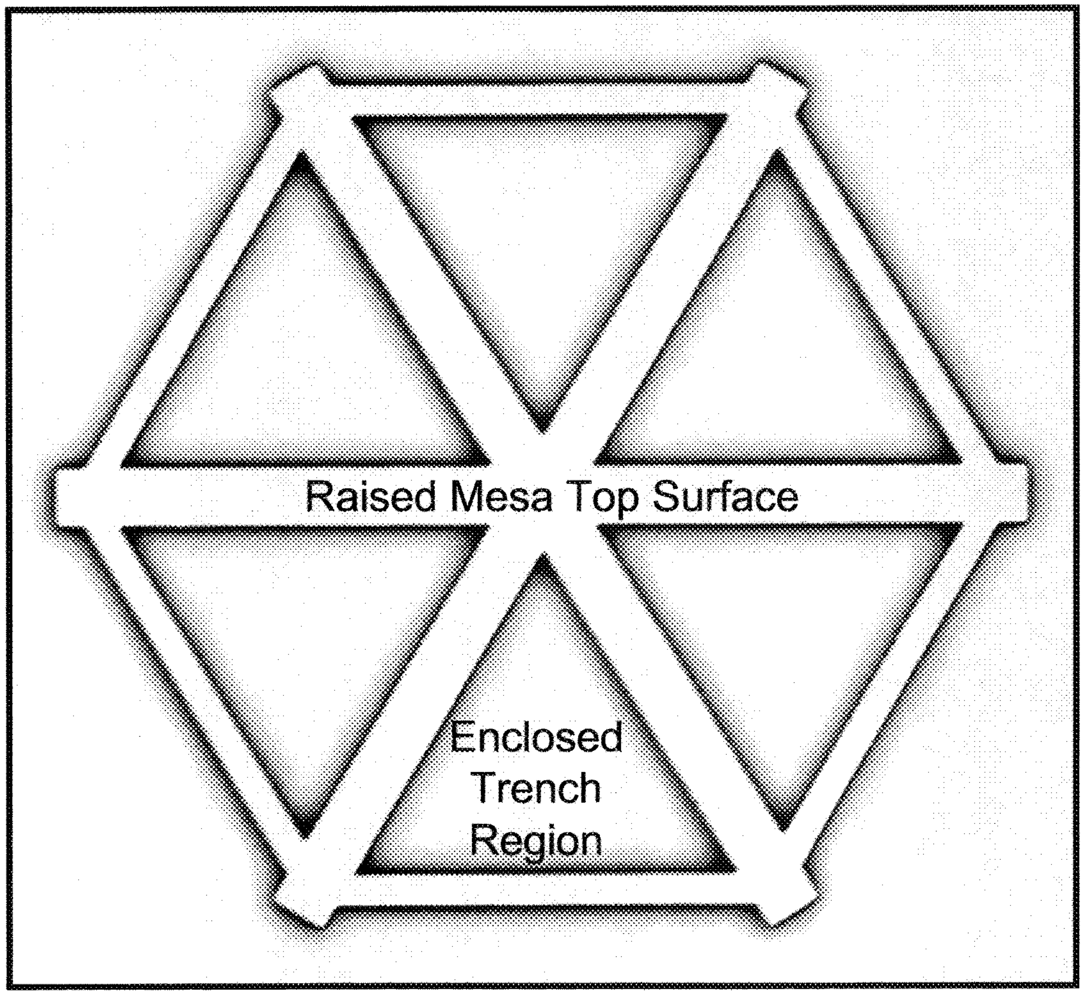
	Substrate	Epilayer
Basal plane (cm^{-2})	$\boxed{2 \times 10^4 \quad 6 \times 10^4}$	$8 \times 10^2 \quad 3 \times 10^3$
Threading edge (cm^{-2})	$8 \times 10^2 \quad 8 \times 10^3$	$\boxed{3 \times 10^4 \quad 2 \times 10^5}$
Threading screw (cm^{-2})	$2 \times 10^3 \quad 6 \times 10^3$	$3 \times 10^3 \quad 6 \times 10^3$



Large Web Structure Following Molten KOH Etch



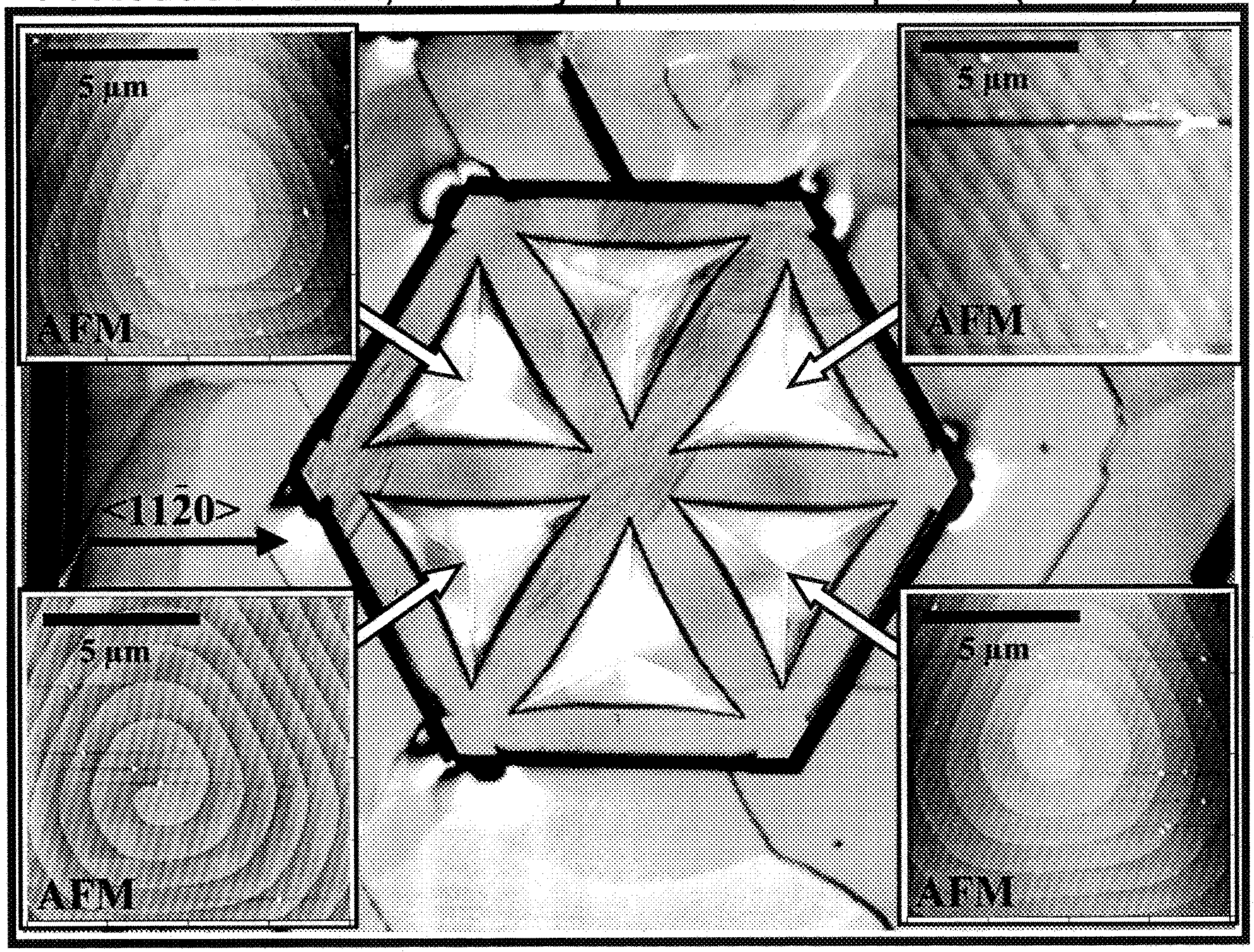
Mesa Shapes With Enclosed Trench Regions



Pre-growth mesa designed so that thin cantilevers enlarge from mesa edge to completely roof over enclosed trench regions.

