Development of SiC Large Tapered Crystal Growth

Dr. Philip G. Neudeck

NASA Glenn Research Center

June 17, 2014

Project ID# APE027

Overview

Timeline

- Funding start: Dec. 2009
- Project end: March 2015
- Percent complete: 80%

Budget

- Total project funding

  (4 years Labor + Procurement)
  - DOE: $2.3M
  - NASA: $1.3M
- $300K from DOE for FY13
- $0 from DOE for FY14

Barriers

- Advanced Power Electronics and Electric Machines (APEEM)

Table 2.1-4. Technical Targets for Electric Traction System

	2010[a]	2015[b]	2020[b]
Cost, $/kW	<19	<12	<8
Specific power, kW/kg	>1.06	>1.2	>1.4
Power density, kW/L	>2.6	>3.5	>4.0
Efficiency (10%-100% speed at 20% rated torque)	>90%	>93%	>94%

Partners

- NASA Glenn (Lead)
- Ohio Aerospace Institute
- Sest, Inc.
- NASA Postdoctoral Program (Oak Ridge Assoc. Universities)
- Stony Brook University
- Free Form Fibers, Inc.

Objectives

- SiC power semiconductor devices should theoretically enable vastly improved power conversion electronics compared to today's silicon-based electronics.
  - 2-4X converter size reduction and/or 2X conversion loss reduction (theoretical performance gains vary with system design specifications).
  - Fundamentally improved implementation of smart grid, renewable energy, electric vehicles, aircraft and space power systems.
- SiC wafer defects and cost inherent to existing SiC material growth approach presently inhibiting larger benefits from becoming more widely available.
- **New but unproven NASA "Large Tapered Crystal" (LTC) SiC growth concept proposed to lower SiC material defect and cost technology barrier.**

U.S. DEPARTMENT OF ENERGY | Energy Efficiency & Renewable Energy

Vehicle Technologies Program Multi-Year Program Plan (2011-2015)

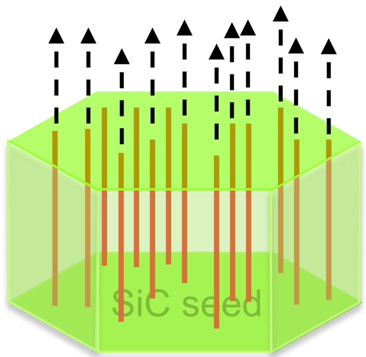
Table 2.1-6 Tasks for Advanced Power Electronics and Electric Motors R&D

Task	Title	Barriers Addressed
Task 1	**Power Electronics Research and Development** *New Topologies*- achieve significant reductions in PE weight, volume, and cost, and improve performance: • Reduce need for capacitance by 50%–90%, to yield 20% – 35% inverter volume reduction and cost reduction • Reduce part count by integrating functionality, to reduce inverter size and cost, and increase reliability • Reduce inductance, minimize electromagnetic interference and ripple, and reduce current through switches, all resulting in reduced cost *WBG semiconductors* - higher reliability and higher efficiency, and enable high-temperature operation	A, B, C, D, E, F

Approach/Strategy

Present SiC Growth Process

(Vapor transport)



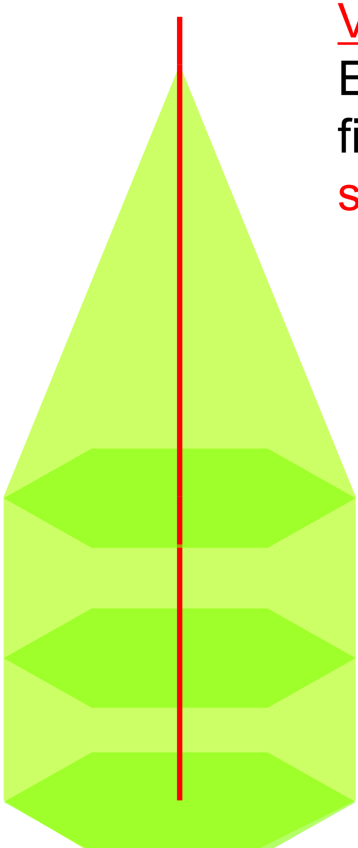
Vertical (c-axis) growth proceeds from top surface of large-area seed via thousands of dislocations. (i.e., dislocation-mediated growth!)

Crystal grown at T > 2200 °C
High thermal gradient & stress.

Limited crystal thickness.

Proposed LTC Growth Process

(US Patent 7,449,065 OAI, Sest, NASA)



Vertical Growth Process:
Elongate small-diameter fiber seed grown from single SiC dislocation.

Lateral Growth Process:
CVD growth to enlarge fiber sidewalls into large boule.
- 1600 °C, lower stress
- Only 1 dislocation

Vertical & Lateral growth are simultaneous & continuous (creates tapered shape).

Radically change the SiC growth process geometry to enable full SiC benefit to power systems.

Objectives

Overall Objectives (Longer Term)

- Open a new technology path to production of large-diameter SiC and GaN wafers with 100-1000 fold total crystal defect (dislocation) density improvement at 2-4 fold lower cost. (Present SiC wafers ~ 10^3-10^4 total dislocations per cm^2.)
- Enable leapfrog improvement in wide bandgap power device capability and cost to enable game changing improvements in electric power system performance (higher efficiency, smaller system size).

Funded Project Objectives (Shorter Term)

- Demonstrate initial feasibility of radically new "Large Tapered Crystal" (LTC) approach for growing vastly improved large-diameter SiC semiconductor wafers.
- Verify LTC growth physics in laboratory setting:
    - Growth of long, small-diameter single-crystal 4H-SiC fibers.
    - Lateral "M-plane" enlargement of 4H-SiC fibers into boules.
  - Both above demonstrations represent experimental firsts for hexagonal SiC.

Milestones

SiC experimental demonstrations of the two critical growth actions required for Large Tapered Crystal (LTC) process.

Month/Year*	Milestone
September 2012	Demonstrate epitaxial radial (lateral) growth of a 5 mm diameter boule starting from a simulated SiC fiber crystal.
September 2013	Demonstrate laser-assisted fiber growth of a SiC fiber crystal greater than 10 cm in length.

LTC is **NOT** viable without success of BOTH processes.

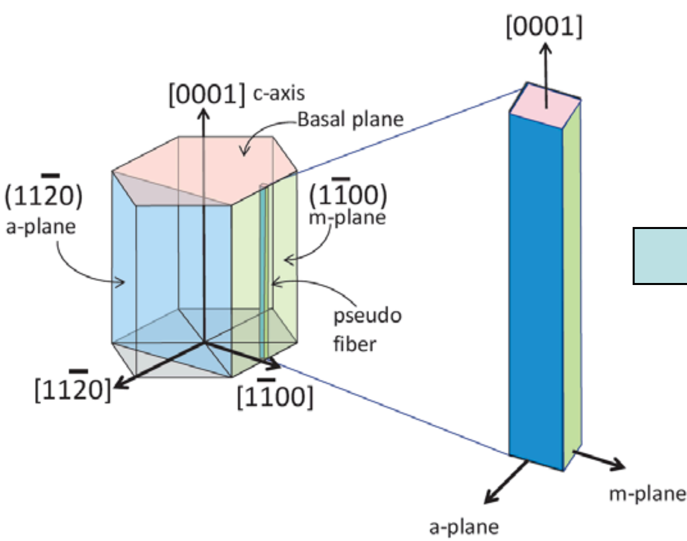
As discussed in this presentation, neither above quantitative milestone challenges have been met within the original project schedule.

* Interagency Agreement Amendment #2 Executed June 2012.

