A New Method to Grow SiC: Solvent-Laser Heated Floating Zone

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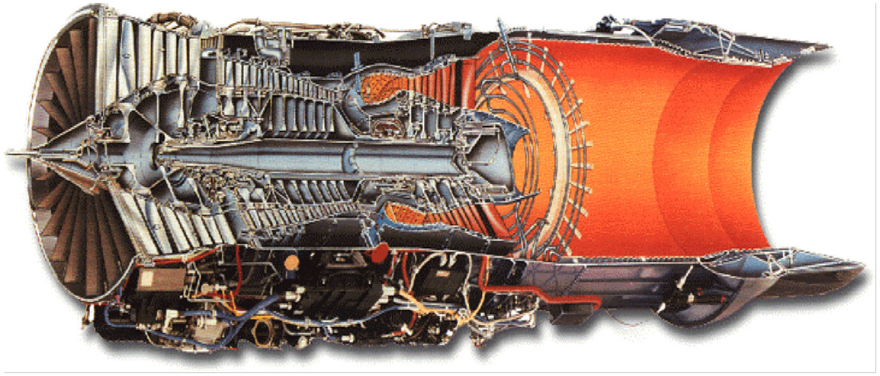
SiC Electronics Benefits to NASA Missions

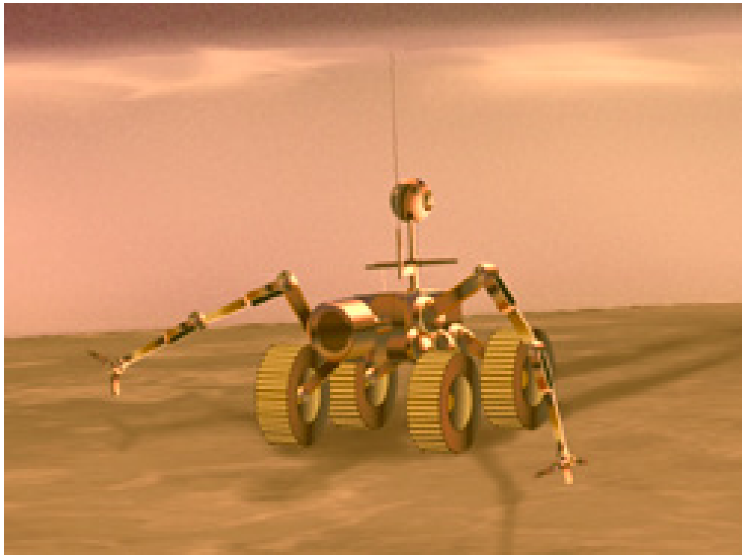
Intelligent Propulsion Systems



Space Exploration Vision PMAD

More Electric + Distributed Control Aircraft

Venus Exploration

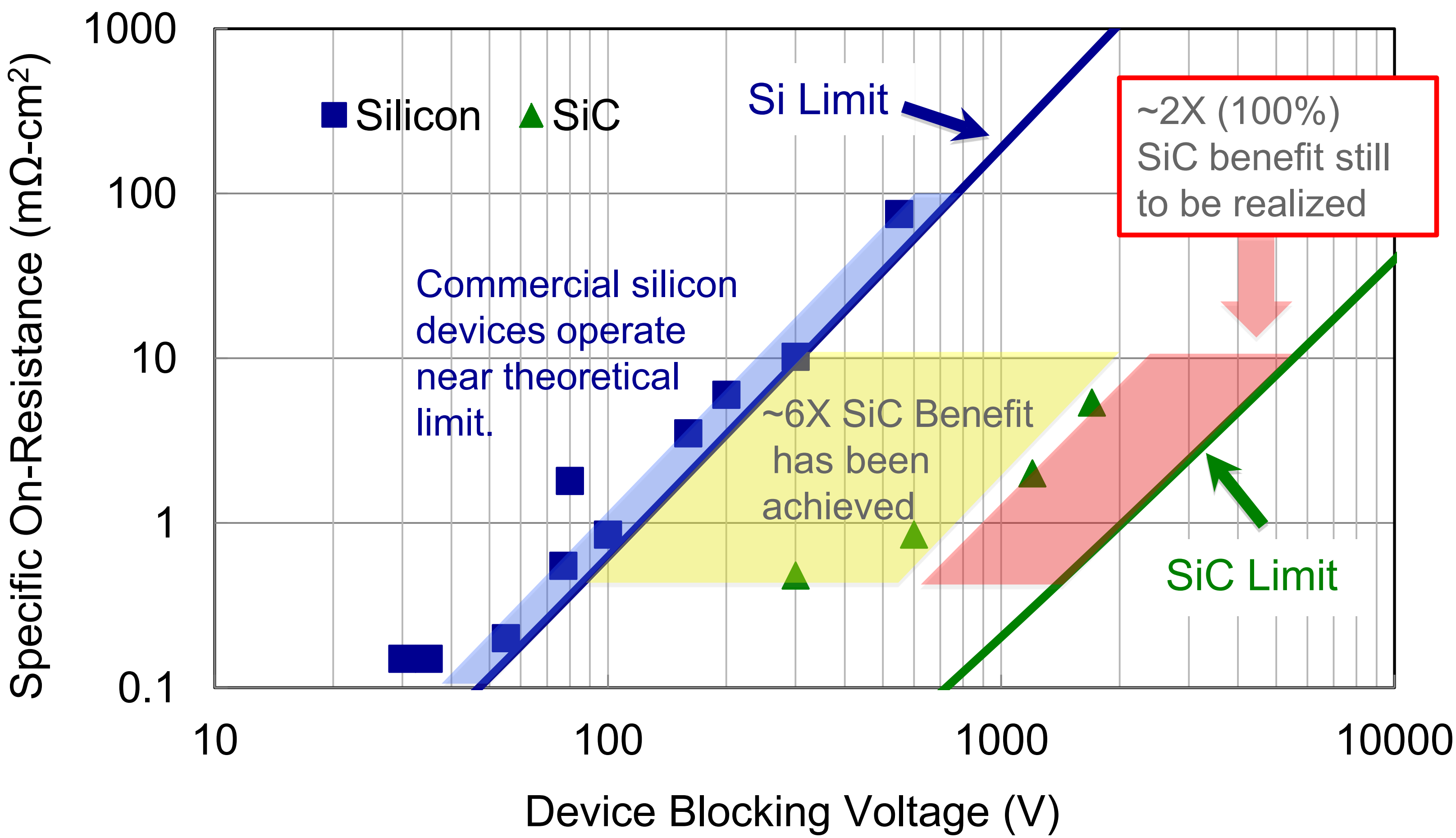


All combinations of <u>high temperature and/or high power</u> applications!

Unipolar Power Device Comparison

(Volume Production Commercial Devices)

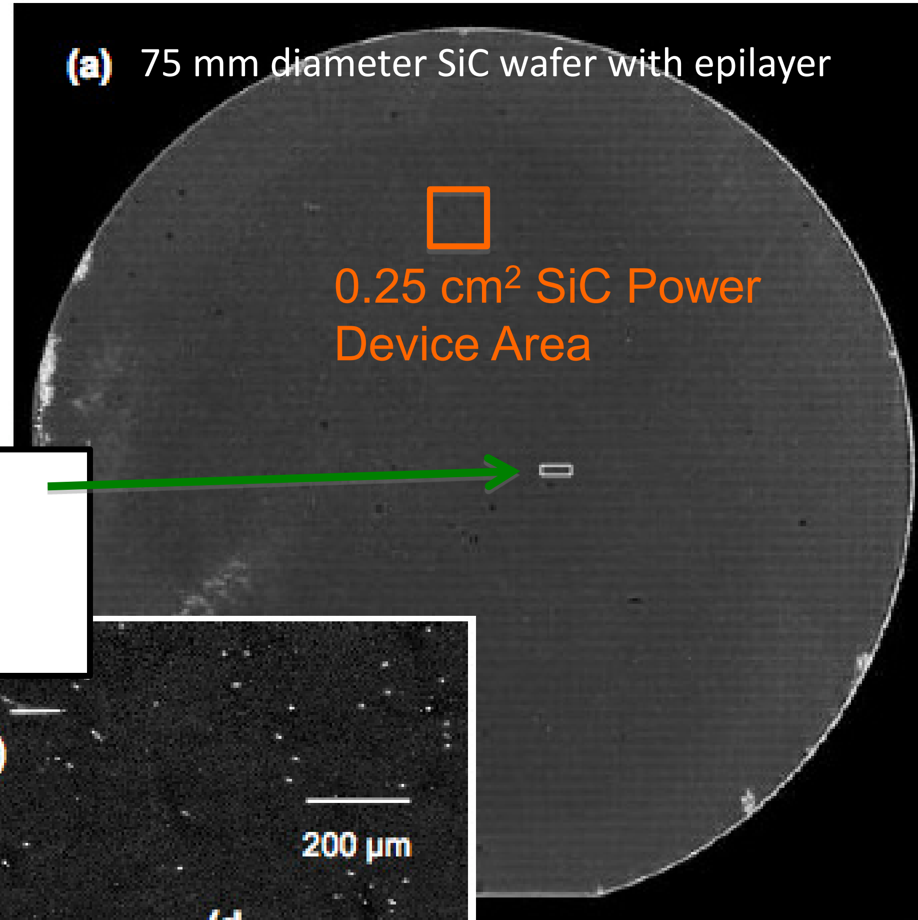
SiC devices are ~2X voltage or current-density **de-rated** from theoretical material performance.