Post-Growth Mesa Roofed by Thin Cantilevers

P. Neudeck et al., MRS Symp. Proc. **742** p. 241 (2003)



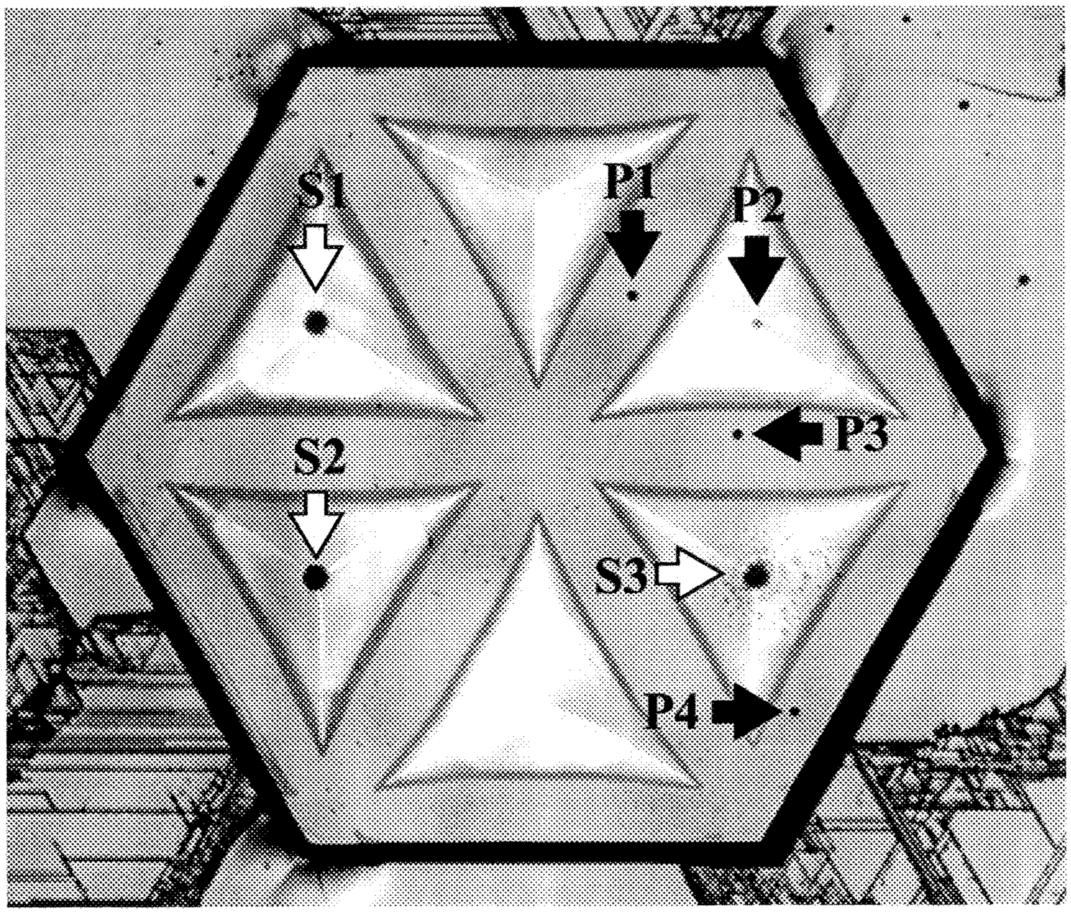
Three screw dislocations formed at three points of final roof closure.

- Kinetic stepsources for <0001> film growth.

Post-Growth Mesa Roofed by Thin Cantilevers

P. Neudeck et al., MRS Symp. Proc. **742** p. 241 (2003)

Mesa Following Molten KOH Etching



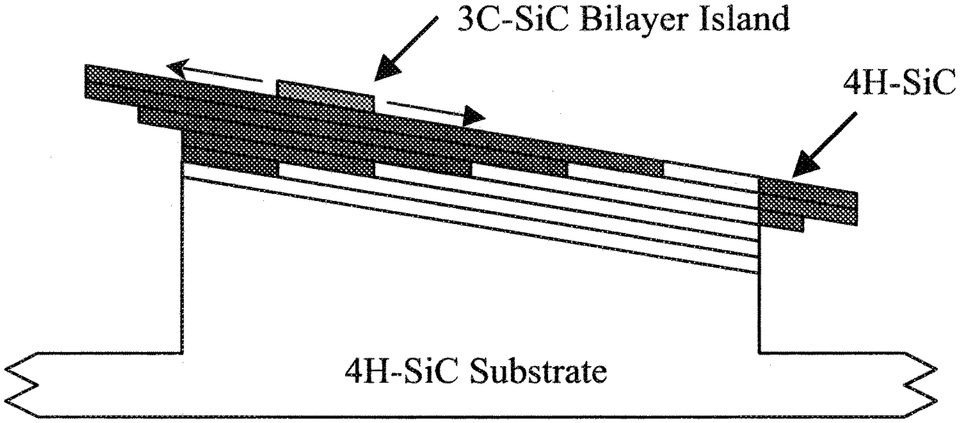
Pits only appear in cantilevers at final coalescence points.

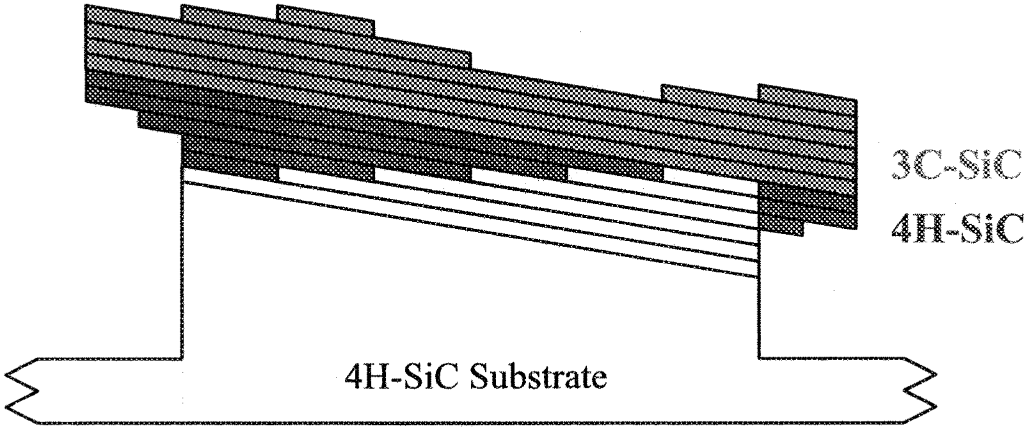
Large pits S1-3 correspond to screw dislocations.