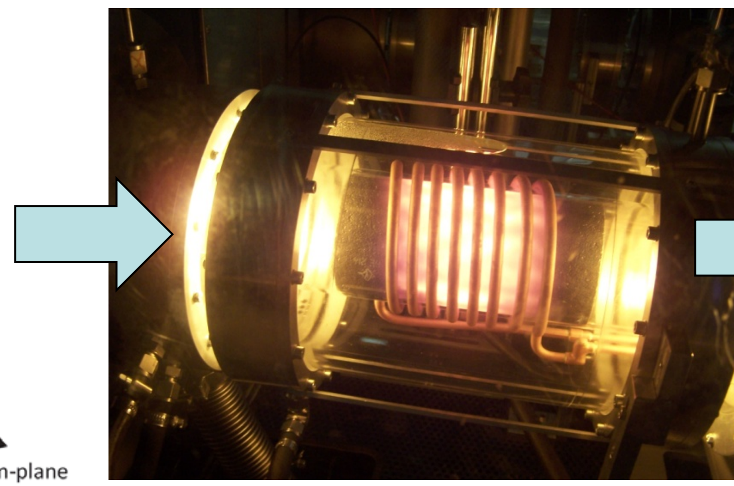
Technical Accomplishments and Progress

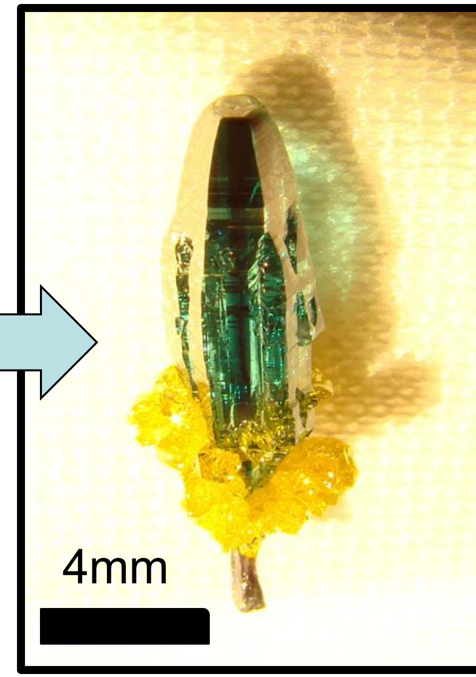
Radial/Lateral CVD Epi-Growth (viewgraph of FY12 Progress)

Radial/Lateral growth experiments to date have been conducted starting from "seed slivers" (i.e., "psuedo fibers") cut from commercially purchased a-plane and m-plane SiC wafers.



Hot-Wall CVD Growth





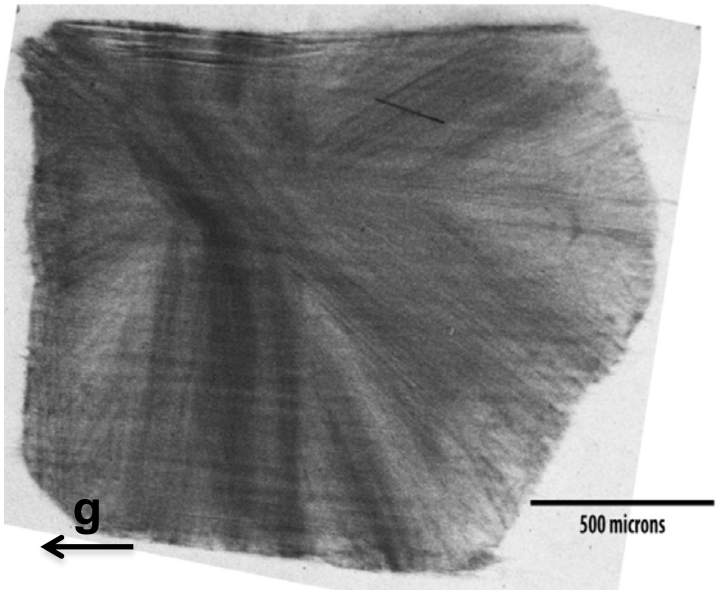
A 4 mm diameter 4H-SiC "mini-boule" was grown using this process during FY12, but sample exhibited abundant parasitic 3C-SiC growth in region where sliver is held by graphite carrier.

Technical Accomplishments and Progress

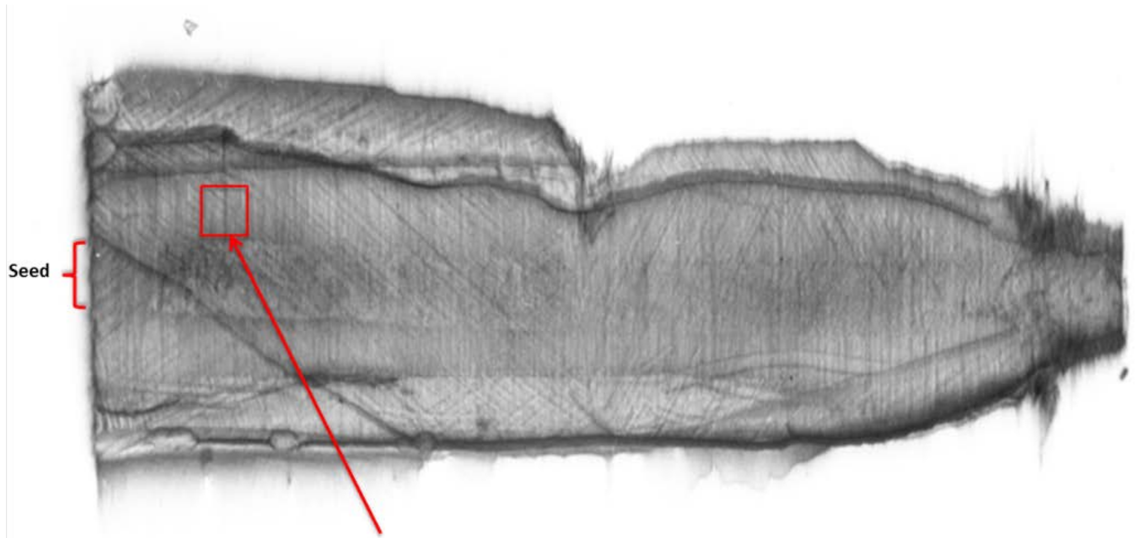
Radial/Lateral CVD Epi-Growth

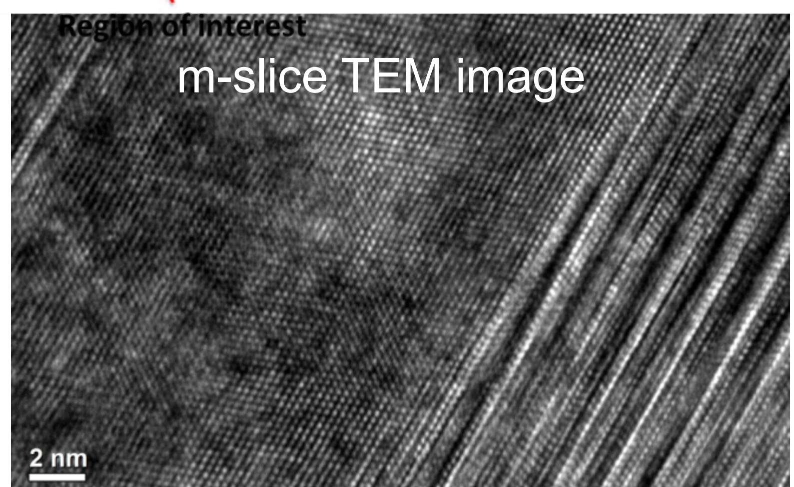
Crystal Characterization by Stony Brook University (Prof. Dudley's Group)

X-ray image of c-plane slice



X-ray image of m-plane slice





Abundance of defects observed propagating from seed crystal defects.

Need to study much thicker epilayers grown on much better seed crystals.