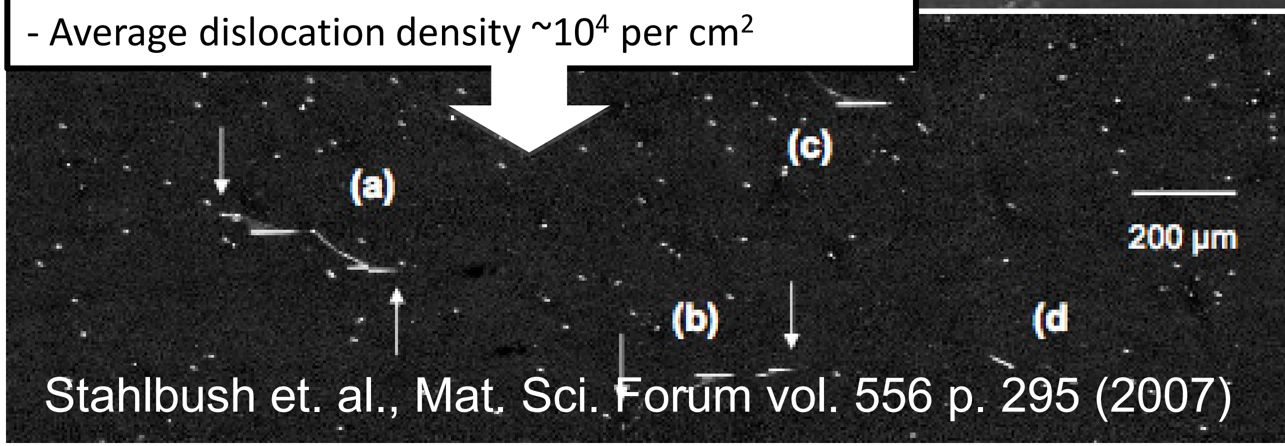
Above comparison does NOT take yield, cost, other relevant metrics into account.

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



Stahlbush et. al., Mat. Sci. Forum vol. 556 p. 295 (2007)

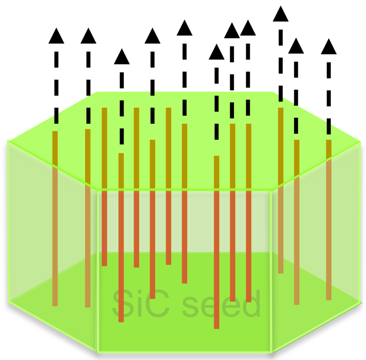
Two-fold defect-induced SiC device over-design roughly translates into corresponding energy loss and/or power circuit size increase trade-off.

Description of Technology/Approach

Large Tapered Crystal (LTC) SiC Growth

Present SiC Growth Process

(Vapor transport)



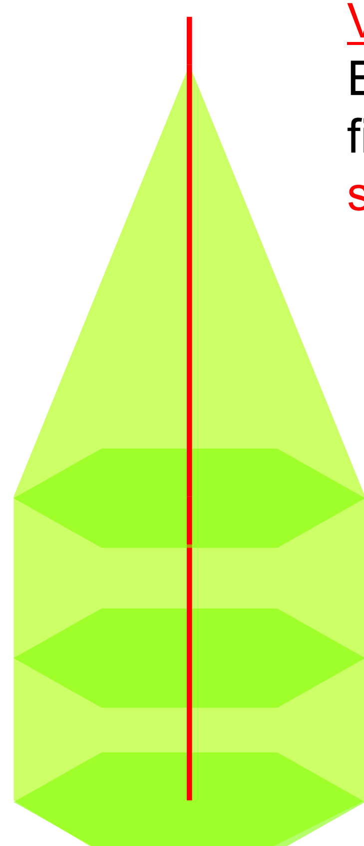
Vertical (c-axis) growth proceeds from top surface of large-area seed via thousands of screw dislocations.(i.e., dislocation-mediated growth!)

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.

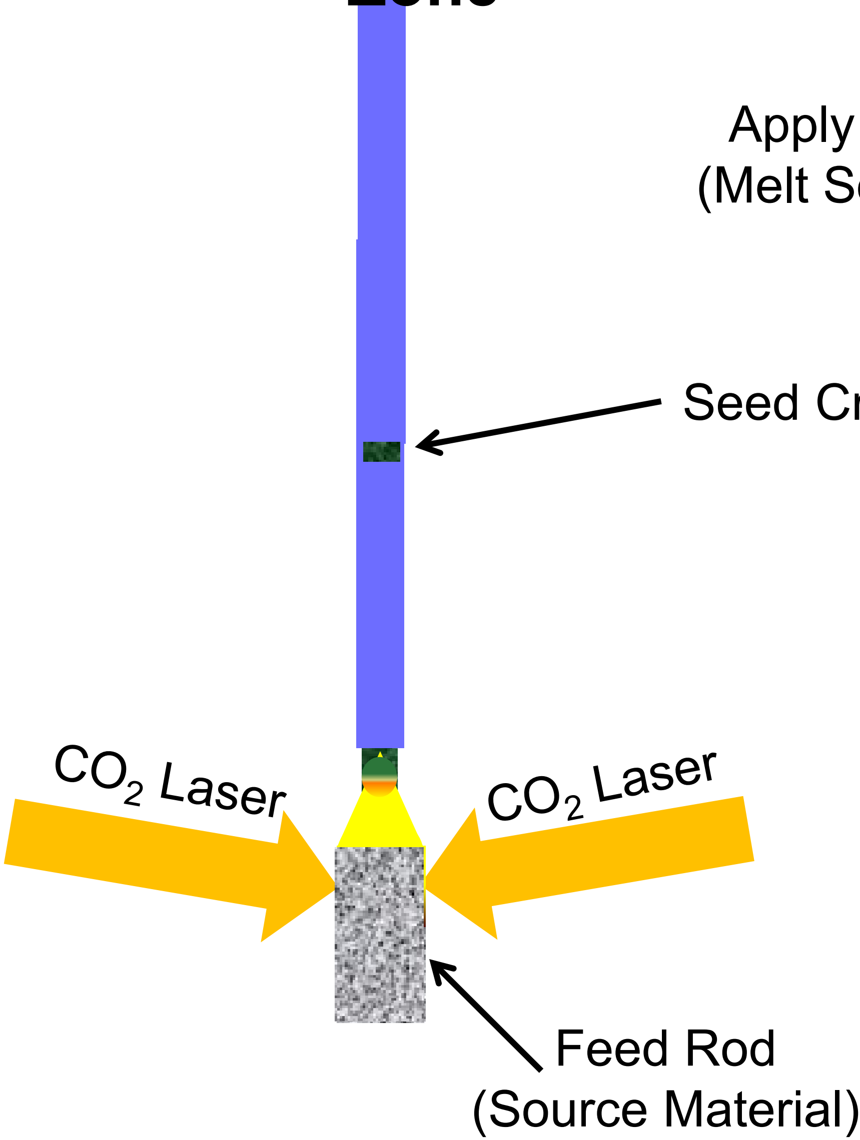
Lateral Growth Process:
CVD grow to enlarge fiber sidewalls into large boule.
- 1600 ° C, lower stress
- Only 1 dislocation

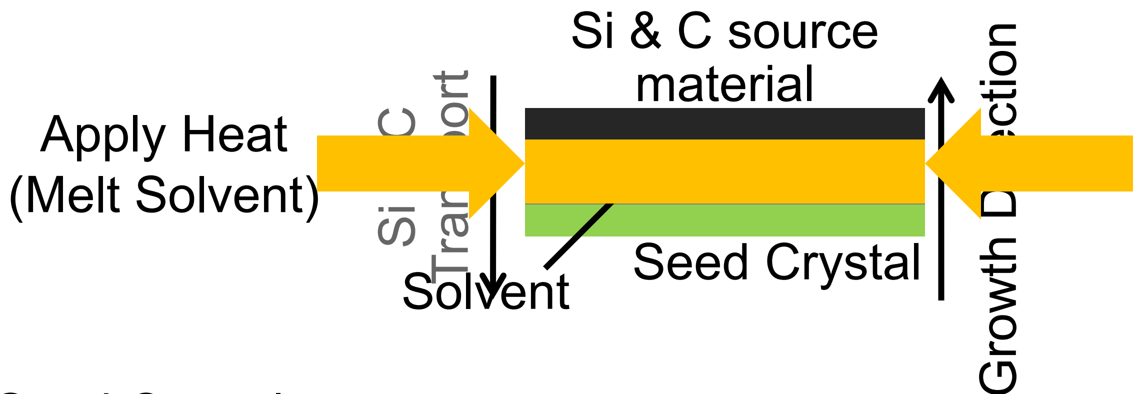
Lateral & vertical growth are simultaneous & continuous (creates tapered shape).