Small pits P1-4 correspond to threading edge dislocations.

Based upon the principle of Burgers vector conservation, we hypothesize that all c-axis propagating substrate dislocations enclosed within a hollow trench region become combined into a single dislocation in the cantilever film roof at the point of final roof coalescence.

Heteroepitaxial Growth of 3C-SiC on Step-Free 4H/6H-SiC Mesas
("Step-Free Surface Heteroepitaxy" [1])





1. Establish step-free 4H/6H-SiC mesa surface via pure stepflow growth above 1600 °C (with or without large cantilevers).

2. Intentionally terrace nucleate 3C-SiC on step-free 4H/6H surface in situ by lowering growth temperature 100-200 °C (without interruption from Step 1 growth above).

3. Continue nucleation and growth of subsequent 3C-SiC bilayers to produce thicker films.

Experimental results indicate that low initial nucleation rate in Step 2 is important in obtaining high yield of 3C mesa films free of stacking fault (SF) defects [1].

[1] P. Neudeck et al., Mater. Sci. Forum **389-393** p. 251 (2002).

Characterization of 3C-SiC Mesa Heterofilms

(Unintentionally doped 3C films less than 4 μm thick)

3C-SiC polytype of mesas independently confirmed by:

- Thermal Oxide Color Mapping (oxidation rate of 3C different from 4H/6H)
- Synchrotron White Beam X-Ray Topography (SWBXT) and HRXRD.
- High Resolution Cross-Sectional Tunneling Electron Microscopy (HRXTEM)

No evidence of other SiC polytypes have been observed to date.

3C-SiC mesa heterofilm defect structure studied by:

- Thermal Oxide Color Mapping (enhanced oxidation at SF and DPB defects).
- Molten KOH Etching (enhanced etching at all extended defects).
- HRXTEM (reveals stacking disorder through film thickness).
- SWBXT and High Resolution X-Ray Diffraction (HRXRD).
    - 3C film stress due to lattice constant mismatch ($\Delta a/a$ 0.01-0.1%).
    - 111(3C) peaks 17 - 25 arcsec FWHM (same as 4H substrate).

High quality 3C-SiC films on step-free 4H/6H mesas (SF-free yields 50-95%).

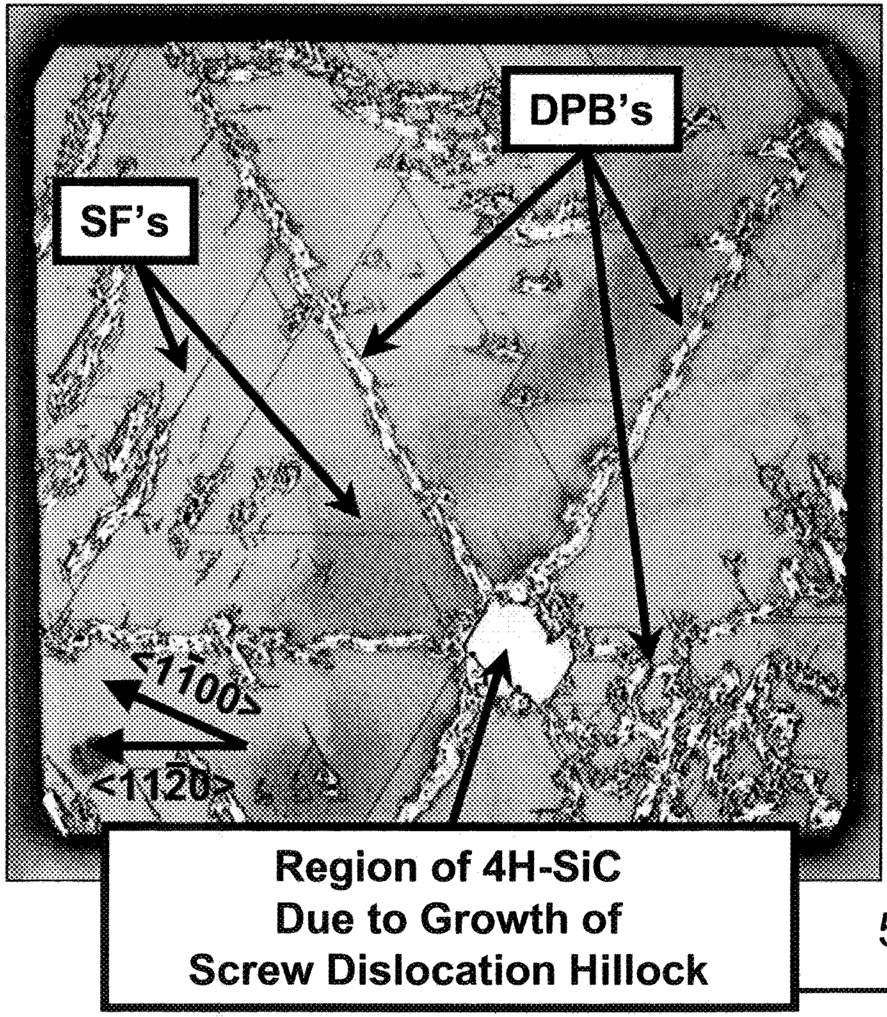
Poor quality 3C-SiC films on 4H/6H mesas with screw dislocations (SD's).

[1] P. Neudeck et al., Mater. Sci. Forum **389-393** p. 251 (2002).
[2] M. Dudley et al., Mater. Sci. Forum **389-393** p. 391 (2002).
[3] P. Neudeck et al., Mater. Sci. Forum **433-436** p. 213 (2003).
[4] M. Dudley et al., Mater. Sci. Forum **433-436** p. 247 (2003).
[5] X. Huang et al. Mat. Res. Soc. Symp. Proc. **742** p.205 (2003).