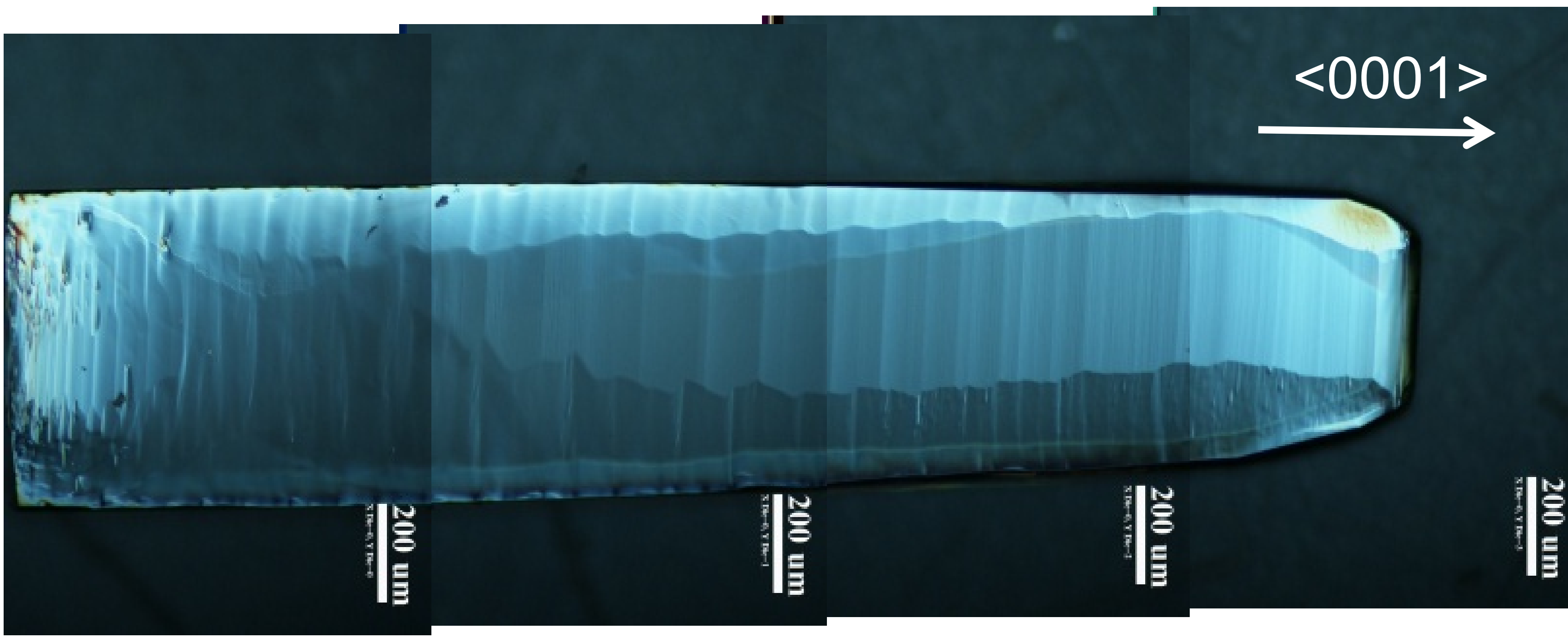
Technical Accomplishments and Progress

Radial/Lateral CVD Epi-Growth

Significantly improved 4H-SiC seed crystals, cut from a-plane and m-plane 4H-SiC slices purchased from Dow Corning.

Improved TaC coated reactor hot-zone hardware.

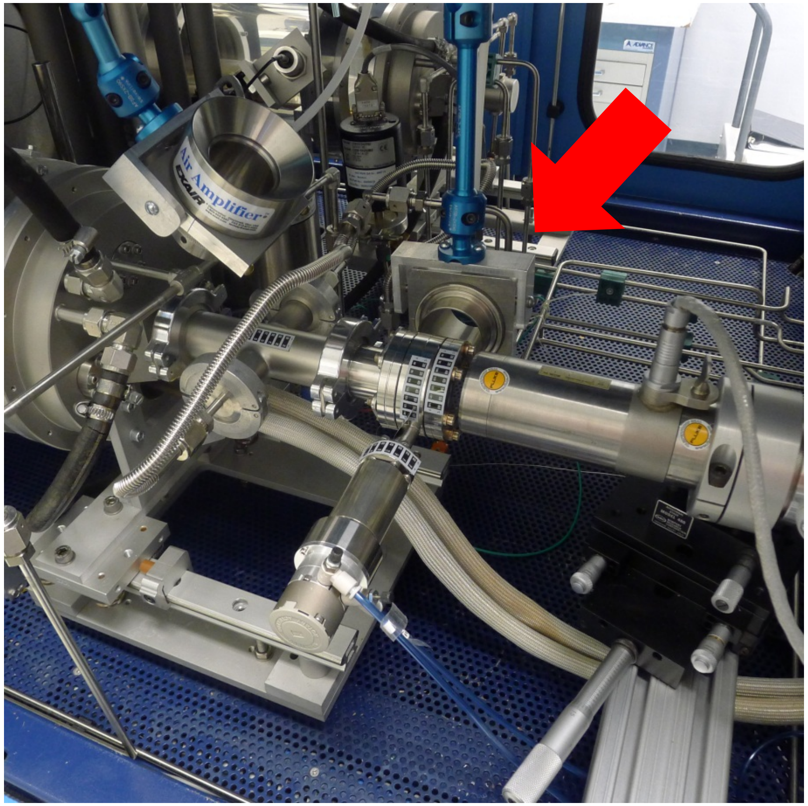
Initial growths have shown desired reduction in unwanted 3C-SiC growth in the 4H-SiC radial epilayers compared to prior experiments.

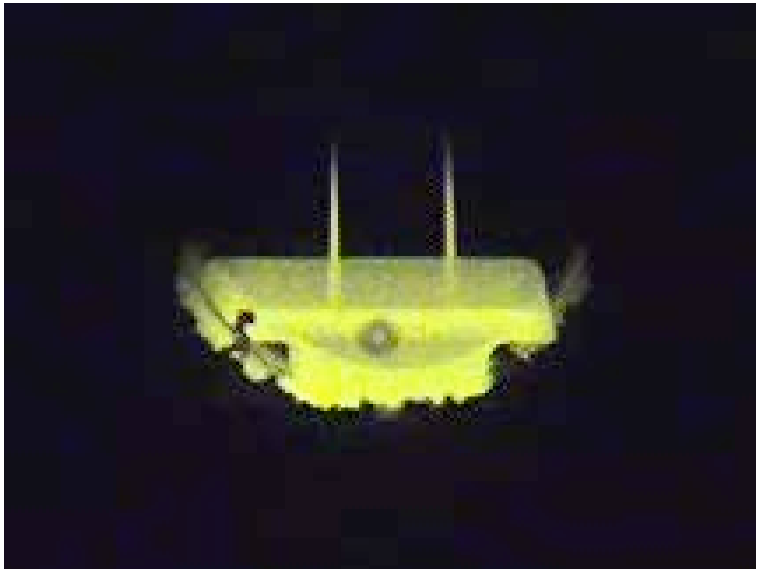


Technical Accomplishments and Progress

Radial/Lateral CVD Epi-Growth

Added thermal imaging capability to hot-wall CVD epitaxial growth reactor.





Thermal image of TaC-coated carrier and 4H-SiC seed slivers inside NASA hot-wall CVD reactor during T > 1400°C pre-growth H_2/HCl etch.

New capability key to control and understanding of radial CVD growth, especially during planned longer-duration runs to realize larger-diameter mini-boules.

Technical Accomplishments and Progress

Two toxic gas detection alarm events during that past year have severely delayed CVD radial growth experimental progress.

Both detections were below the ACGIH recommended limits for silane and HCl and occurred upon hot-wall reactor opening during maintenance activities.

HCl alarm on 21 May 2013 confined inside the sample loading glovebox area.