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

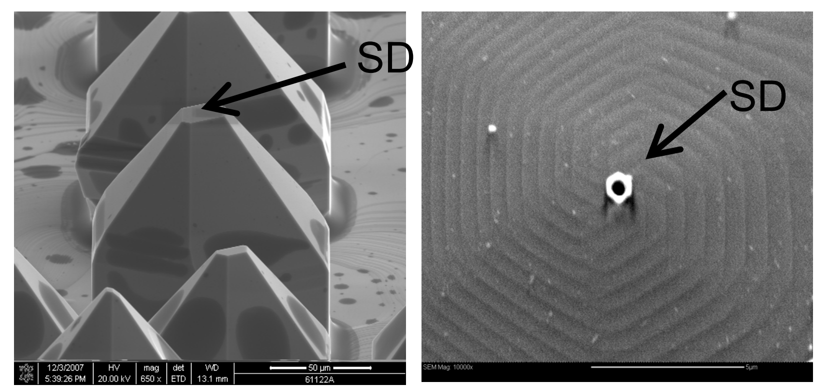
Solvent-LHFZ Technique

Laser Heated Floating Zone **+** **Solvent Growth Method**

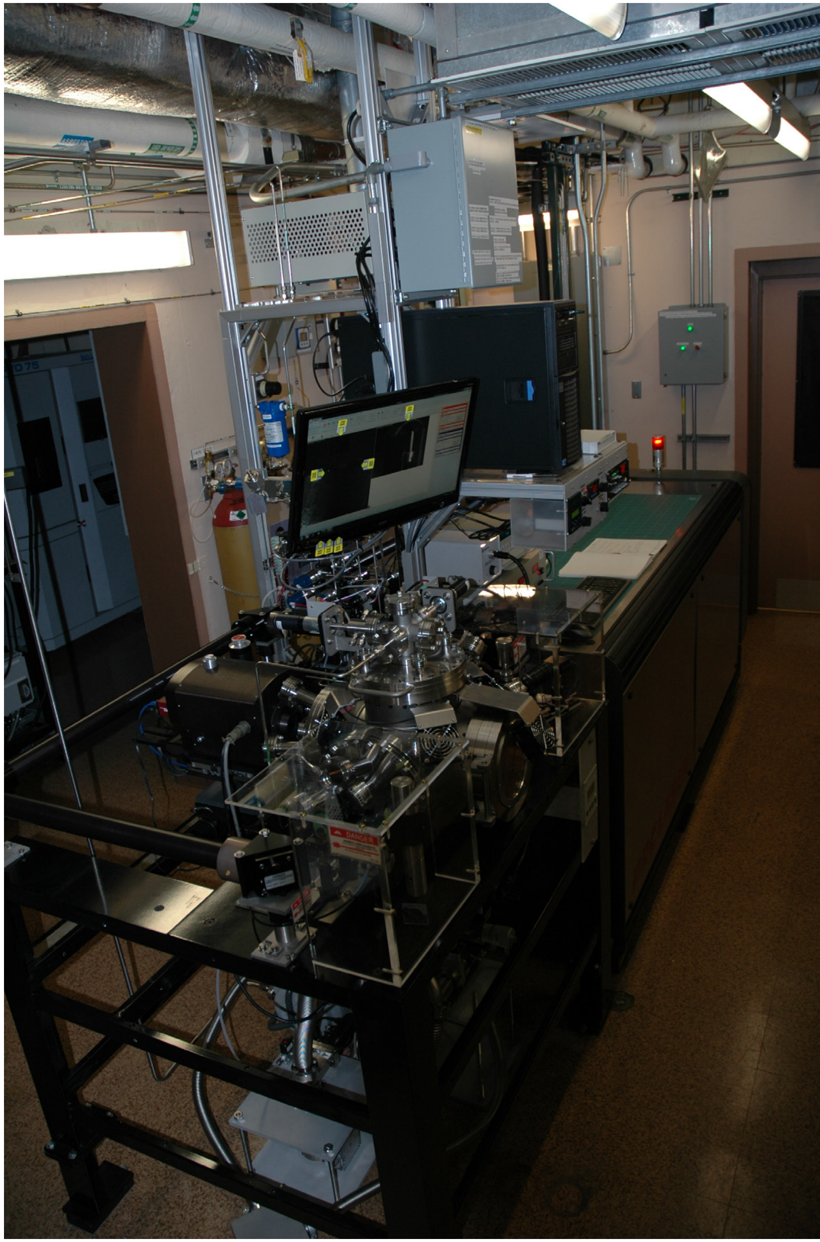




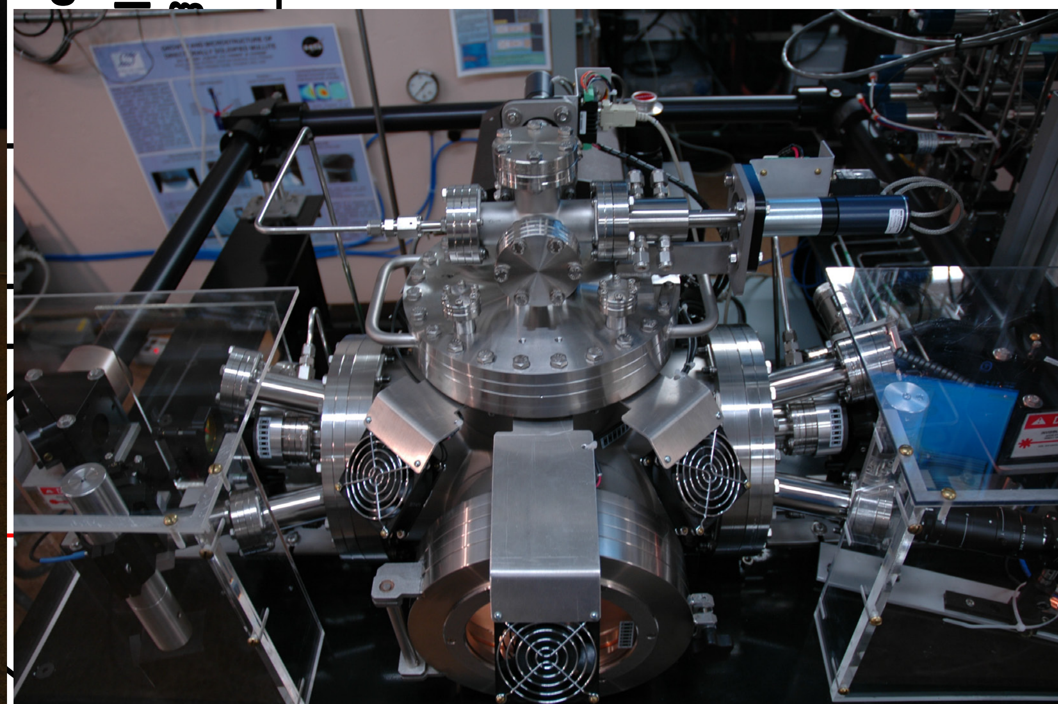
Seed Crystal



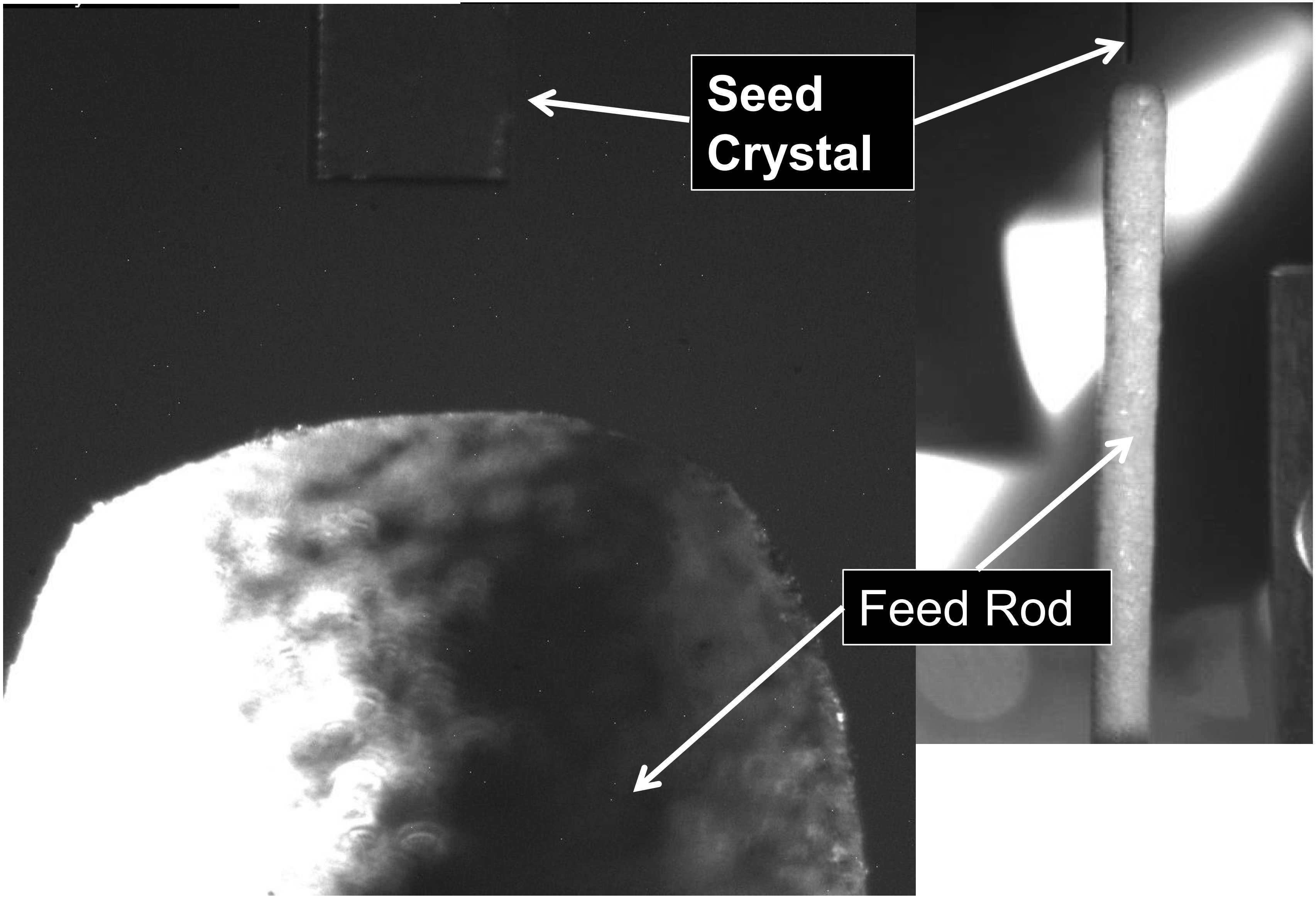
The Solvent-LHFZ System



Top Down View