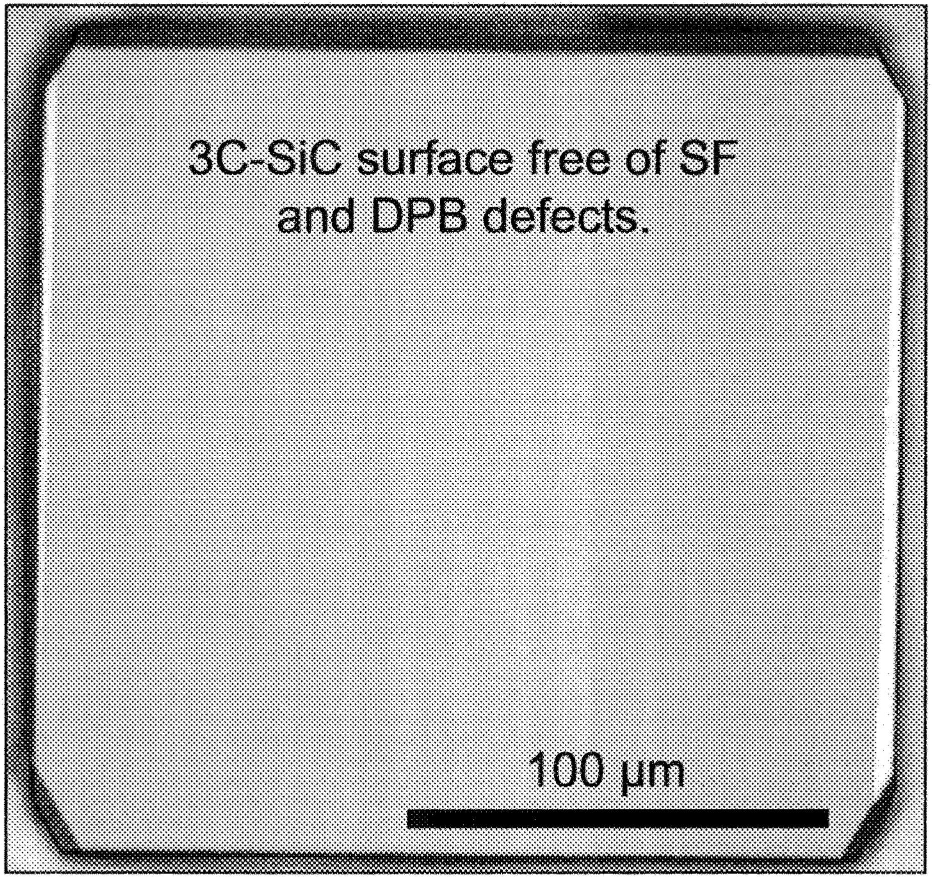
3C-SiC Mesa Heterofilms

Films have been dry-oxidized to map polytype and reveal stacking fault (SF) and double-positioning boundary (DPB) defects intersecting the film surface.

3C Film Grown on 4H Mesa With Steps



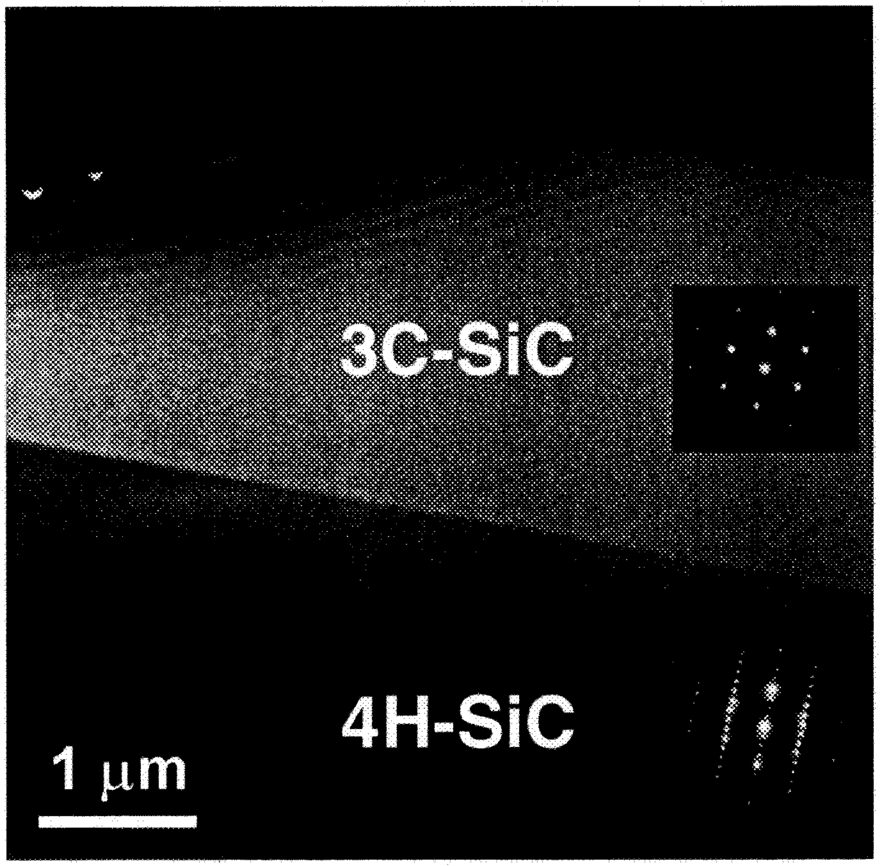
3C Film Grown on Step-Free 4H Mesa

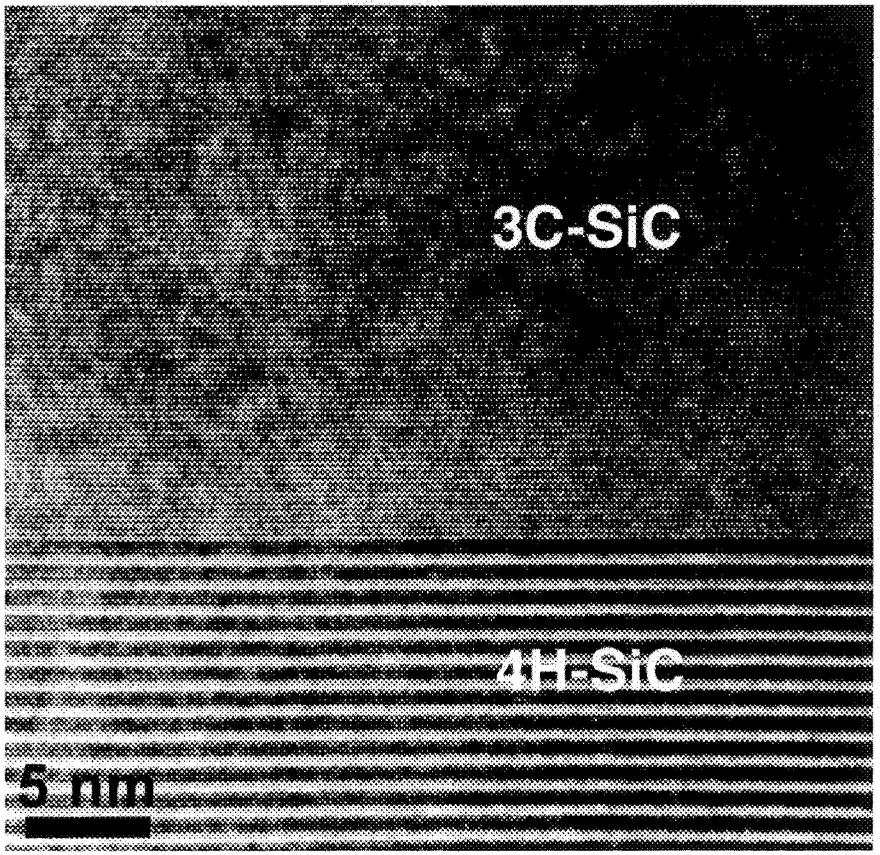


5-8 hours 1150 °C dry oxidation

Cross-Sectional TEM

(M. Skowronski and J. Liu, Carnegie Mellon University [1])



