Tampere University

yCAM 2024
Tampere, Finland – 6-8 May 2024

Abstract for oral presentation

Enhancing Microstructure of Siliconized Silicon Carbide Fabricated Using Binder Jetting: Influence of Printing and Infiltration Variations

Michael Isakhani Zakaria, Setareh Zakeri, Jorma Vihinen, Erkki Levänen

Understanding the process-structure relations of Siliconized Silicon Carbide (SiSiC) is crucial in unraveling their role in subsequent properties. This research aims to enhance microstructural homogeneity in additively manufactured SiSiC, which is essential when isotropic properties are intended. In this regard, samples were fabricated via Binder Jetting (BJ) and densified using Liquid Silicon Infiltration (LSI) (where capillary infiltration of Silicon melt produces secondary SiC once in contact with the available carbon). Attempts were made to minimize printing and layering marks while diminishing porosity levels, which were assessed by examining final siliconized samples.

Binder jetting variables were selected to be layer thickness (35 and 70 µm) and printing strategy (normal and shell). In normal printing strategy, the binder bonds particles in the whole geometry whereas in shell printing only the walls with a specified thickness are exposed to the printing binder, leaving loose powder entrapped in the core. For LSI, two initiation sides were selected on the sample according to the build-up direction.

The findings reveal notable differences in the green density of samples correlated with the employed printing variables. Specifically, lower layer thickness contributes to increased green density, regardless of the printing strategy. Moreover, shell printed samples lead to lower density compared to their normally printed counterparts.

Furthermore, this study investigates the directionality of siliconizing process in debinded and carbonized samples. The latter includes an additional step of phenolic resin impregnation and pyrolysis for the green part to embed extra carbon within the structure. The results indicate that siliconizing carbonized shell printed samples produce a significantly more uniform microstructure, compared to normally printed ones. In addition, this phenomenon is augmented when the LSI is conducted opposite to the build-up direction. On the other hand, debinded shell printed samples deny complete access of silicon melt to the non-bonded powder core.

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Content

Introduction

Reaction Bonded Silicon Carbide-RBSiC

A composite consisting of SiC and varied amounts of Silicon

From 1960s, Alternative to Sintered SiC

Properties

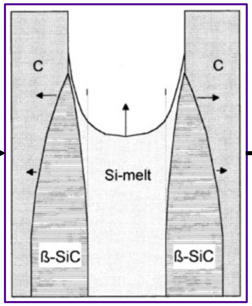
- Wear
- Impact
- Corrosion
- Refractory
- Thermal Shock

- Near net shape
- No Porosity

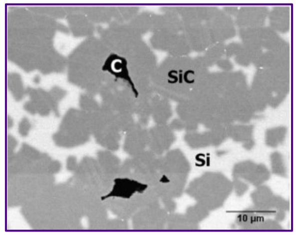
- T limit 1400 °C
- Low strength

Process

(Carbonized) Porous Preform → Capillary Infiltration / Reaction →



→ Secondary SiC / Residual Si →



Liquid Silicon Infiltration (LSI)

Applications *High Temperature*

Kiln Furniture
Radiant Tube
Heat Exchanger
Combustors
Burners
Thermocouple tube

Applications

Ballistic Armor

Sandblasting Nozzles

Inserts/Rings/Seals/Liners
Flow Control

Microstructural evolution during silicon carbide (SiC) formation by liquid silicon infiltration using optical microscopy

Melt Infiltration: an Emerging Technique for the Preparation of Novel Functional Nanostructured Materials

https://gdsic.com-http://m.cnsicah.com-http://fineawayceramics.com-https://www.omegaslate.com-https://www.calixceramics.com-https://www.entegris.com-www.ceramtec-group.com/