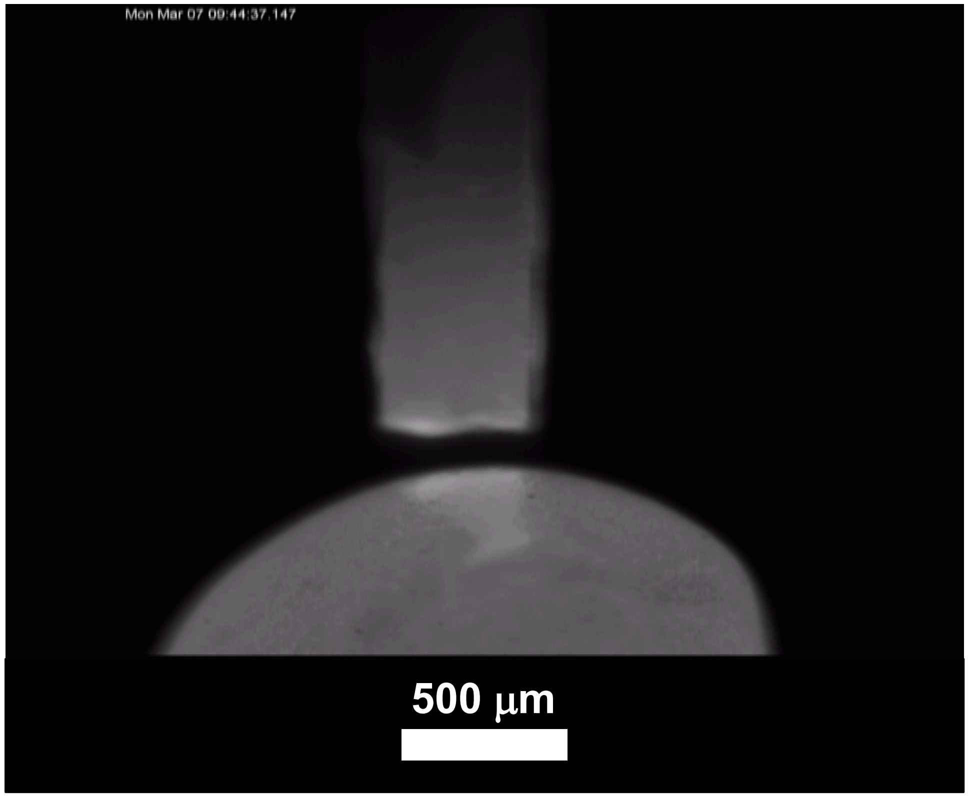


Growth Mechanics



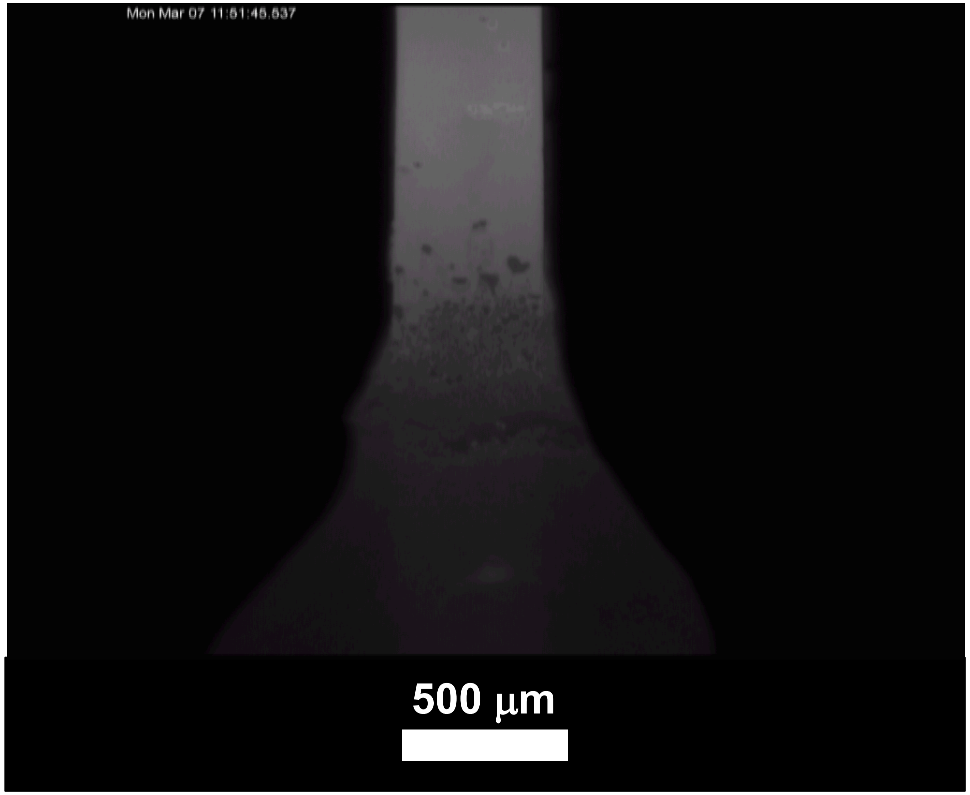
Growth Mechanics

Contact and Wetting

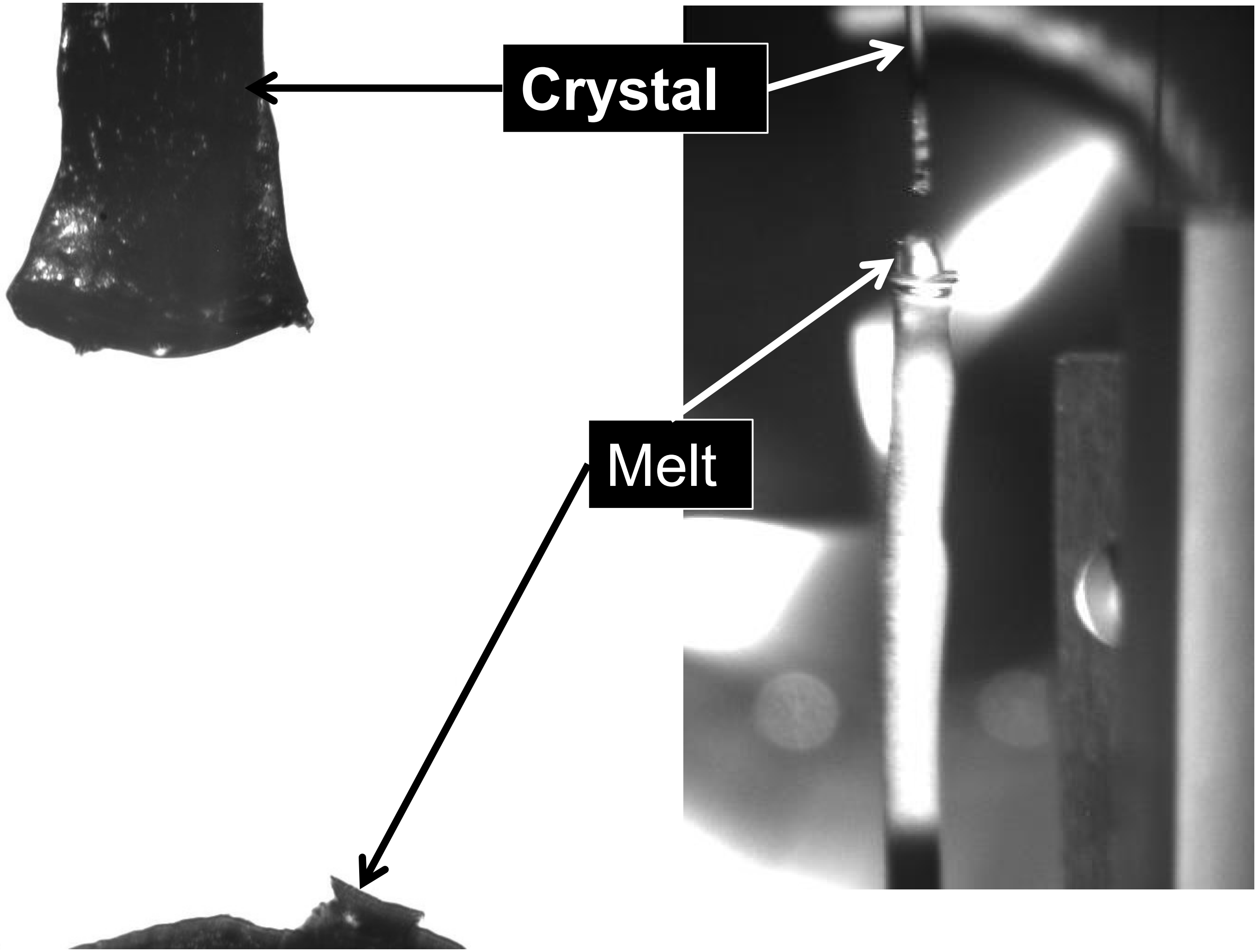


Growth Mechanics

End of Growth Experiment



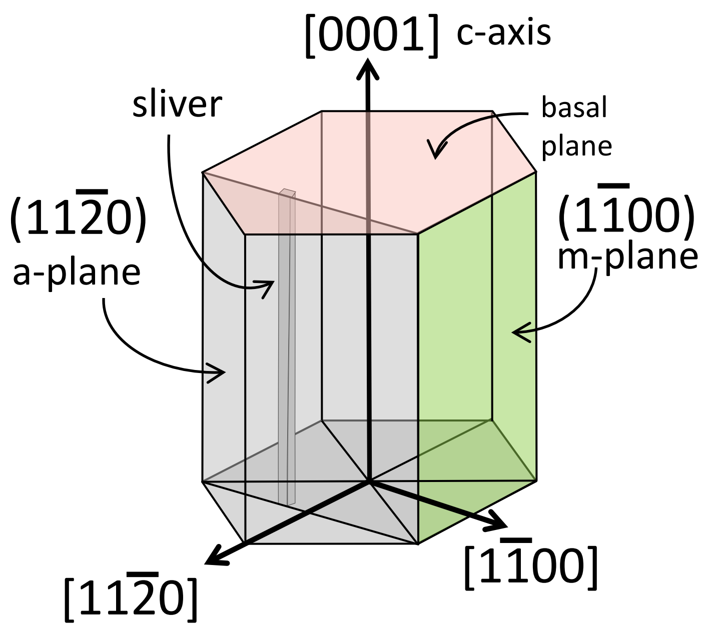
Growth Mechanics-After

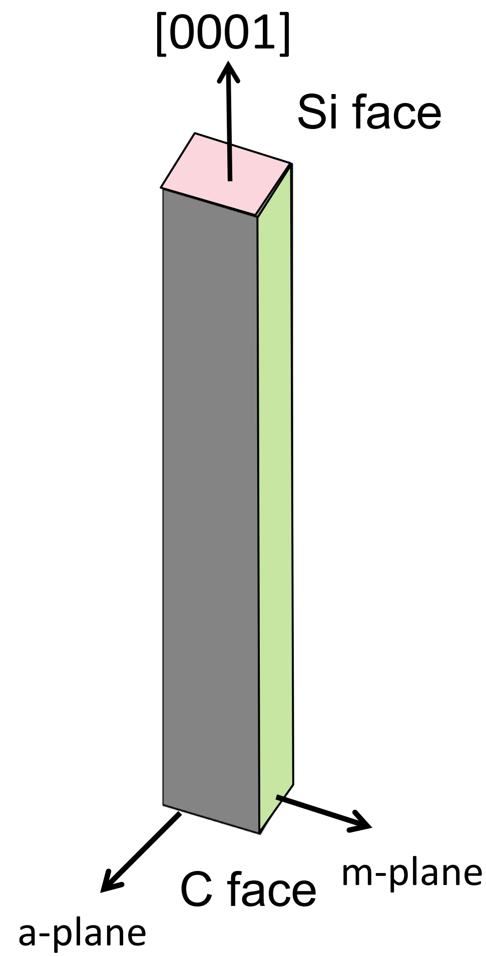


After growth the crystal is put into HF:HNO3:HCl (1:1:2) bath to remove extra source material