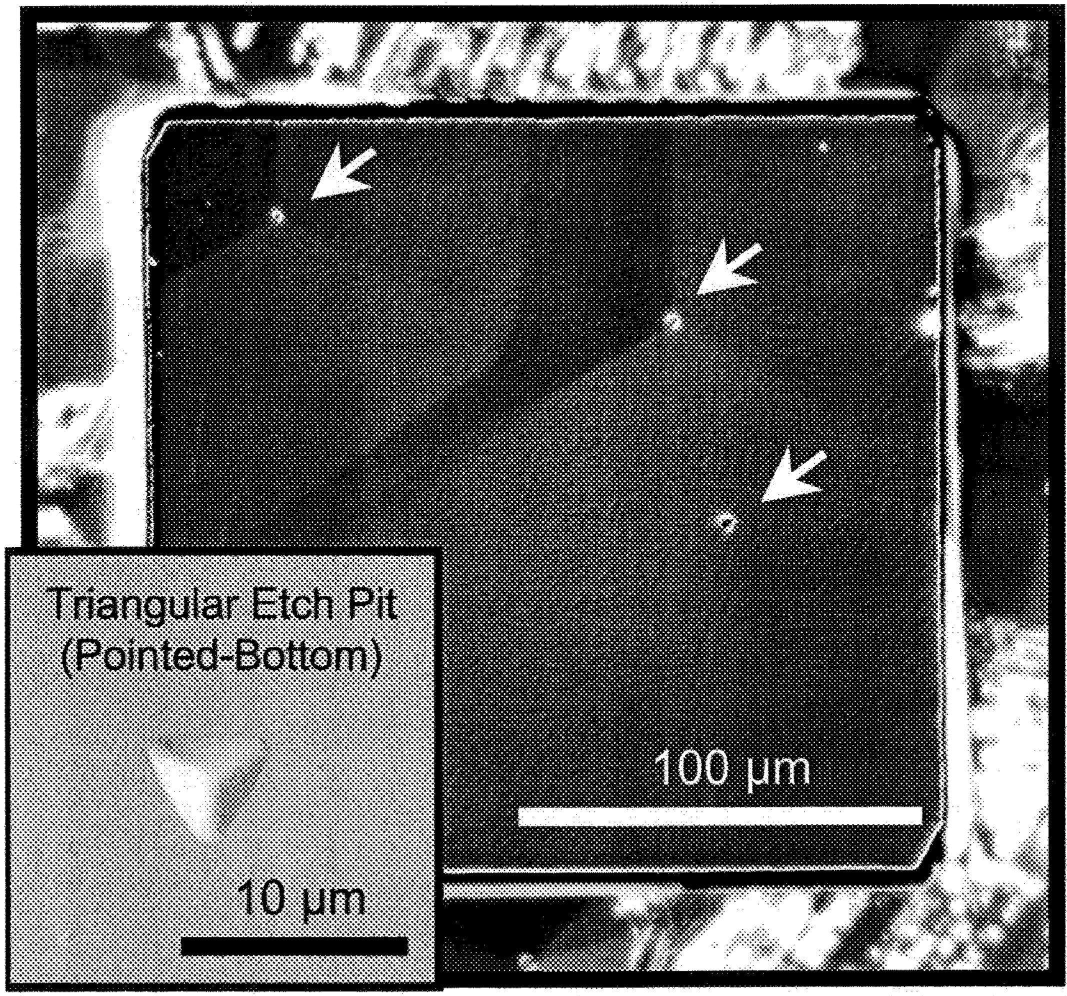


- Atomically abrupt 3C/4H interface with no steps.
- No stacking disorder observed throughout the entire 3C-SiC film thickness.
- Absence of any hexagonal stacking (twin-planes) suggests strong thermodynamic preference for new bilayers to continue cubic stacking of underlying bilayers.

[1] P. Neudeck et al., Mater. Sci. Forum **433-436** p. 213 (2003).

Defect-Preferential Etching of 3C-SiC Mesa Heterofilms

3C Mesa After 70 sec. 500 °C KOH Etch

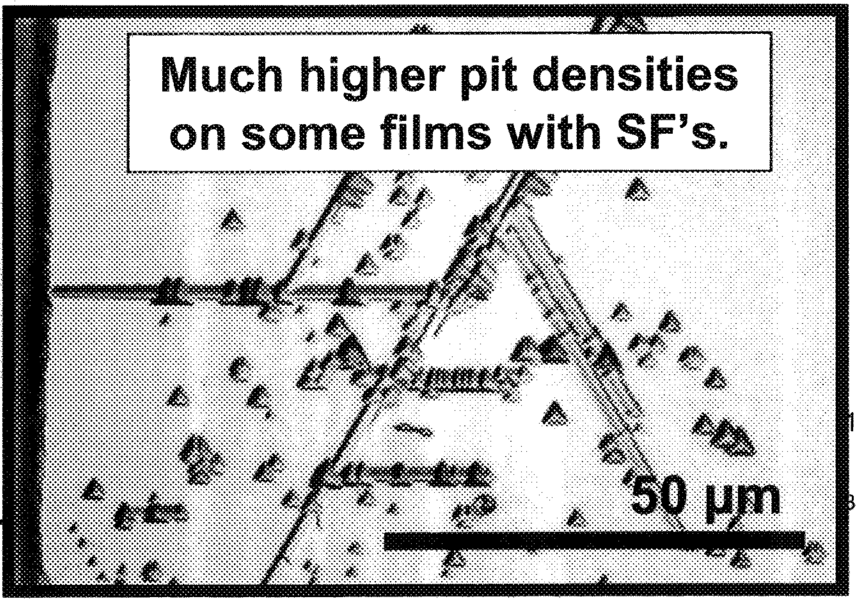


3C Film Growth Temperature 1400 - 1450 °C

Isolated triangular etch pit defects were revealed on SF-Free 3C-SiC mesa heterofilms.

In 3C-SiC films grown at 1400 - 1500 °C, pits are observed at peaks of triangular growth hillocks (but not all pits have hillocks).

Area-normalized etch pit density <u>on SF-Free 3C Films</u> ~ 10,000/cm^2

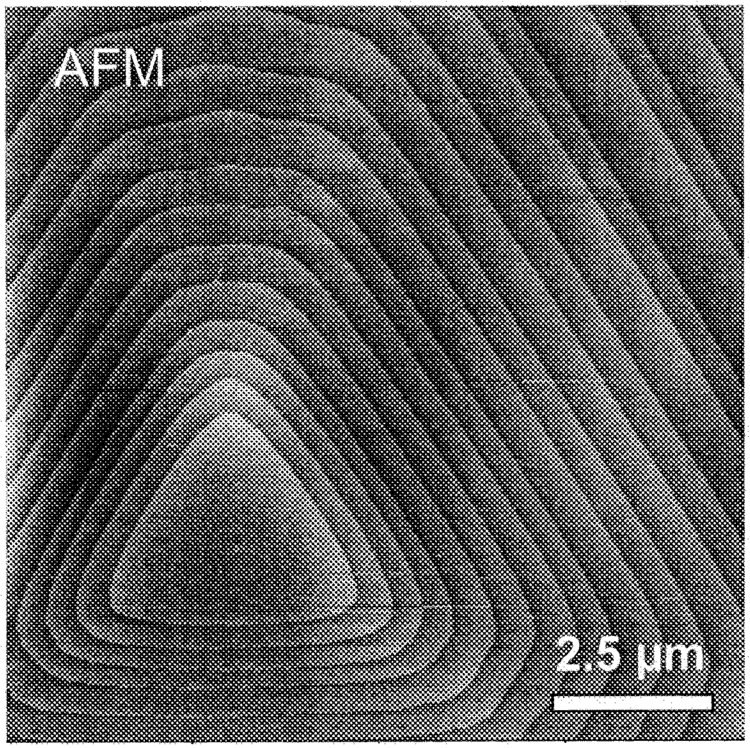


Much higher pit densities on some films with SF's.