Introduction

Binder Jetting- Material/Process parameters

Feedstock

Powder
- Flowability
- Packing Rate

Binder
- Mostly Commercial

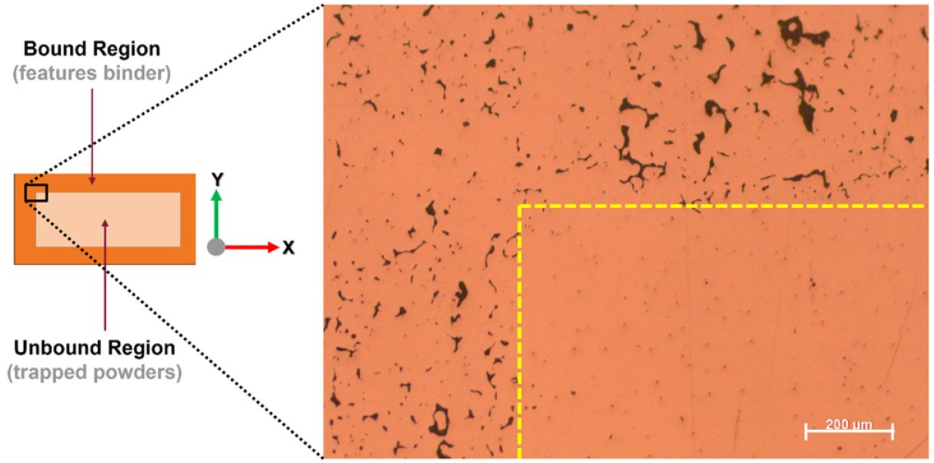
Printing & Machine

Main Parameter
- Layer Thickness
- Bed Drying Time
- Binder Set Time
- Binder Saturation

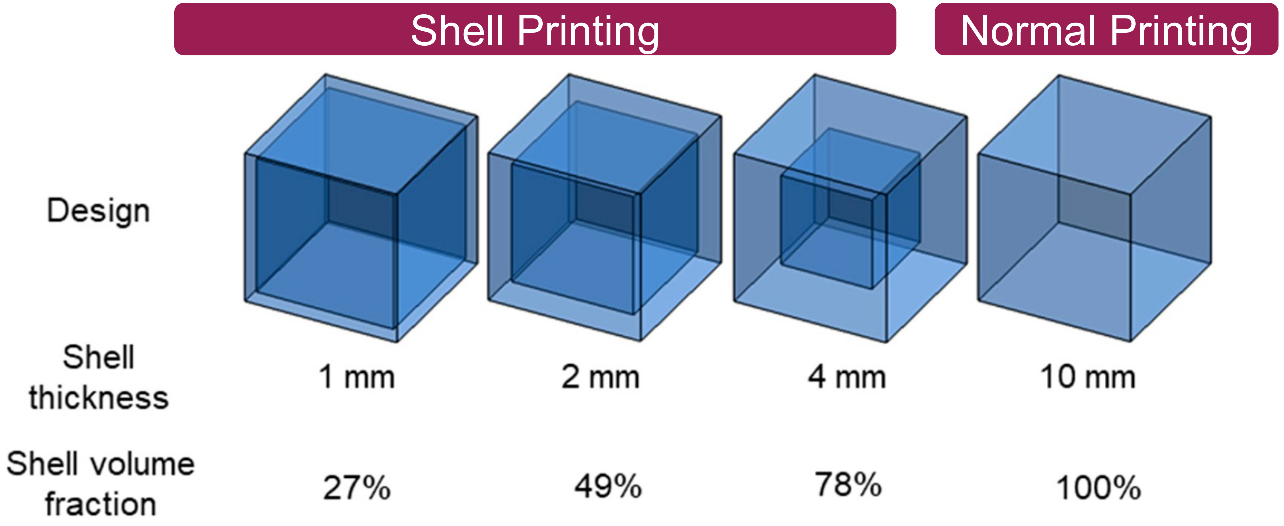
Other parameters
- Compaction thickness
- Roller Traverse Speed
- Roller RPM
- Recoater Speed
- Roughing roller RPM