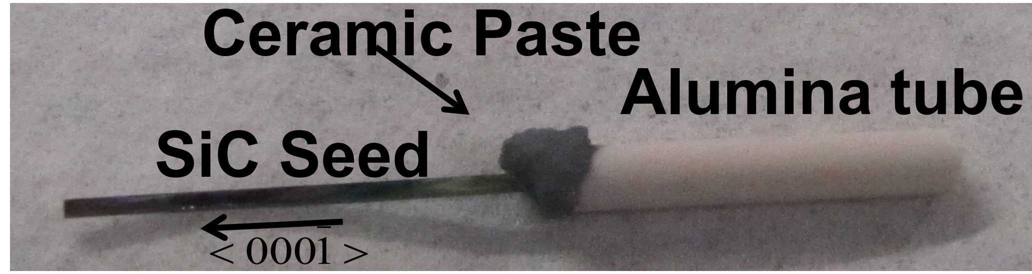
Seed Crystals

4H-SiC





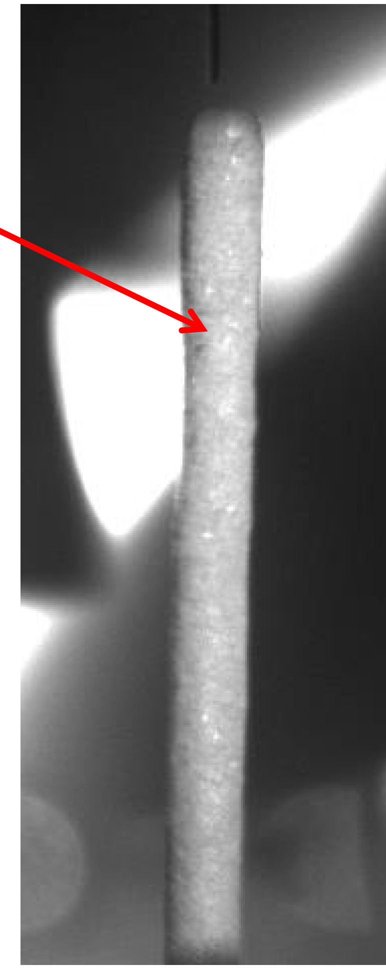
Seed Crystals



Growth face
- 4H-SiC C-face (0-10° off axis)
- ~500 μm X ~450 μm

Mounting
- Seed ~1.5 cm long
- Ceramic pasted into an alumina tube
- After curing seed crystals cleaned
  - $HCl:HNO_3$ (2:1)
  - HF

2 mm dia.