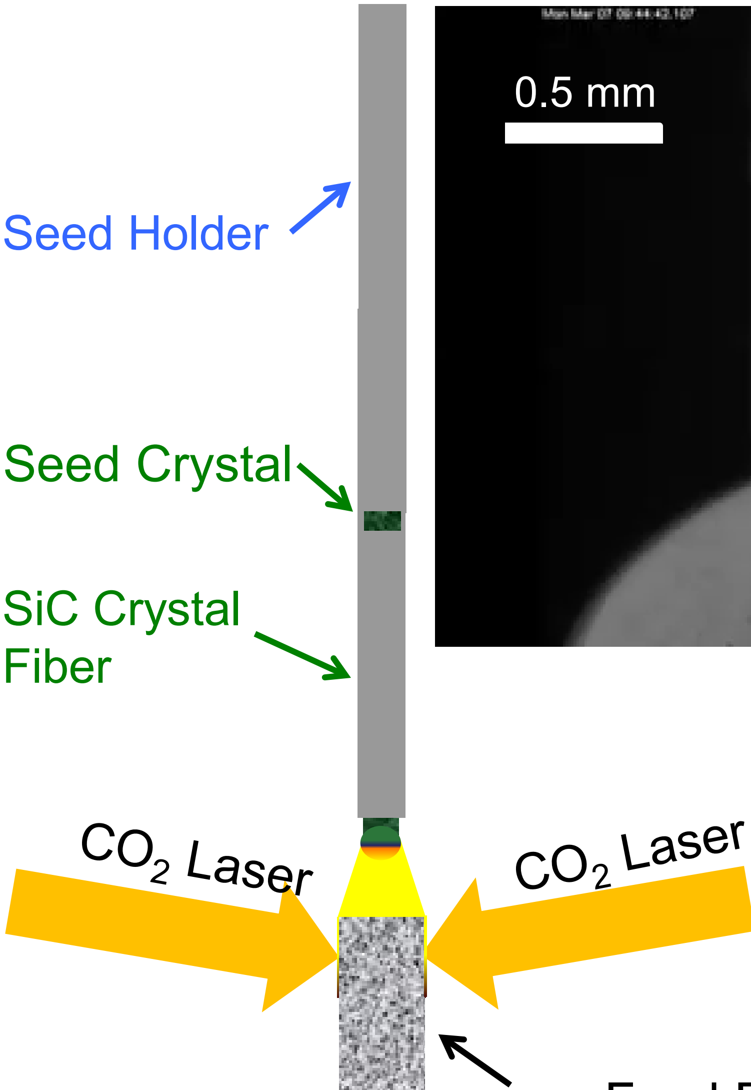
- Growth experiments did not resume until 1 November 2013 (5+ months downtime) due to repair and remediation activities.

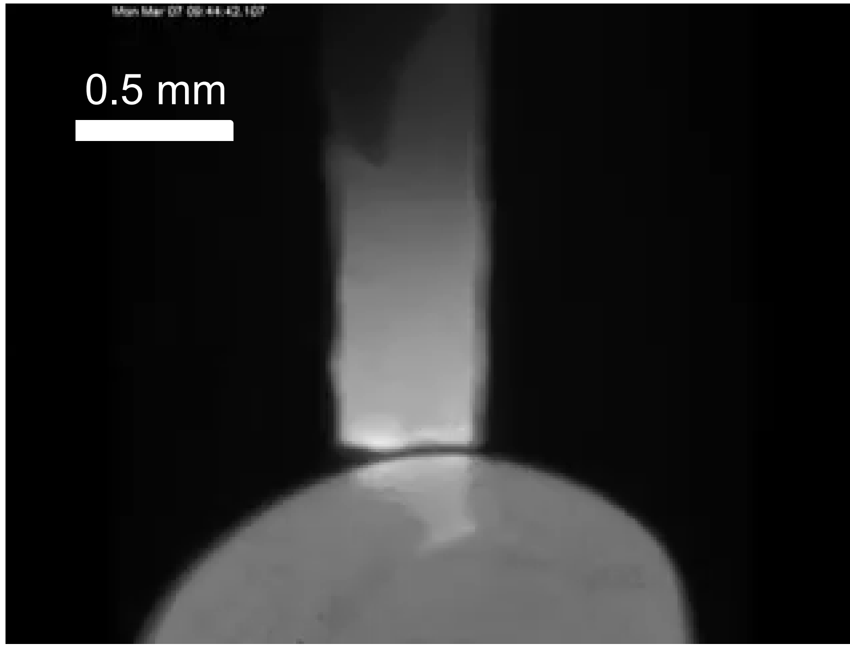
During maintenance activities on 20 January 2014 silane was detected near the reactor.

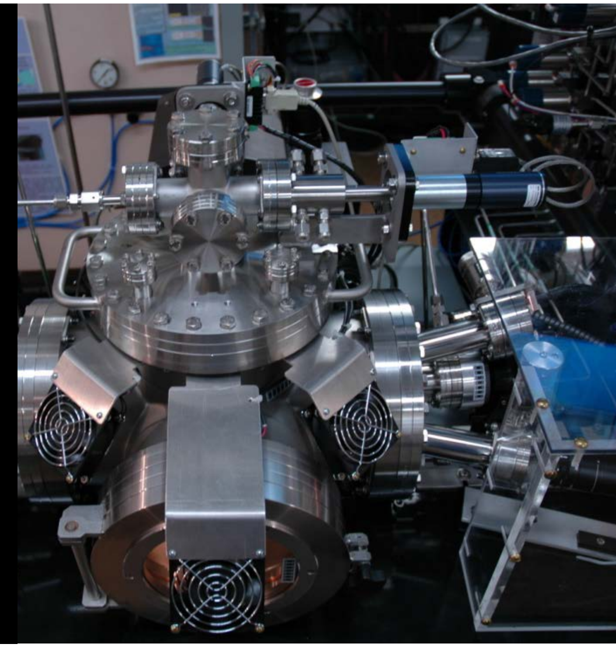
- Growth experiments have not resumed as of 15 April 2014 (3 months downtime).
- Independent safety teams have completed incident review and remediation actions are expected to be complete May 2014.

Approach/Strategy

(Solvent-LHFZ) - A New and Unique SiC Fiber Growth Method







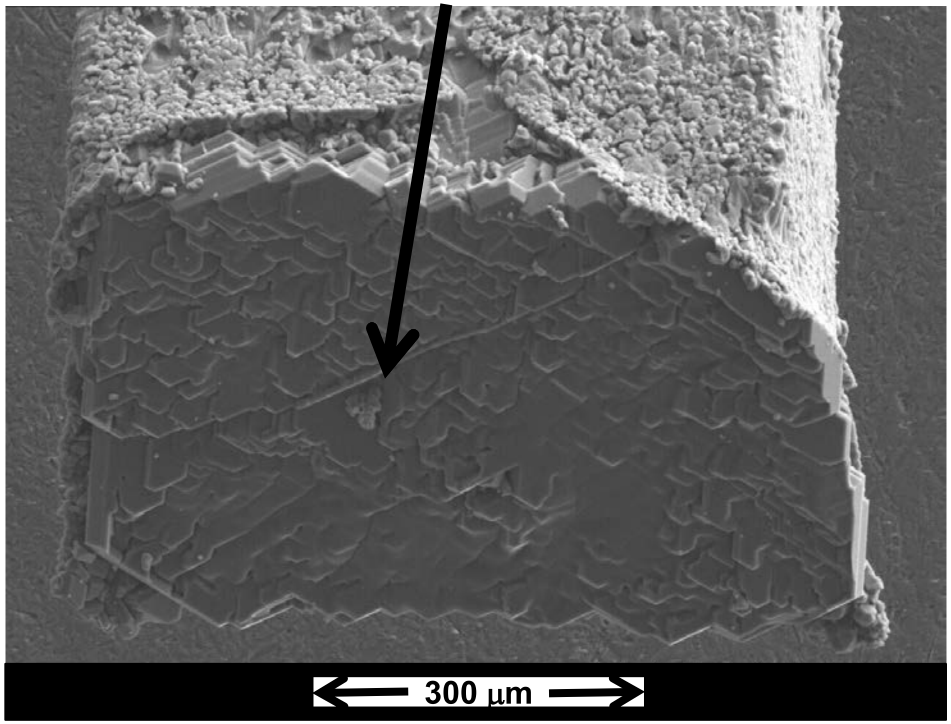
Combines the advantages of Traveling Solvent Method (TSM) & Laser Heated Floating Zone (LHFZ)

- TSM: Known SiC growth method
- LHFZ: Semi-infinite growth material

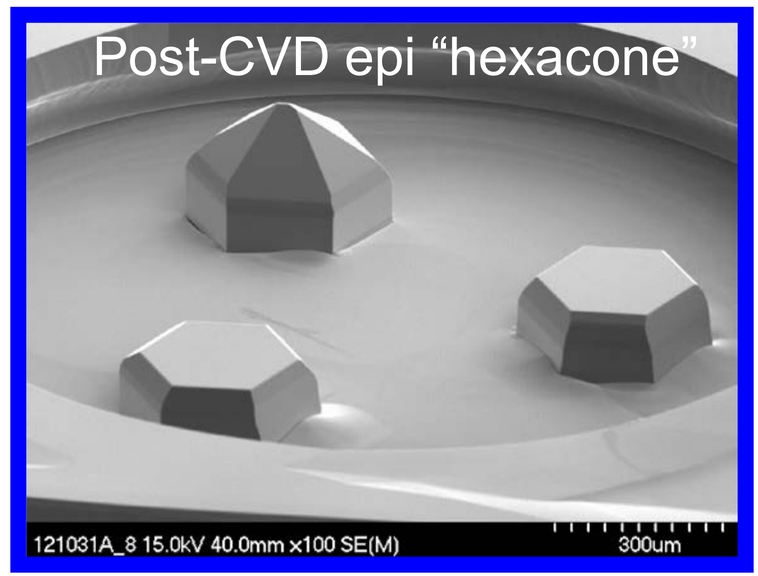
Technical Accomplishments and Progress

Fiber Growth via Solvent-LHFZ (Data from 2012 & 2013 Reviews)

First Solvent-LHFZ growths of 4H-SiC were carried out on large saw-cut seeds. These resulted in highly disorganized growth fronts and layer defects.