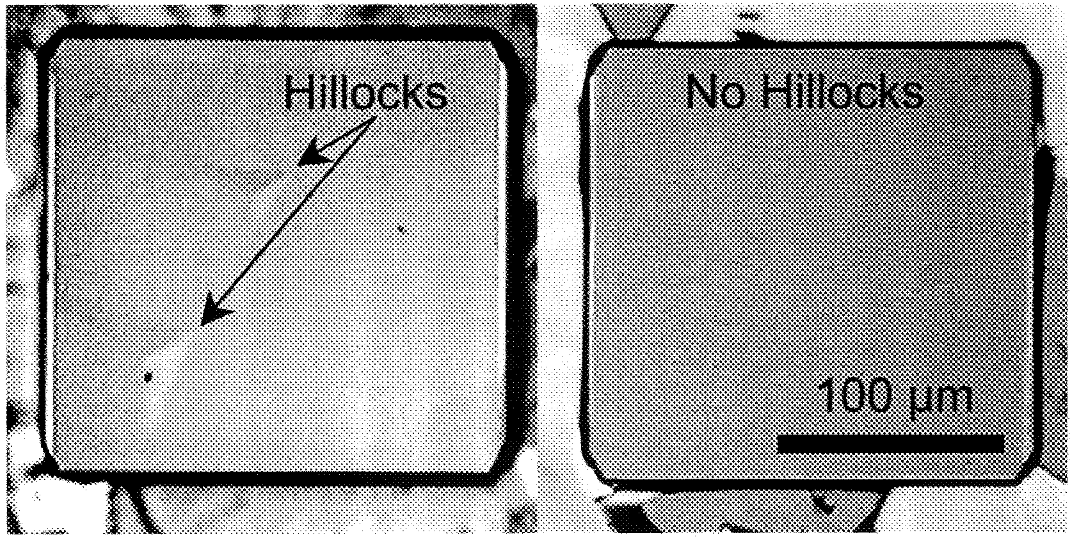
3C-SiC Mesa Film Surfaces

Almost all 3C-SiC surface steps have single-bilayer (0.25 nm) height.

(1470 °C 1-Minute 3C-SiC Growth)



Triangular hillock formation suppressed by higher 3C-SiC growth temperature.



1400 - 1450 °C

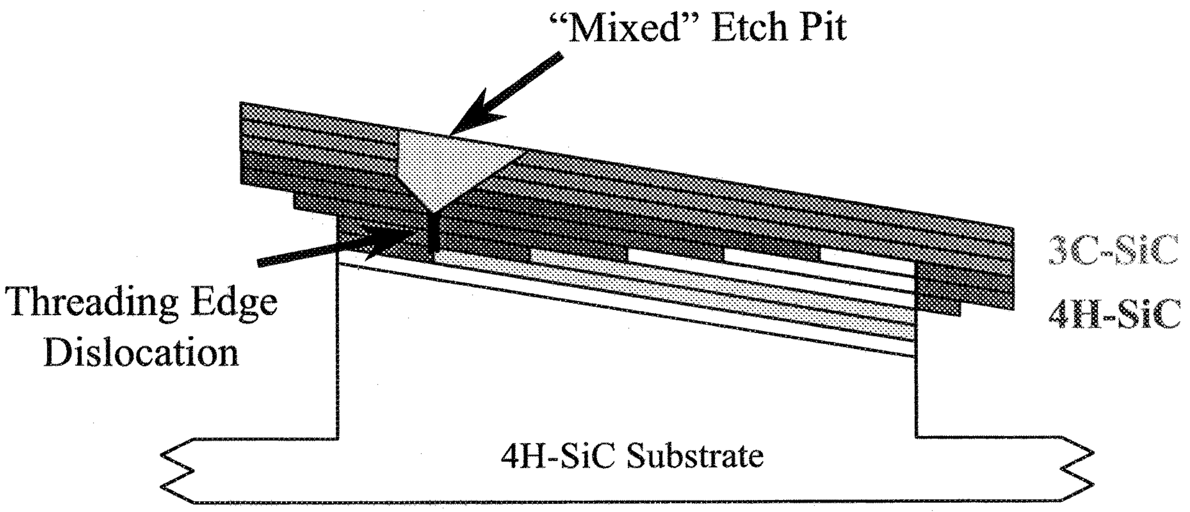
> 1550 °C

Triangular hillock formation initiates during initial stages of 3C-SiC film growth.

Triangular hillocks have concentric (not spiral) step pattern.

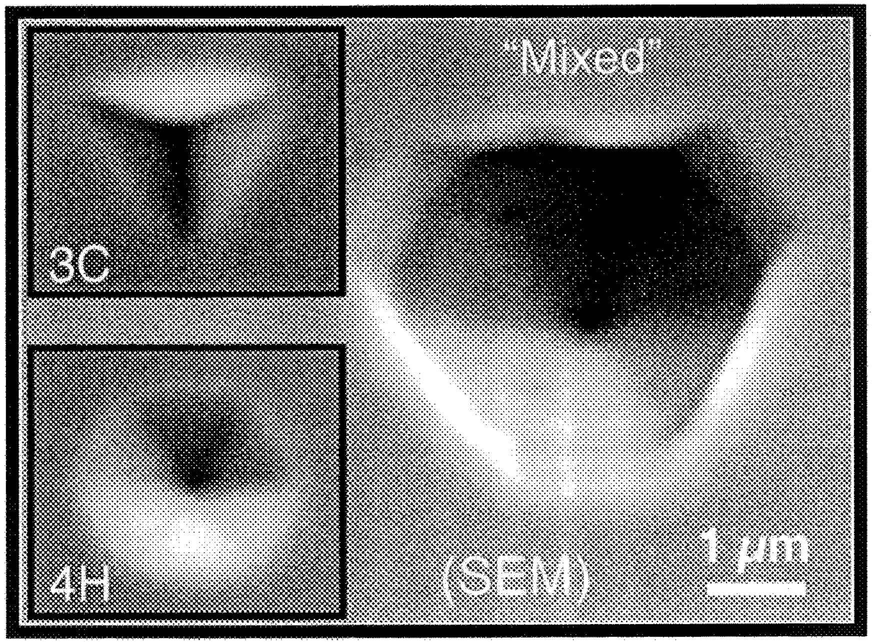
- Consistent with defect-assisted 2D nucleation.
- Inconsistent with screw dislocation (single or paired) kinetic stepsource.

Origin of Etch Pits in SF-Free 3C-SiC Films

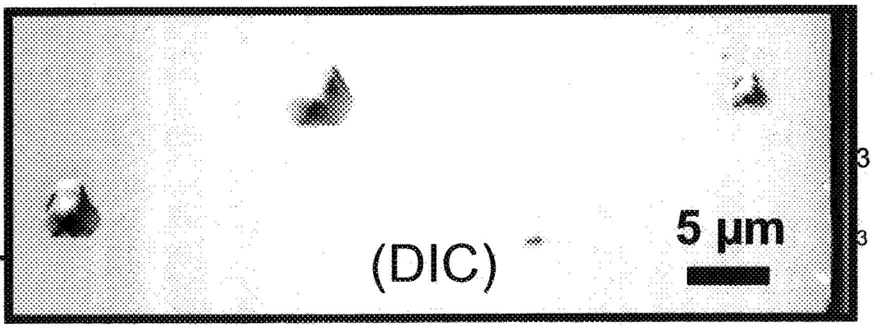


Molten KOH etching of thin 3C mesa films produces "mixed" etch pits that penetrate 3C/4H heterointerface.