Source Material / Feed Rod

Powders
- Fe(3N5), Si(2N), graphite (3N)
- -325 mesh or < 44 μm in dia.

Feed Rod Processing
- Powders mixed by ball mill
- Formed into rods by cold isostatic press
- Sintered @ 1150°C,1 hour in hydrogen

Growth Conditions

- 15 slm, Ultra High Purity Ar, @ ~115 Torr
- Pull rate: 100 μm/hour
- Feed rate: 10,000-1,000 μm/hour
- CO_2 laser (λ=10.6 μm) @ 200-500 Watts
- Beam at heated zone :~4 mm wide x 2 mm high

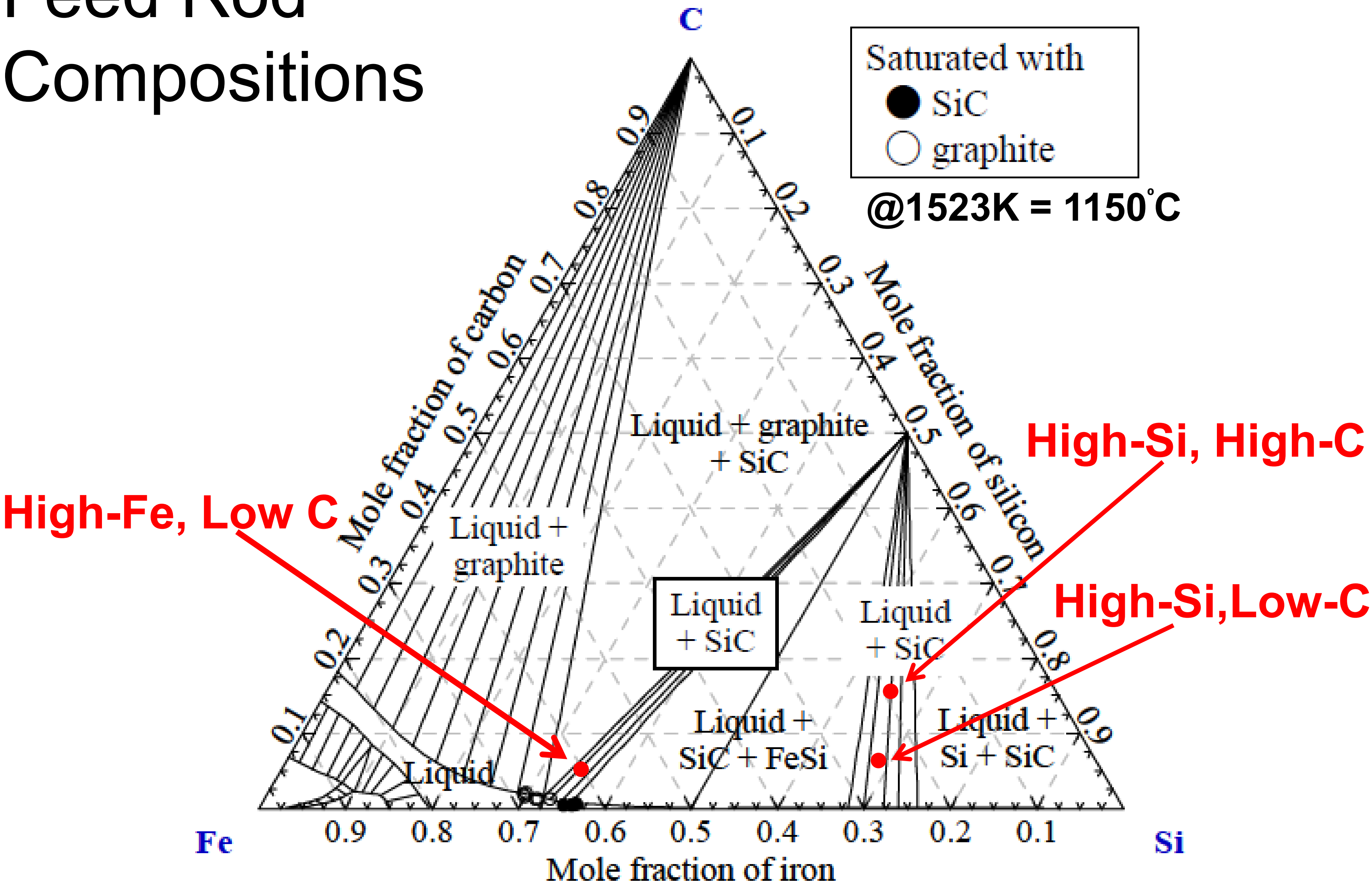
Note: Un-corrected temperature by optical pyrometer

Emissivity of the melt is unknown, ($\varepsilon_{graphite}$ = 0.9, ε_{Fe}=0.2-0.4) [1,2]

1 M.R. Null et al., J. Appl. Phys., 29, (1958) 1605

2 H. B. Wahlin et al., Phys. Rev., 74, (1948) 687

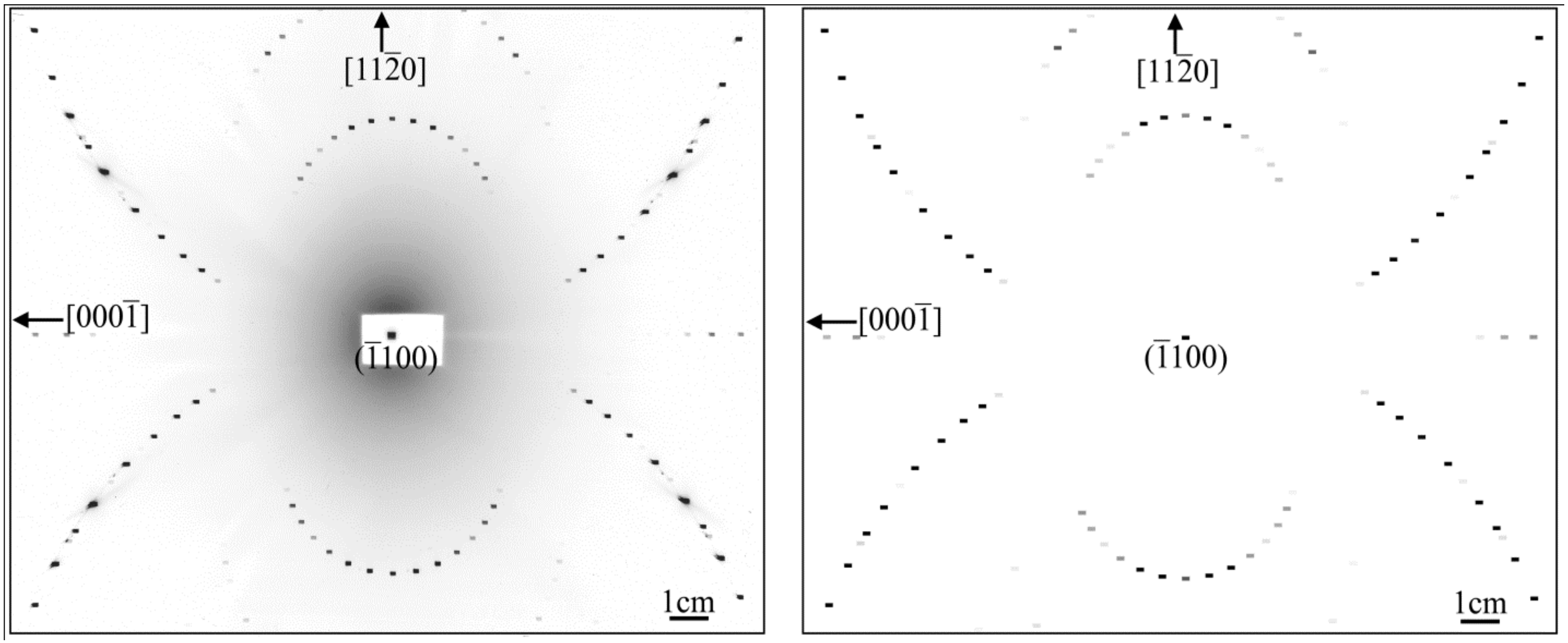
Feed Rod Compositions



"Fundamental study for solvent growth of silicon carbide utilizing Fe-Si melt", T Yoshikawa, S Kawanishi and T Tanaka, *International Conference on Advanced Structural and Functional Materials Design 2008,* Journal of Physics: Conference Series **165** (2009) 012022