Seeking to use ordered growth front of 4H-SiC "hexacone" (below, grown via CVD epitaxy on patterned SiC mesas) for Solvent-LHFZ growths.



Source feed rod diameter must be greatly shrunk (about 4X) in order to achieve desired wetting to single patterned hexagonal SiC seed mesa.

Technical Accomplishments and Progress

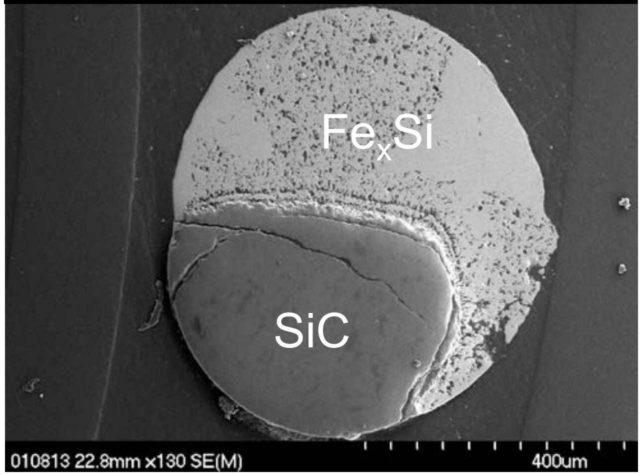
Fiber Growth via Solvent-LHFZ

Extrusion-based feed-rod fabrication method developed to achieve needed 4X shrink of feed rod diameter.

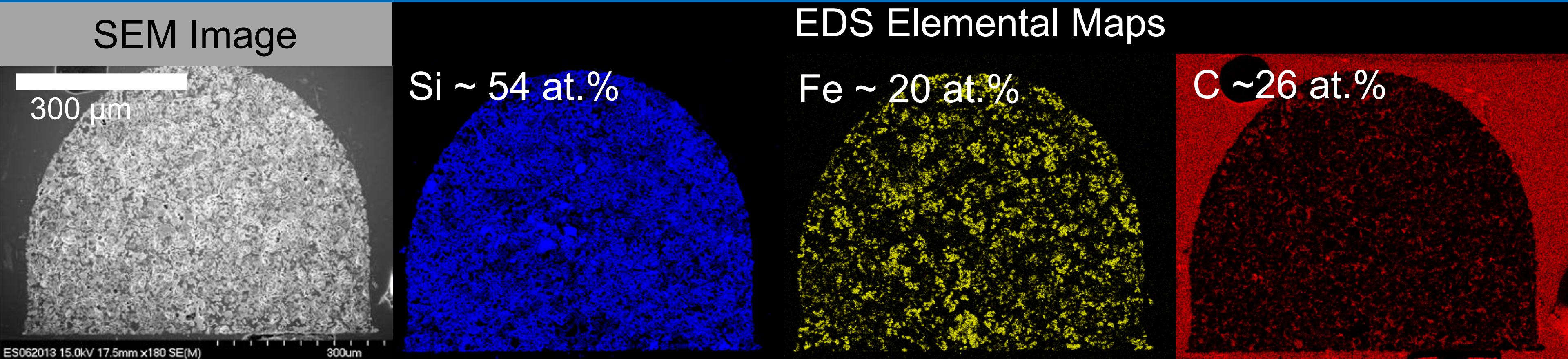
- Four other feed rod fabrication approaches were iteratively investigated and discarded.

- Extrude powders using a dispersant binder system.
- Dispersant/binder system requiring single step burnout and sintering procedure
  - Feed rod diameters as low as 0.25 mm.
  - Acceptable composition and density.
  - Improved control of C content still under investigation.

Failed SiC rod +Fe powder experiment



Cross sectional SEM image & EDS elemental analysis of successful extrusion and sintering of Si-Fe-graphite feed rod (sample embedded in epoxy)



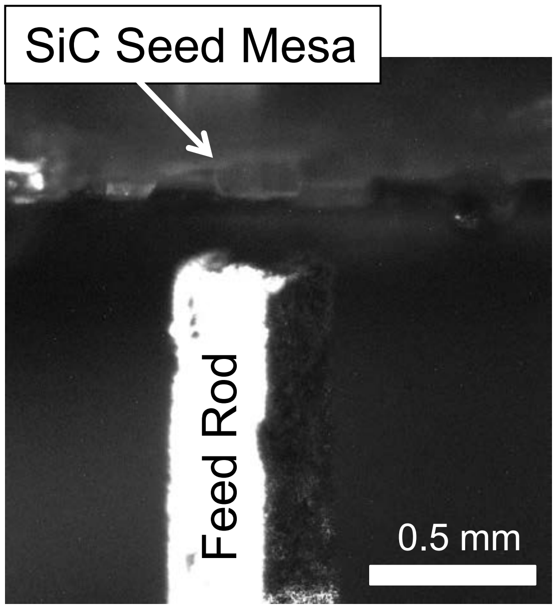
Technical Accomplishments and Progress

Fiber Growth via Solvent-LHFZ

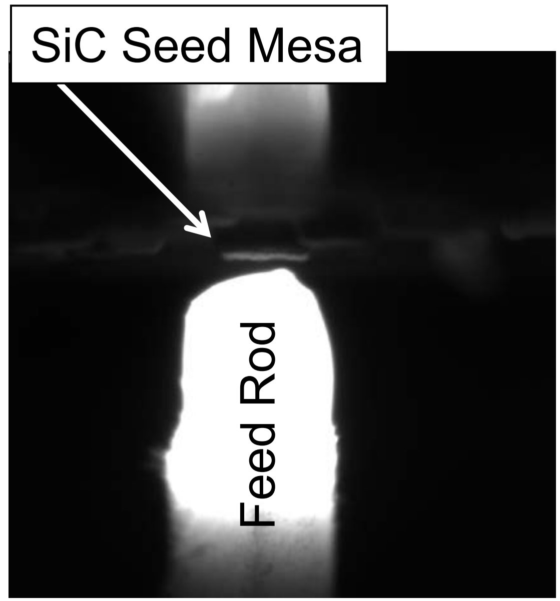
Completely new Si-Fe-C Solvent-LHFZ growth source feed rod process developed.

- Achieved needed 4X reduction in source feed rod diameter.
- First demonstration of good wetting to single patterned hexagonal SiC seed mesa.

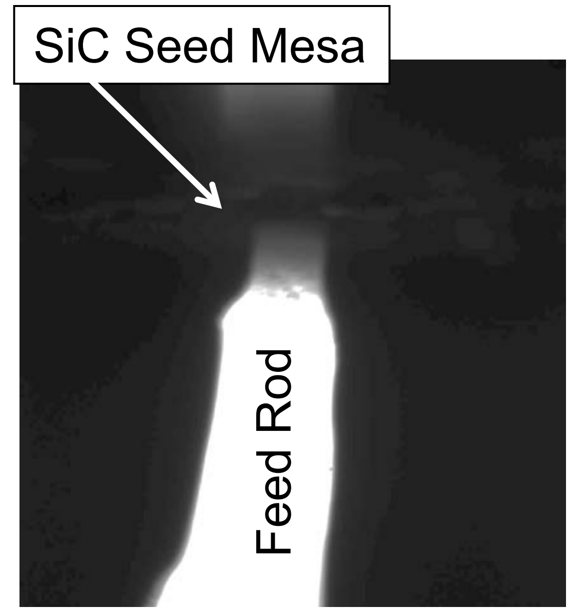
(Images of single-mesa wetting experiment)



Pre-growth: un-melted feed rod beneath patterned SiC seed mesas.