Printing Strategy

Scanning Strategy
- Normal Printing: Consolidation of the whole geometry
- Shell Printing: Consolidation of an exterior wall (Shell) →Interior (Core) with loose powder



Impact of binder on part densification: Enhancing binder jetting part properties through the fabrication of shelled geometries - ScienceDirect



Binder Jetting Additive Manufacturing: Powder Packing in Shell Printing (mdpi.com)

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Introduction

Binder Jetting- Shell Printing

Laser Powder Bed Methods		Binder Jetting (BJT)	
1999	**Metal** - To form a 'can' for containerless **HIP**		
		2016	**Metal -** To obtain **higher density** after **sintering**
2017	**Metal Carbide** - **Patent-** To improve sinter-**HIP** final density		
2019	**Metal -** To form a 'capsule' and **HIP** for new alloys		
2021	**Metal -** To form shells and **HIP** to enhance productivity		
2021	**Metal -** To form shells and **HIP** to assess fatigue		
		2022	**Metal -** To spatially control phase fraction after infiltration
		2022	**Metal** – To assess densification and properties after sintering
		2022	**Ceramic –** To assess powder packing in shell/core

The 2017 patent row spans both columns.

Shell printing Term (bracket around the three 2022 entries)

Shell printing affects powder packing of the green body, also affected by layer thickness

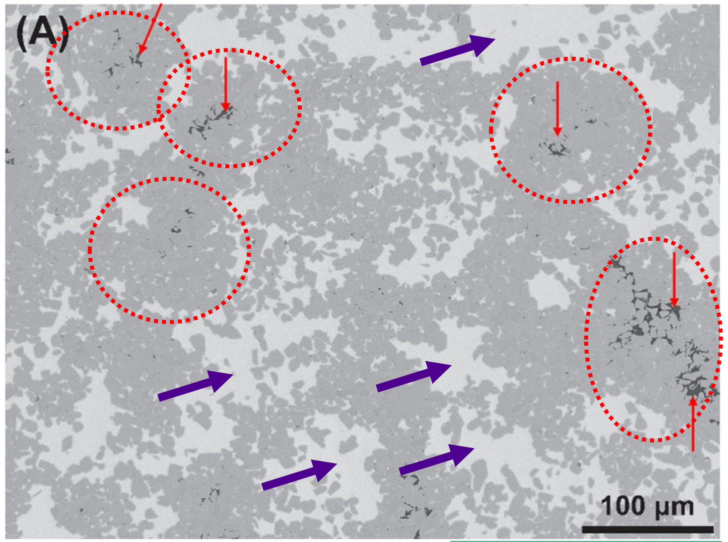
Infiltration direction affects microstructure of the core in shell printed samples

Patent: US 2019 / 0168299 A1
Solid State Sintered 3-D Printing Component by Using Inkjet (Binder) Method
Binder Jetting Additive Manufacturing: Powder Packing in Shell Printing (mdpi.com)
A process to spatially control the fraction of SS420 and bronze phases in binder jet infiltrated parts - ScienceDirect
Impact of binder on part densification: Enhancing binder jetting part properties through the fabrication of shelled geometries - ScienceDirect
Processing of titanium net shapes by SLS/HIP
Fatigue performance of shelled additively manufactured parts subjected to hot isostatic pressing
Tailor-Made Net-Shape Composite Components by Combining Additive Manufacturing and Hot Isostatic Pressing
Productivity enhancement of laser powder bed fusion using compensated shelled geometries and hot isostatic pressing