X-ray Transmission Laue Diffraction Pattern of Grown Crystals

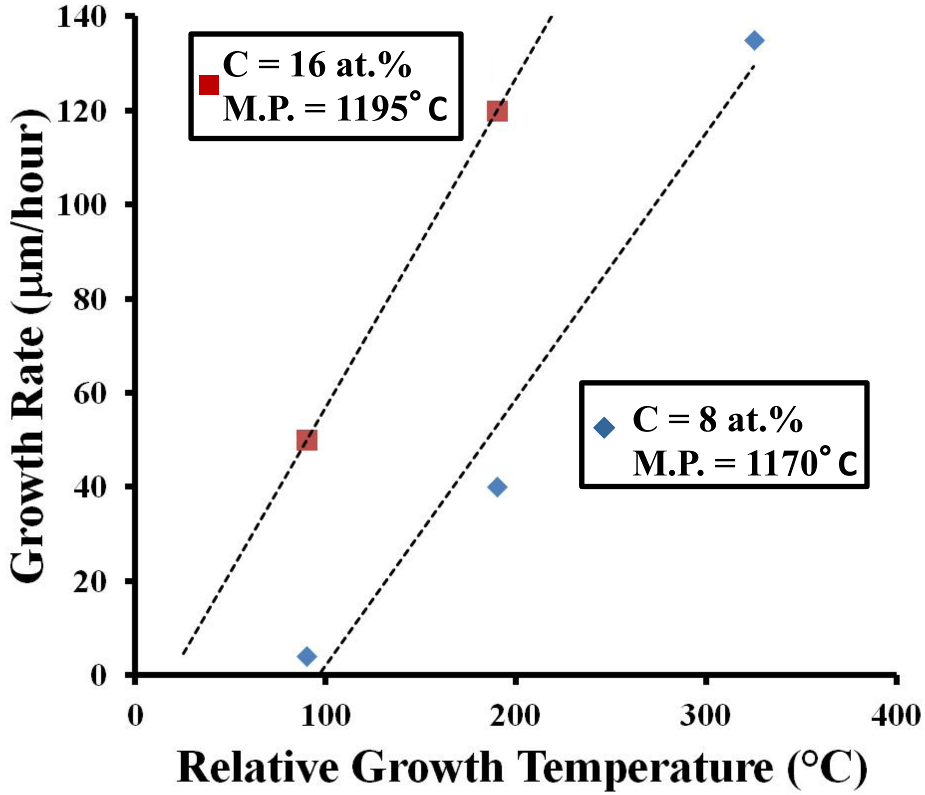
Grown Crystal | **Simulated* 4H-SiC (1-100)**



- Exact match between recorded and simulated patterns
- Grown crystals retain polytype and direction

- *X. R. Huang, J. Appl. Cryst. (2010). 43, 926–928.
- Figure previously published: A.A. Woodworth, A. Sayir, P.G. Neudeck, B. Raghothamachar, M. Dudley, Characterization of 4H< 000-1> Silicon Carbide Films Grown by Solvent-Laser Heated Floating Zone, in: MRS Spring Symposium, San Francisco 2012

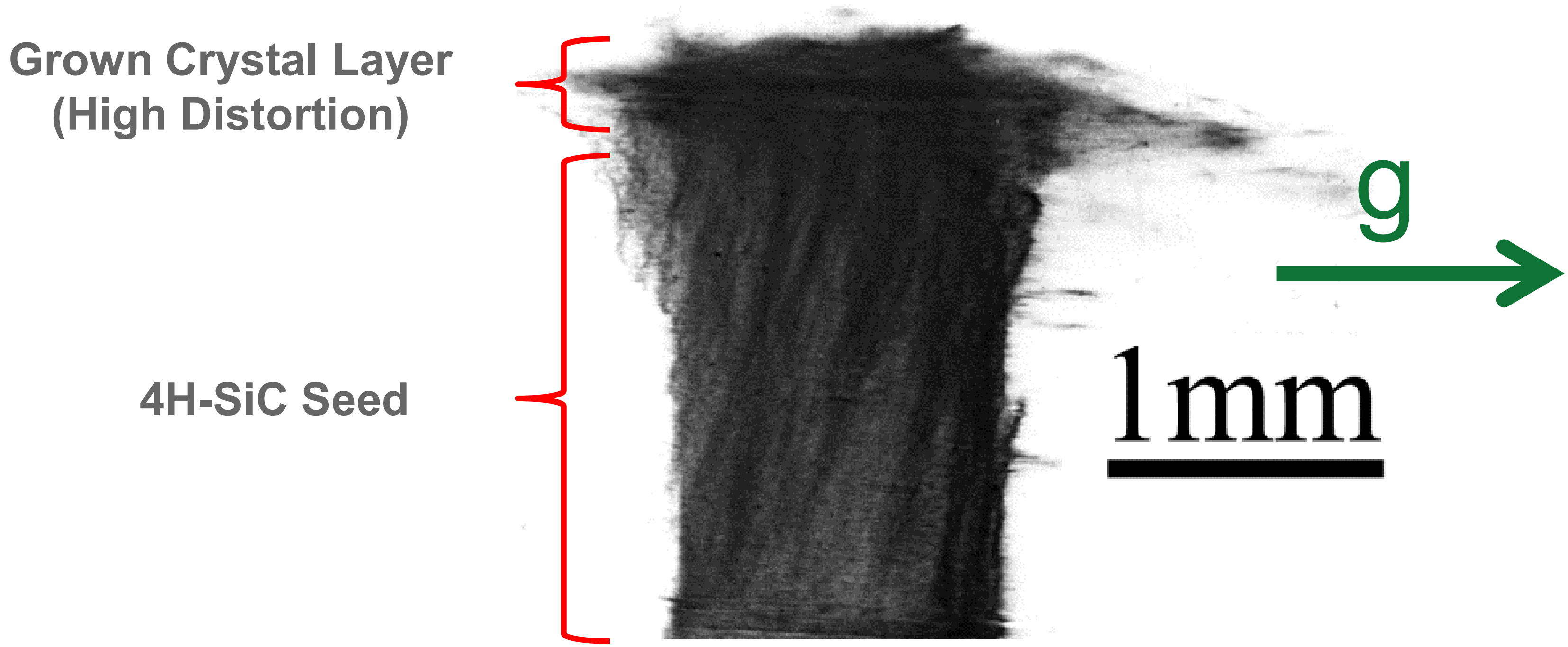
Summary of Results



			Fe Concentration (atom/cm³)	
Fe/Si (atomic ratio)	C (at.%)	M.P. (°C)	M.P.+90 °C	M.P.+190 °C
High-Si (Fe/Si~0.35)	8	1170	~10^{17}	~10^{17}
	16	1195	~10^{18}	~10^{18}
High-Fe (Fe/Si~1.9)	8	N/A	No Growth	

- M.P.= temperature at which the feed rod formed a melt
- at.% =atomic percent
- Temperatures are not corrected for emissivity