Melt formed on tip of feed rod just prior to contact with patterned SiC seed mesa.



Melted feed rod tip successfully wetted to single hexagonal patterned SiC seed mesa.

Proposed Future Work

Radial growth of larger (> 5mm diam.) mini-boules to retire overdue project metric.
- Greatly improved seed crystals combined with TaC coated reactor parts and optimized growth conditions to suppress parasitic 3C-SiC nucleation.
  - Assistance from thermal imaging of samples and reactor hot-zone.
- This will enable longer growth times needed to produce thicker/larger radially-grown 4H-SiC “mini-boule” crystals.
- Thorough characterization studies of larger mini-boules to demonstrate “defect free” LTC radial growth expansion feasibility (Stony Brook U.).

Fiber growth demonstration will become focus of further work at NASA.
- Continue attempts at Solvent-Laser growth method, but if demonstration fibers cannot be produced, other growth approaches must be considered.
  - Laser-assisted CVD (building on initial FFF SBIR work).
- Systematically attack experimental condition control (including hardware) needed to achieve 4H-SiC fiber growth breakthrough demonstration.

Collaboration and Coordination with Other Institutions

- NASA Glenn Research Center (Prime)
  SiC crystal growth and ceramic fiber growth research branches
    - Ohio Aerospace Institute (Non-Profit)
    - Sest, Inc.
    - NASA Postdoctoral Program (Oak Ridge Assoc. Universities)

- State University of New York at Stony Brook – Brookhaven NY and Argonne IL X-ray sources (Dept. of Energy).
    - Prof. Dudley's group - recognized leader in X-Ray topographic mapping characterization of SiC crystals and defect structure.
- Carnegie Mellon University – Materials Transfer Agreement initiated March 2014 with Prof. Picard for Electron Channeling Contrast Imaging (ECCI) study of NASA-grown 4H-SiC seed crystal "hexacone" mesas.

- Free Form Fibers LLC (NY) – SBIR Phase III (NASA Funded $100K) for laser-assisted SiC fiber growth using gas precursors.
  - Small business laser-growing **polycrystalline** SiC fiber shapes.
  - Parallel path (risk mitigation) to realize **single-crystal** SiC fiber growth if technical challenges of Solvent-LHFZ approach cannot be overcome.
  - SBIR has ended WITHOUT SUCCESSFUL 4H-SiC FIBER GROWTH.

Summary

Progress continues towards SiC Large Tapered Crystal growth feasibility demonstration, but major project feasibility goal will NOT likely be accomplished prior to agreement expiration.

Radial growth feasibility milestone still on track to be accomplished.
- CVD downtime has severely delayed radial epitaxial growth progress.
- Improved seed crystals and CVD hardware will be used for further growths.

Fiber growth feasibility remains toughest challenge of LTC.
- No “breakthrough” fiber crystals yet at NASA or Free Form Fibers.
- Additional fundamental experiments & understanding required.
- Fiber growth milestone unlikely to be met during FY 2014.

LTC demonstration work expected to continue as NASA funding permits.

Technical Acknowledgements

NASA LTC Co-Investigators:
Andrew Woodworth (NPP/NASA),
Andrew Trunek (OAI/NASA),
Ali Sayir (NASA), Fred Dynsys (NASA),
David Spry (NASA).

NASA LTC Support Team:
Terry Ferrier (NASA),
Tom Sabo (Case Western/OAI),
Jim Mazor, Wentworth John, Kimala Laster, Beth Osborn &
Michelle Mrdenovich (Sierra Lobo Inc),
Scott Panko (Vantage Partners),
Chuck Blaha, Frank Lam & Kelly Moses (Jacobs Technology)

Dow-Corning for higher quality a-plane and m-plane wafers.

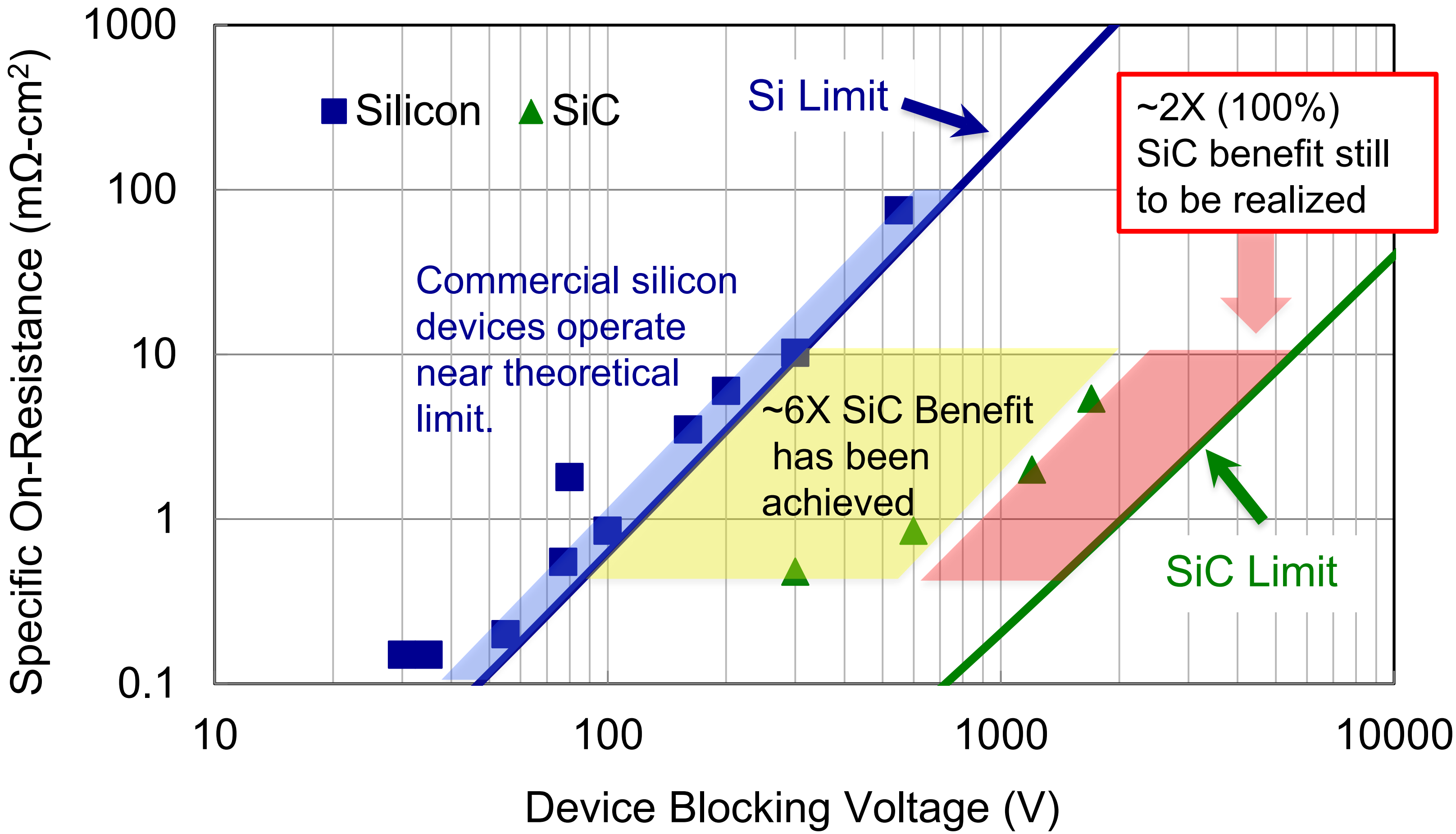
Technical Back-Up Slides

(Note: please include this “separator” slide if you are including back-up technical slides (maximum of five). These back-up technical slides will be available for your presentation and will be included in the DVD and Web PDF files released to the public.)