Introduction

BJTed RBSiC - Carbonization

Multiple Phenolic Impregnation and Pyrolysis (I&P) cycles



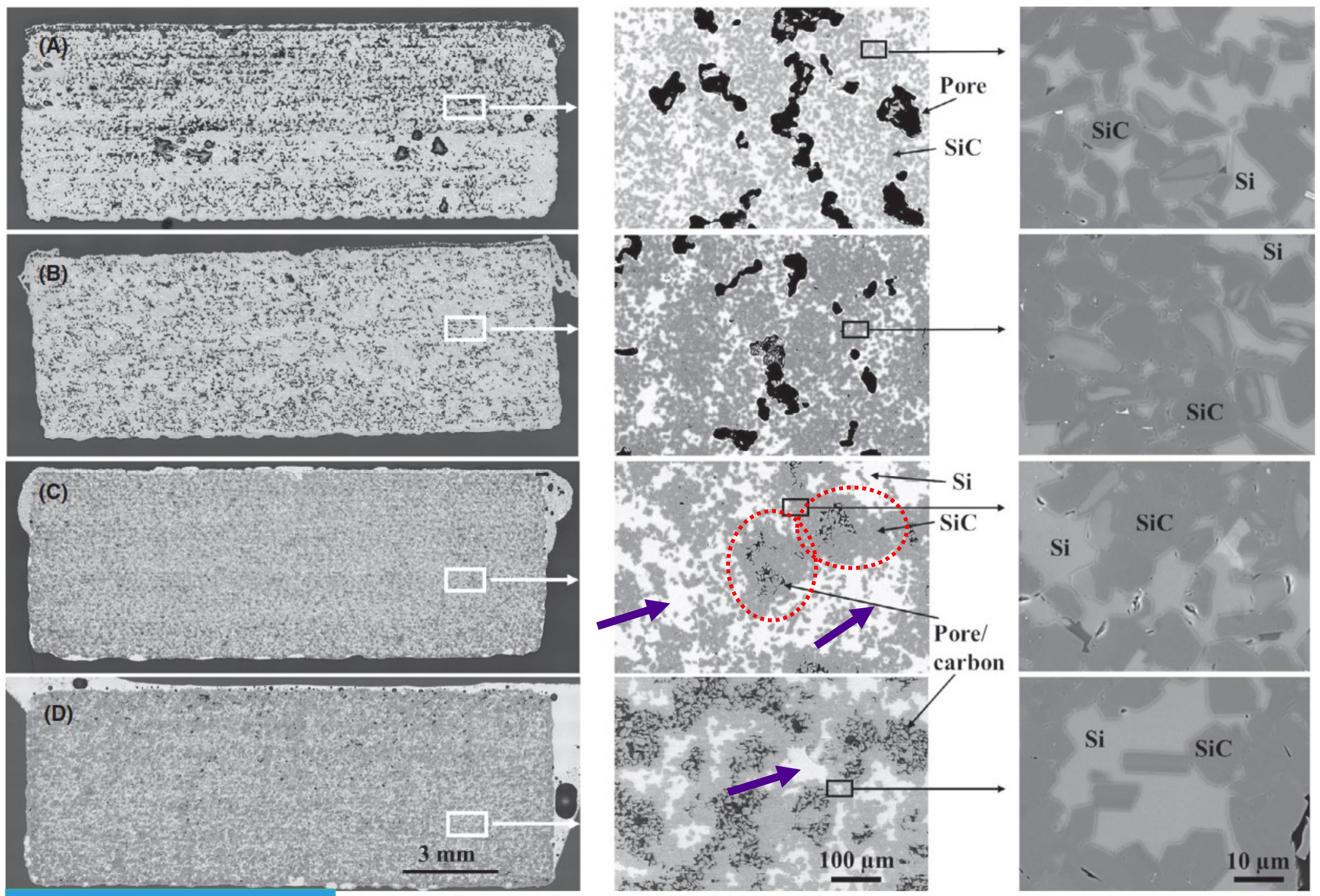
d_{50} = 16.1 µm

Particle accumulation

Si accumulation

Isolated primitives

Binder droplet impact locally agglomerates the powder within each layer



d_{50} = 11.32 µm

Environmental stability of additively manufactured siliconized silicon carbide for applications in hybrid energy systems

Properties of SiC-Si made via binder jet 3D printing of SiC powder, carbon addition, and silicon melt infiltration

Introduction

Research objective

Can we enhance the microstructure by modifying SiC BJT and LSI variables?