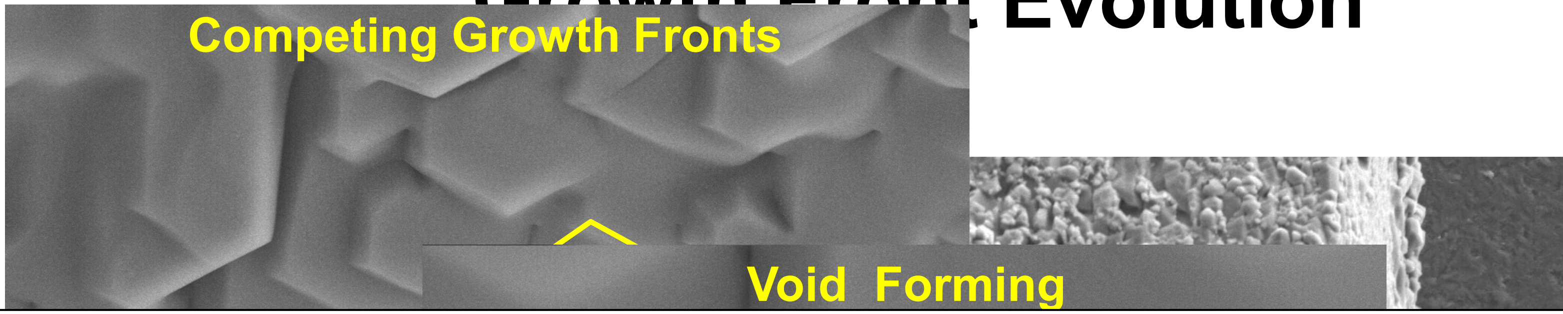
Synchrotron White Beam X-ray Topography



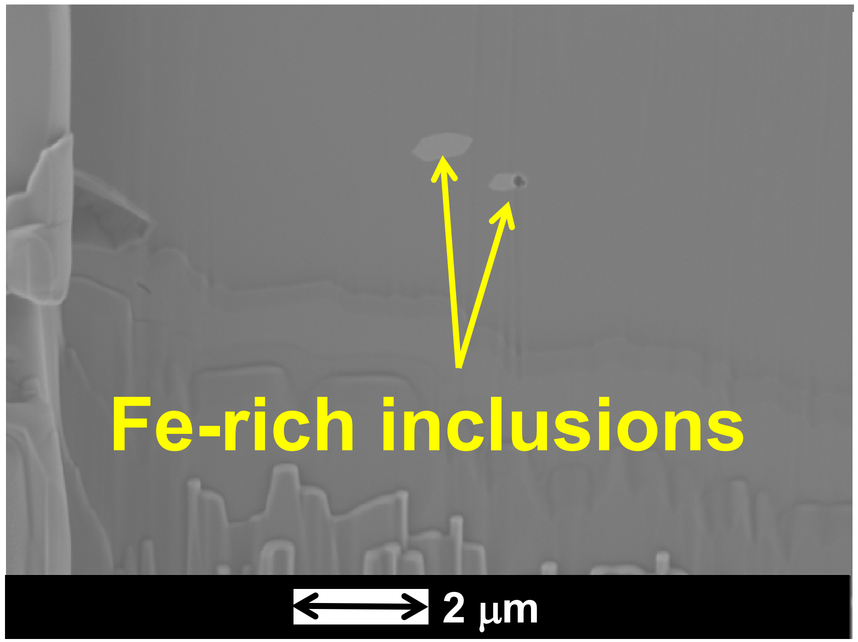
- Highly distorted X-ray topograph indicates significant inhomogeneous strain in the grown crystals

- Courtesy of Balaji Raghothamachar and Michael Dudley
- Image collected at Stony Brook Synchrotron Topography Station, Beamline X19C at the National Synchrotron Light Source, Brookhaven National Laboratory

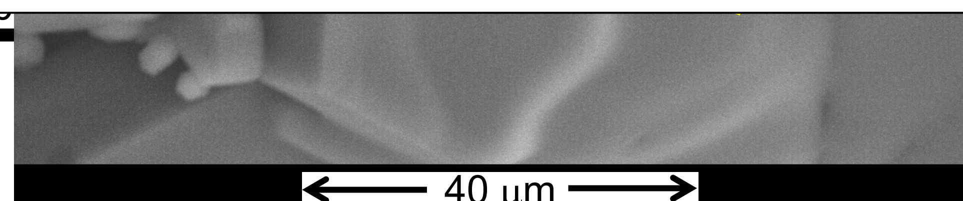
Growth Front Evolution



FIB/EDS Cross Sectional Analysis

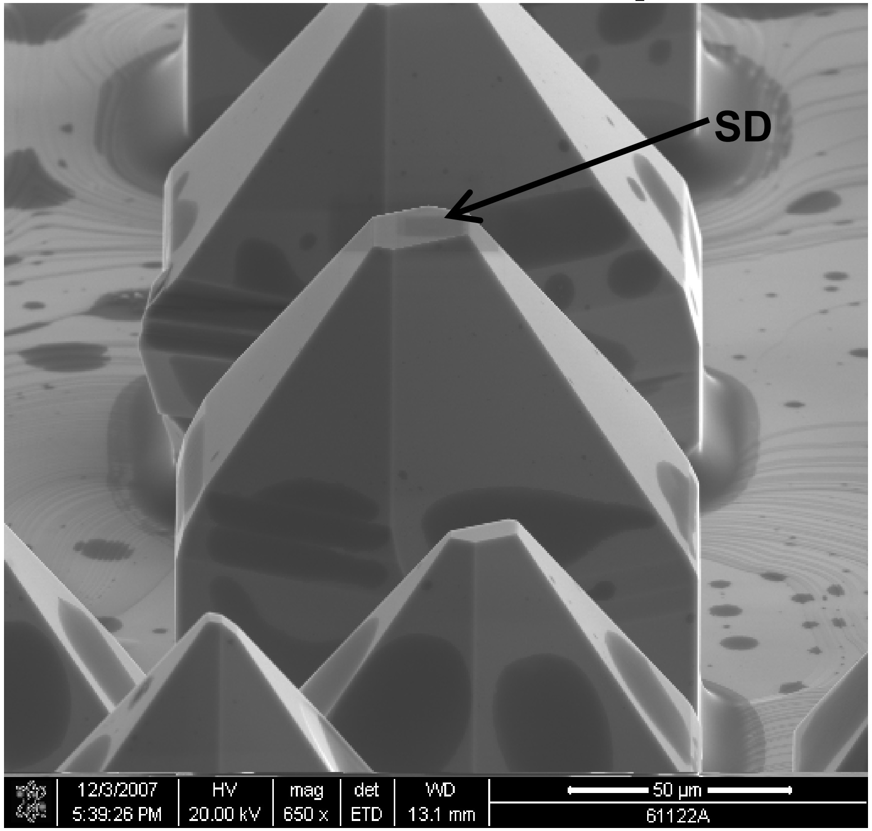


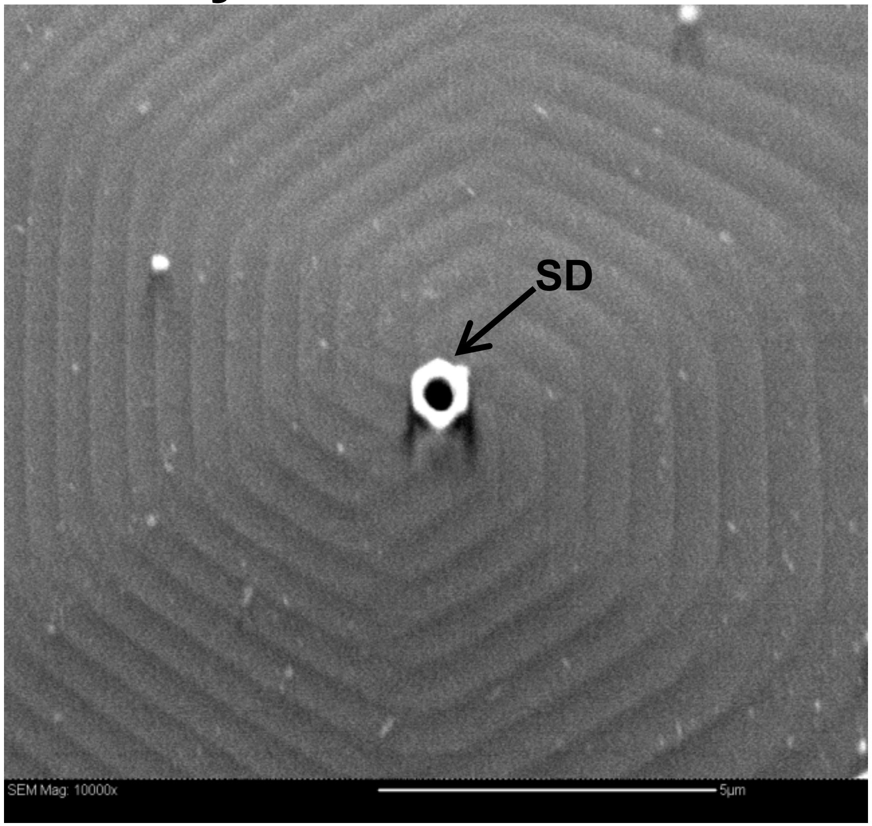
- Courtesy of Francisco Solá-López
- Figure previously published: A.A. Woodworth, A. Sayir, P.G. Neudeck, B. Raghothamachar, M. Dudley, Characterization of 4H<000-1> Silicon Carbide Films Grown by Solvent-Laser Heated Floating Zone, in: MRS Spring Symposium, San Francisco 2012



Growth Front Evolution (Cont.)

Proposed Seed Crystal





Y. Picard *et al.,* MRS Symp. Proc. Vol. 1069, p. 151 (2008)

Summary

- Initial studies show that Solvent-LHFZ readily grows SiC
- Growth rates increased with temperature and C present in the feed rod
- Fe incorporation increases with C present in the feed rod
- Grown crystal retained polytype but inclusions present create unwanted defects

Future Work

- Implement seed single screw dislocation seed crystals
- Further refinement of source materials

Team Members

RHS
(SiC growth, sensors & electronics)

Phil Neudeck
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David Spry
Michelle Mrdenovich-Hill (Sierra Lobo)
Beth Osborn (Sierra Lobo)
Chuck Blaha (Jacobs Technologies)

RXC
(Ceramics)

Ali Sayir
Fred Dynys
Thomas Sabo (CWRU/OAI)

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SiC Research at the NASA Glenn Research Center

http://www.grc.nasa.gov/WWW/SiC/