Unipolar Power Device Comparison

(Volume Production Commercial Devices)

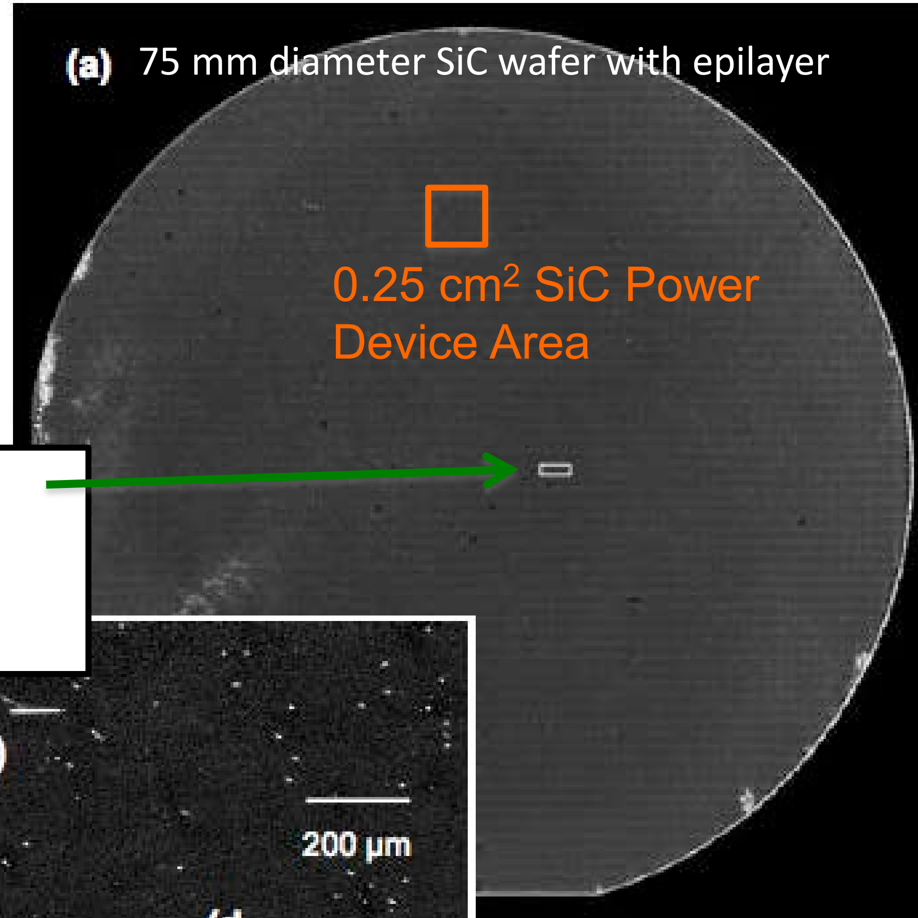
SiC devices are ~2X voltage or current-density **de-rated** from theoretical material performance.



Above comparison does NOT take yield, cost, other relevant metrics into account.

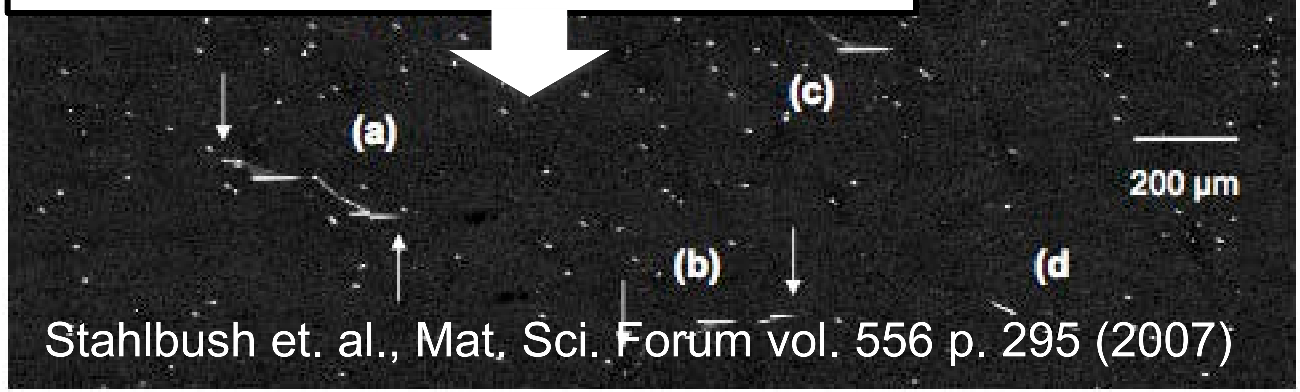
SiC Wafer Material Defects

Over the past decade there have been numerous studies (including NASA GRC) linking degraded SiC power device performance, yield, and reliability to the presence of defects in the SiC wafer crystal.



Magnified view small area in middle of wafer imaged by Ultra-Violet Photoluminescence

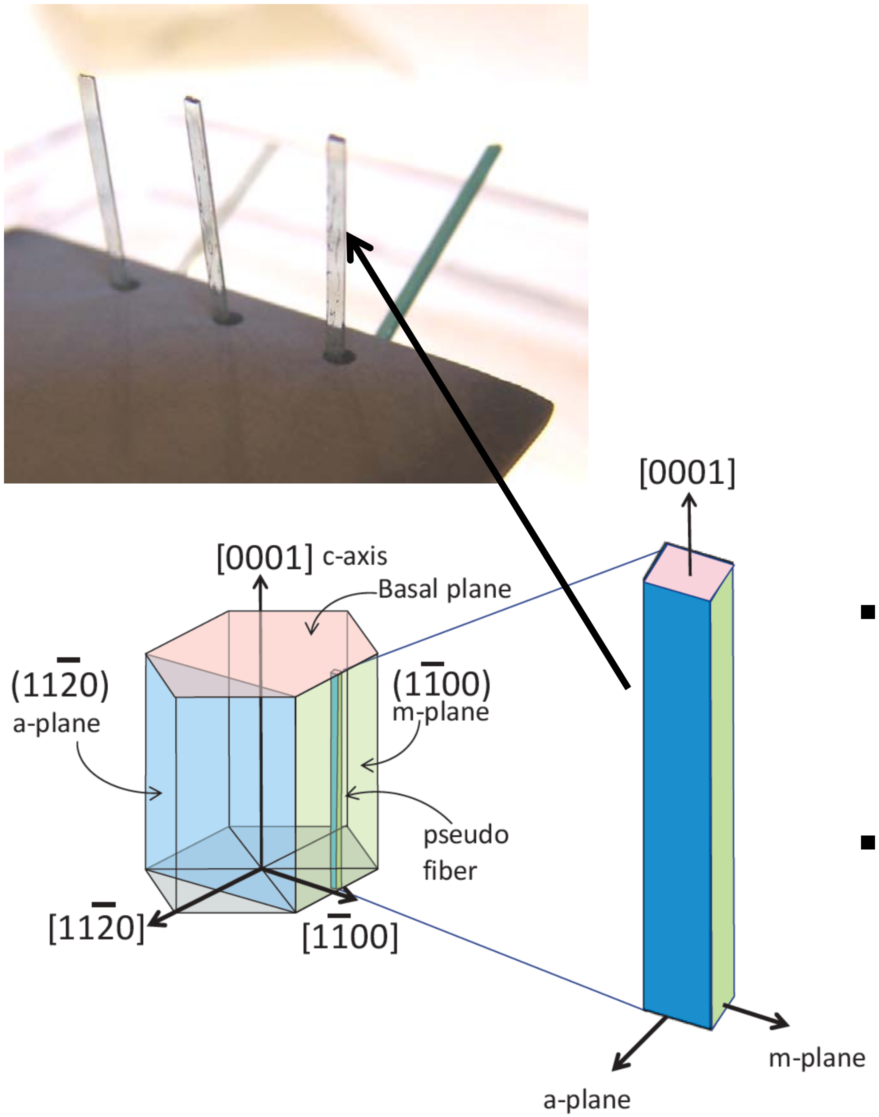
- Each white dot or line is a dislocation defect!
- Average dislocation density ~10^4 per cm^2