Enhancing based on ceramic engineering guidelines:
- ✓ Fine microstructure
- ✓ Low porosity
- ✓ Microstructural homogeneity

- ✓ **Selecting a relatively fine SiC powder feedstock**
- ✓ **Variables in printing**: Normal & Shell, Layer Thickness (LT35 & 70 µm)
- ✓ **One cycle of** phenol impregnation and pyrolysis (I&P)
- ✓ **Two variations in direction** of LSI with respect to build-up

- ✓ **Green density and porosity?**
- ✓ **Role of carbonization (I&P)?**
- ✓ **Directionality in processing of BJTed-RBSiC?**
- ✓ **Microstructural homogeneity and phase fraction after LSI?**

Some insight on Process-Structure-Property of BJTed-RBSiC

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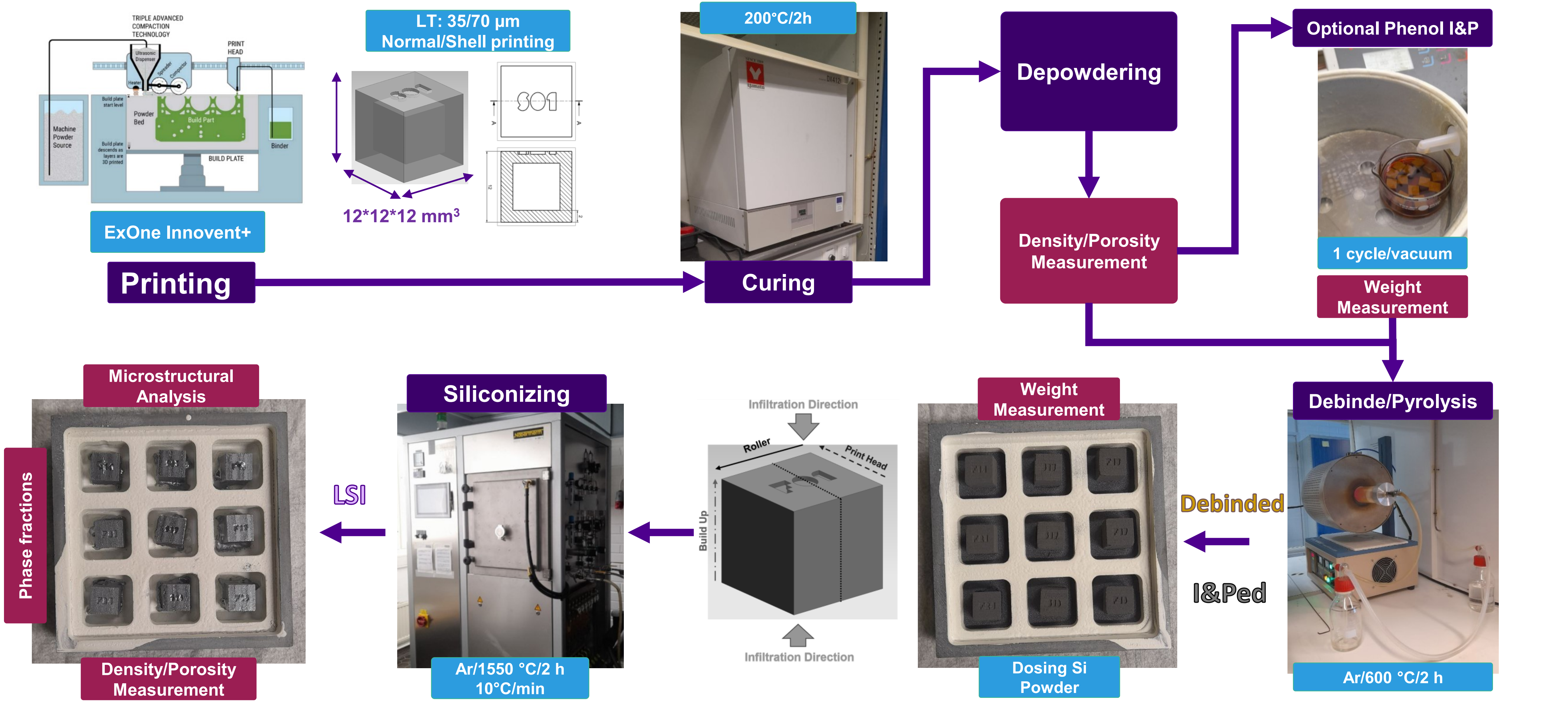
Experimental