Upper portion of "mixed" pits exhibit triangular (3C) geometry, while deeper portion exhibits hexagonal pointed-bottom geometry previously associated with 4H-SiC threading edge dislocations.



We propose that the majority of isolated etch pits in SF-free 3C-SiC films are threading edge dislocations that propagate from the step-free 4H-SiC epilayer (that originate from 4H-SiC substrate dislocations [1]).

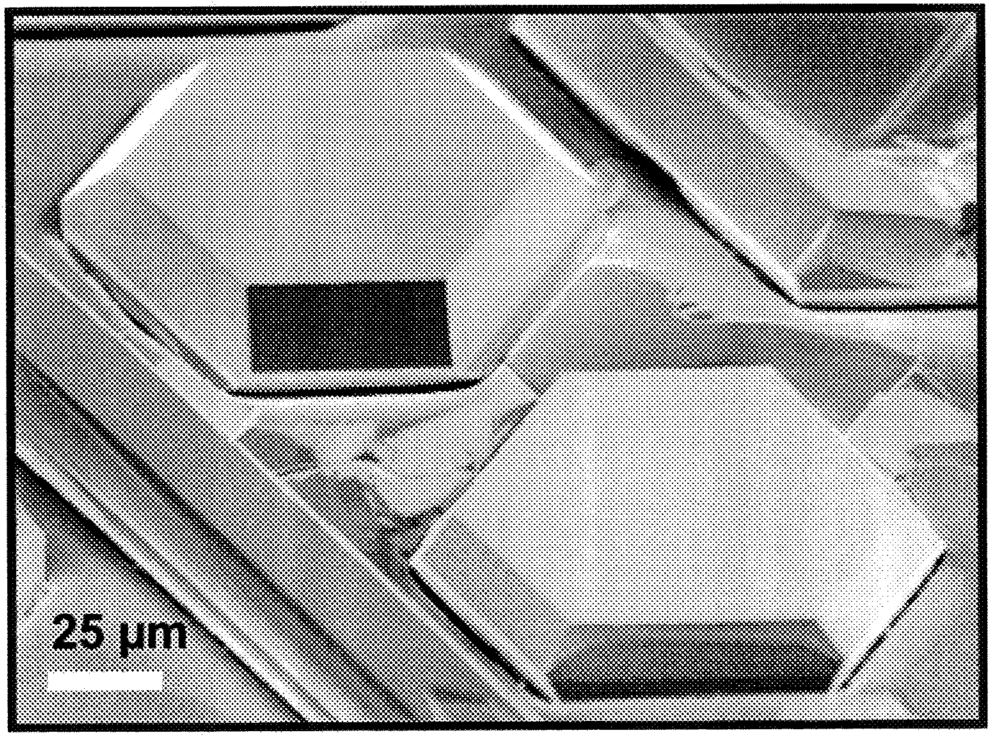


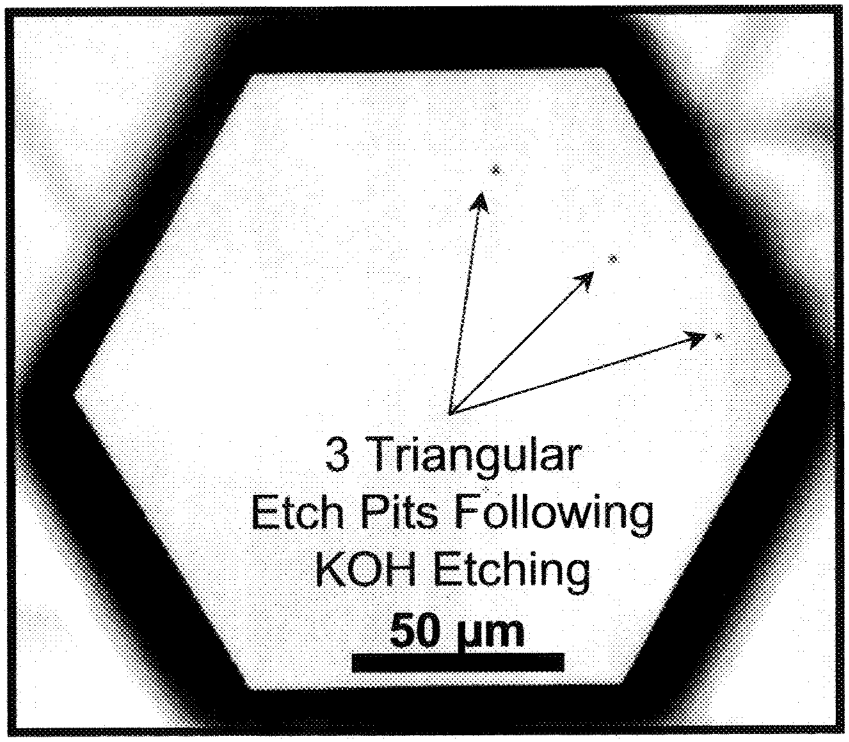
[1] S. Ha, et al., J. Cryst. Growth **244** p. 257 (2002).

Thick 3C-SiC Films

(4-hour 3C-SiC Film Growth at ~1550 °C)

Thick (> 13 µm) 3C-SiC Films Grown on Hexagonal-Shaped Mesas

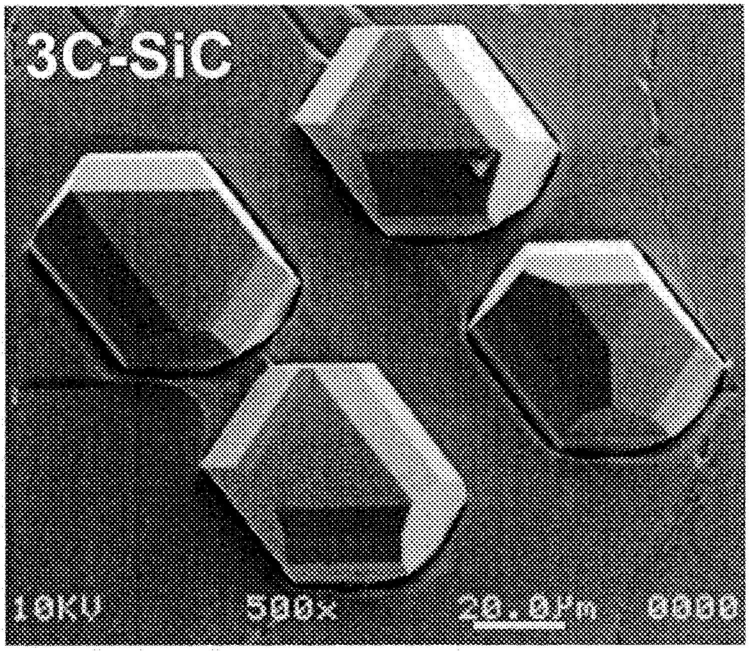




Comparable SF-free yield (> 70 % for SD-free mesas) and etch pit density (~ 10,000/cm^2) as obtained for thin (< 4 µm) 3C-SiC mesa heterofilms.