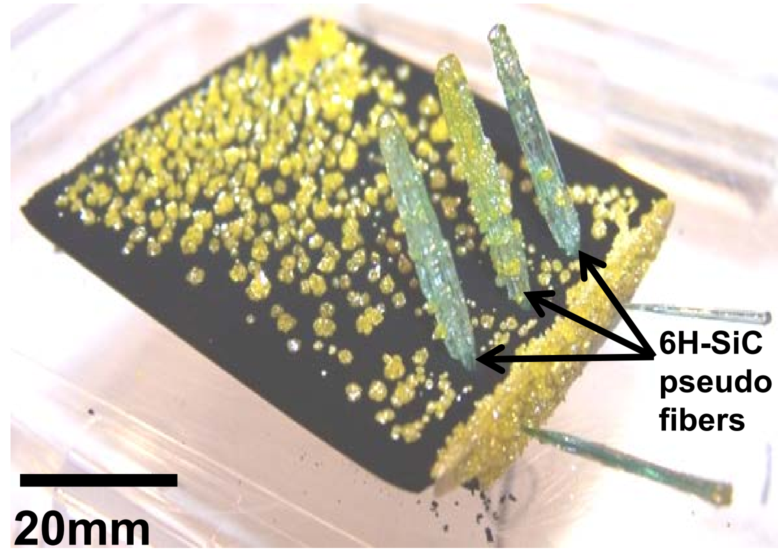
Technical Accomplishments and Progress

Radial/Lateral CVD Epi-Growth (viewgraph from FY12 Review)

4H/6H SiC a/m-plane slivers prior to growth



Slivers after 8 hours of CVD epitaxial growth



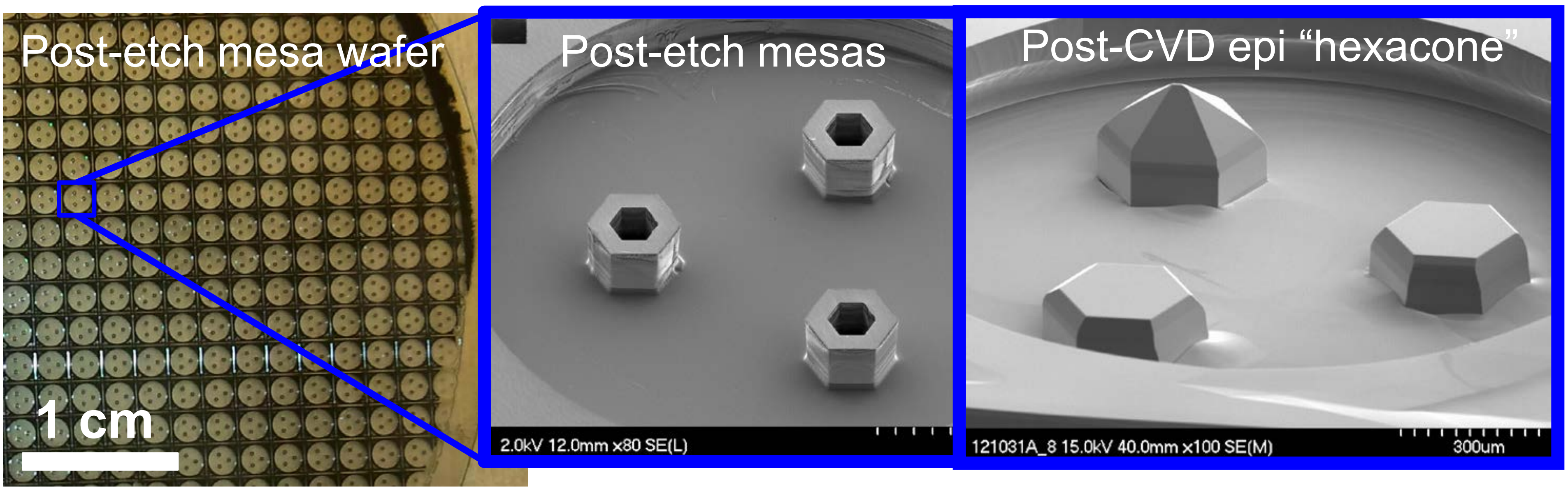
- Post-growth crystals are translucent and exhibit lateral expansion (a/m-face growth).
- 3C-SiC crystallites (yellow) undesirably nucleated in some areas.

Technical Accomplishments and Progress

“Hexacone” Mesa Seed Development

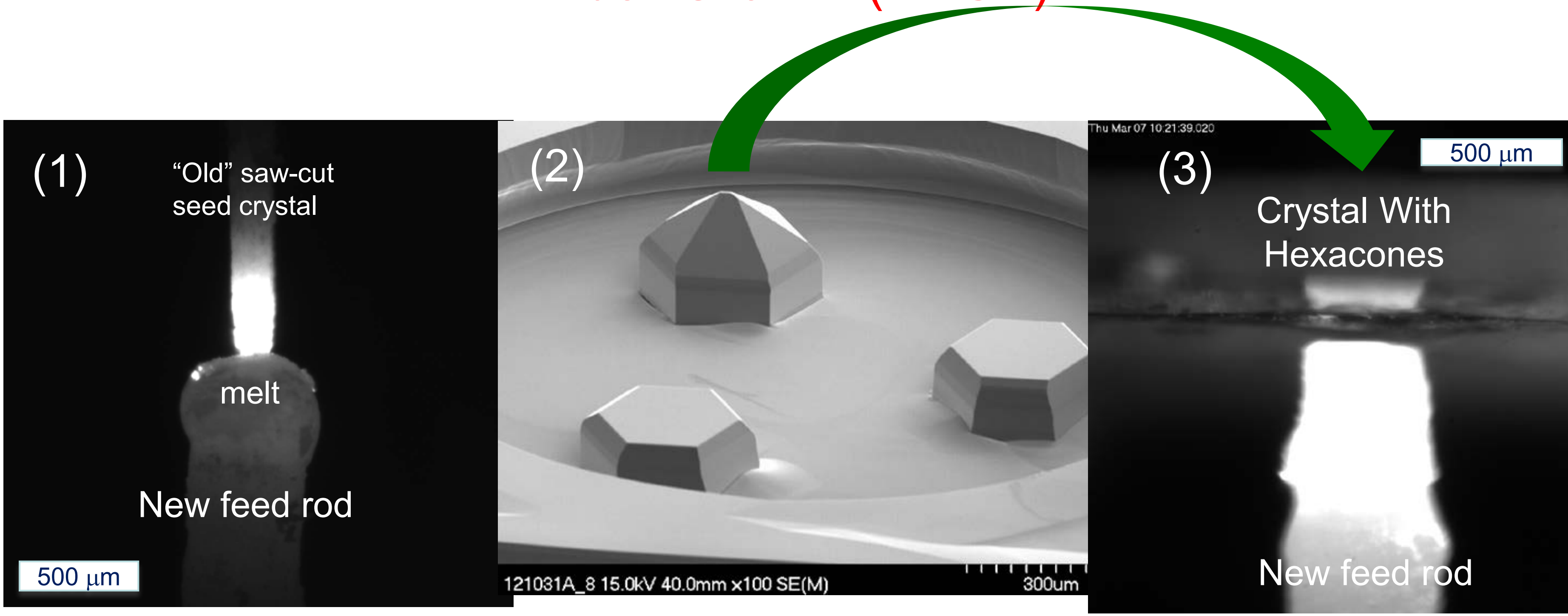
As described in LTC patent (US 7,449,065), both laser-solvent (NASA) and laser-CVD (FFF) fiber-growth approaches require (start from) a small initial 4H-SiC seed mesa with centrally located screw dislocation. **Nominal mesa seed for fiber growth is a pointed “hexacone” achieved via pure stepflow CVD homoepitaxy on mesa-etch patterned etched 4H-SiC wafer.**

Significant effort this year on refining deep dry etching and hot-wall CVD process for growing arrays of 4H-SiC mesa “hexacones” for seeding fiber growth.



Technical Accomplishments and Progress

Fiber Growth (NASA)



(1) New 4X smaller-diameter feed rods for solvent fiber growth developed.

(2) Improved SiC "hexacone seeds" needed to advance fiber development work grown via CVD epitaxy (delivered to Solvent-LHFZ lab & SBIR contractor).

(3) First Solvent-LHFZ experiments using (1) and (2) initiated (March 2013).

Fundamental Challenge: Can solvent process yield a stable single-domain growth front needed to realize a long 4H-SiC single-crystal fiber?