Printing and Post Processing



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Experimental

Printing Variables and Samples

SiC Powder Properties

Property	Value
D_{10} (μm)	6.11
D_{50} (μm)	10.34
D_{90} (μm)	16.19
ρ_{app} (g/cm³)	0.96
ρ_{tap} (g/cm³)	1.99
Hausner Ratio	2.07

ISO 3923-1:2018

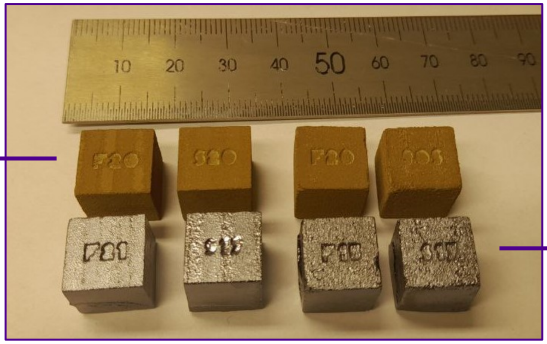
Printing Parameters

Parameter	Remark
Layer Thickness	35 and 70 μm
Binder Saturation	75%
Roller Traverse Speed	25 mm/s
Powder bed Drying Time	5 Sec.
Binder Set Time	5 Sec.
Smoothing Roller	300 RPM
Roughing Roller	300 RPM
Recoater Speed	8 mm/s
Dispense on delay	3 Sec.

Printed Samples

Sample Code	LSI Direction	Layer Thickness	Carbonization	Printing Strategy
T/D/N/35	Top	35 μm	Debinded	Normal
T/D/S/35				Shell
T/IP/N/35			I&P	Normal
T/IP/S/35				Shell
T/D/N/70		70 μm	Debinded	Normal
T/D/S/70				Shell
T/IP/N/70			I&P	Normal
T/IP/S/70				Shell
B/D/N/35	Bottom	35 μm	Debinded	Normal
B/D/S/35				Shell
B/IP/N/35			I&P	Normal
B/IP/S/35				Shell
B/D/N/70		70 μm	Debinded	Normal
B/D/S/70				Shell
B/IP/N/70			I&P	Normal
B/IP/S/70				Shell

Green Samples



Siliconized Samples

Results & Discussion

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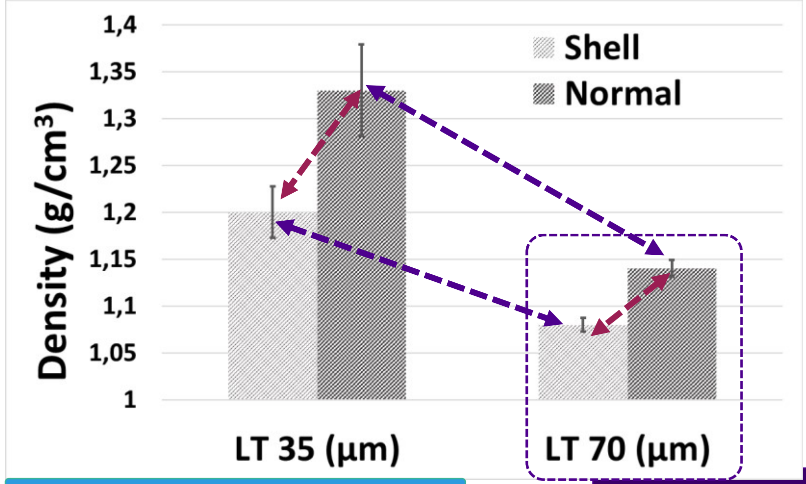
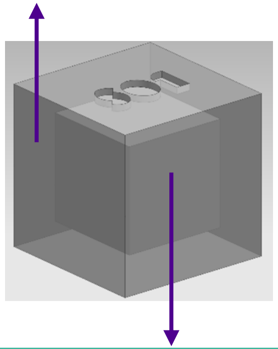
Green Parts : Bulk Density/Apparent Porosity