Suggests that dislocation-generating 3C/4H lattice mismatch strain relief has not occurred at this film thickness.

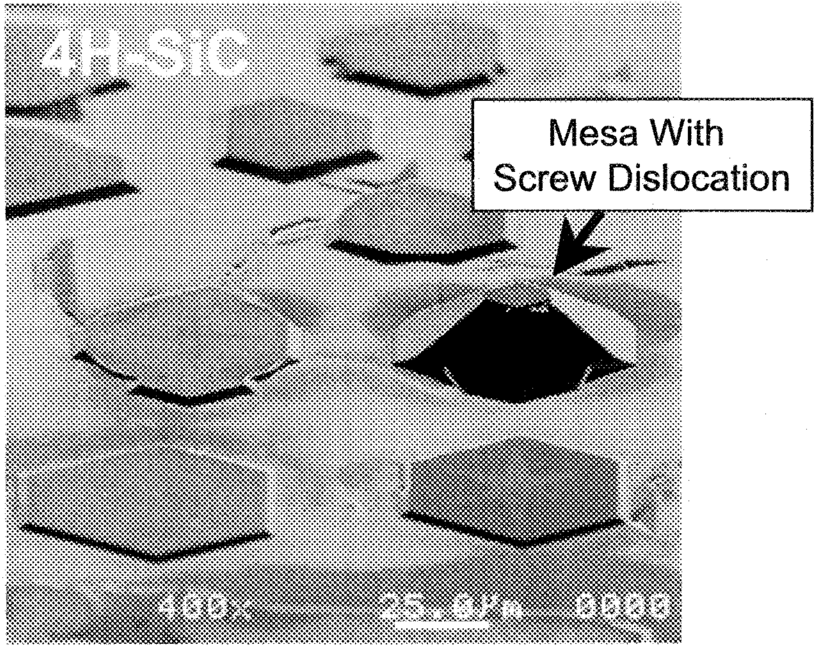
3C vs 4H/6H Mesa Film Growth



Cubic Crystal Structure
- Four (111) closed-packed planes.

Growth with 2D nucleation and stepflow expansion.

Crystal enlargement without dislocations **is 3-dimensional.** Growth occurring on enlarging side-facet surfaces likely to further growth of thicker 3C-SiC films, even when 2D nucleation on the (111) top mesa surface becomes suppressed at high growth temperatures.



Hexagonal Crystal Structure
- Single (0001) closed-packed plane.

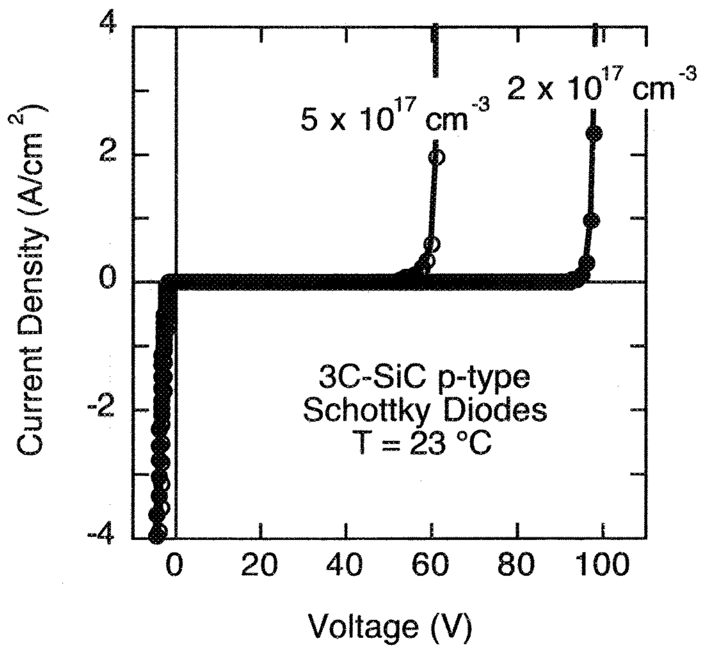
Kinetic crystal growth. Pure stepflow without 2D nucleation is required to maintain polytype.

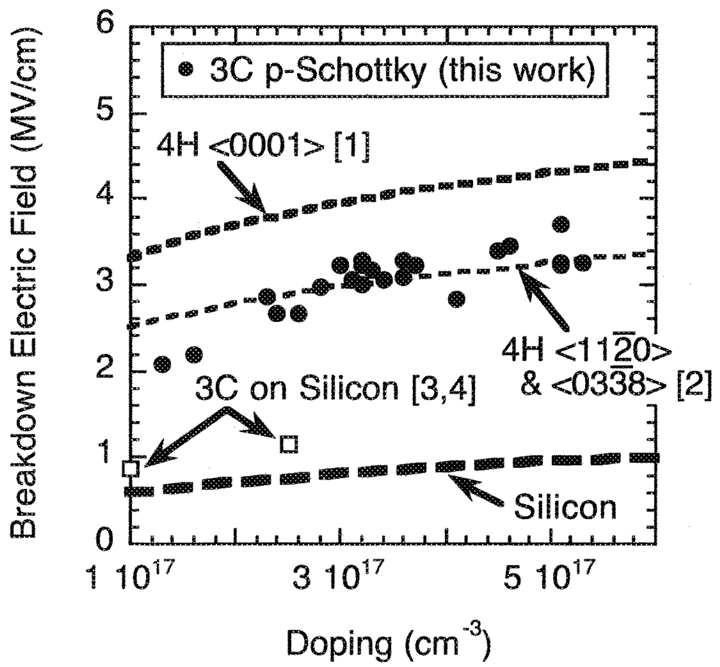
Crystal enlargement without screw dislocations **is confined to 2 dimensions**. No growth in <0001> direction.

Doping and Devices

Initial investigations of doped 3C-SiC films and devices are underway.

Initial results on p-type 3C-SiC Schottky diodes reported in poster.





[1] A. O. Konstantinov et al. : J. Electronic Materials Vol. 27 (1998), p. 335

[2] S. Nakamura et al. : Appl. Phys. Lett. Vol. 80 (2002), p. 3355

[3] C. W. Liu and J. C. Sturm, J. Appl. Phys. Vol. 82 (1997) p. 4558

[4] Y. Ishida et al. : Mater. Sci. Forum Vol. 389-393 (2002), p. 459

Summary

On-axis mesa growth experiments have enabled fundamental insights into SiC epitaxial growth, polytypism, and extended defects.

Device-sized regions of unique SiC epitaxial crystal useful for prototype device studies.

- Step-free (0001) 4H- and 6H-SiC surfaces
  (MOS and metal-semiconductor interfaces and sensors, III-N heteroepitaxial growth and devices, nanodevices).
- 4H- and 6H-SiC cantilevered surfaces free of dislocations
  (Dislocation-free bipolar high-voltage devices, MEMS).
- 3C-SiC heteroepilayers comparable to 4H/6H-SiC homoepilayer quality
  (3C-SiC MOSFET's, bipolar diodes & transistors).
- Atomically flat and abrupt 3C/4H heterojunctions (with abrupt doping).
  (3C/4H heterojunction transistors).

Further process improvements and scale-up are possible if prototype device results demonstrate substantial benefits.