Green Density



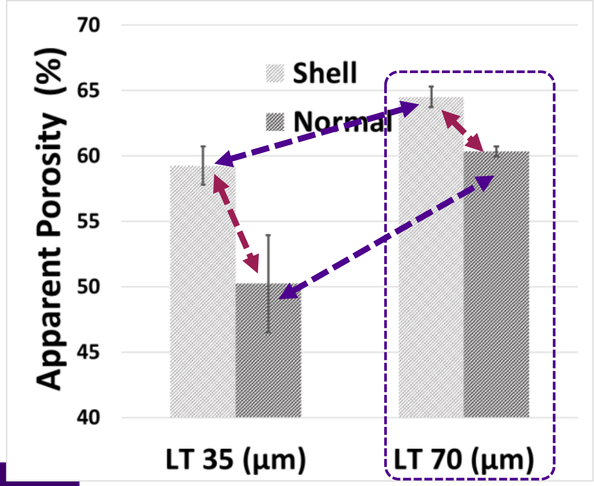
Geometrical Measurement

Shell: bound powder



Core: loose powder

Green Porosity



Archimedes Method

Less packing and more interlayer voids at higher LT

Powder → **Recoating parameters Dispensing/Spreading** → **Binder deposition** → **Green Density**

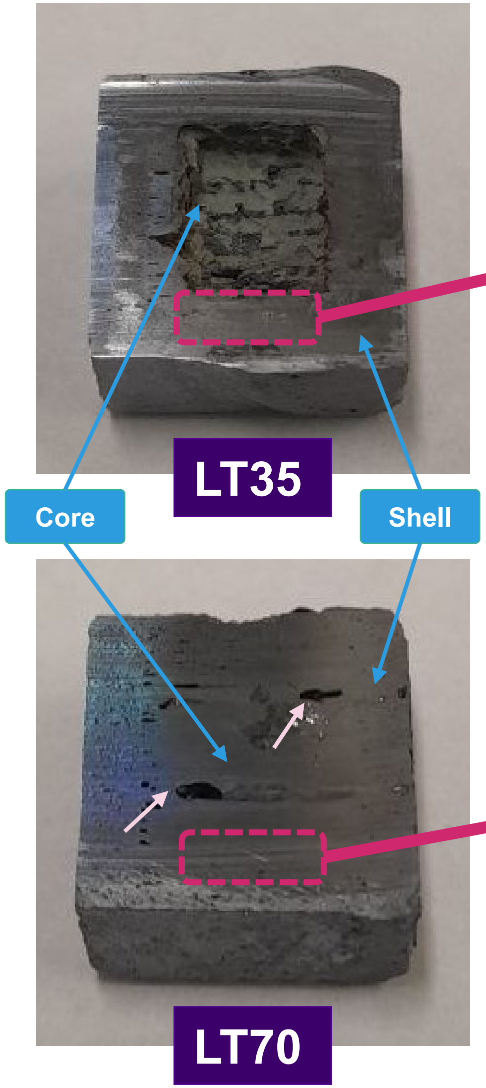
with specific ρ_{app} | bed density could reach ρ_{tap} | bed density could increase/decrease

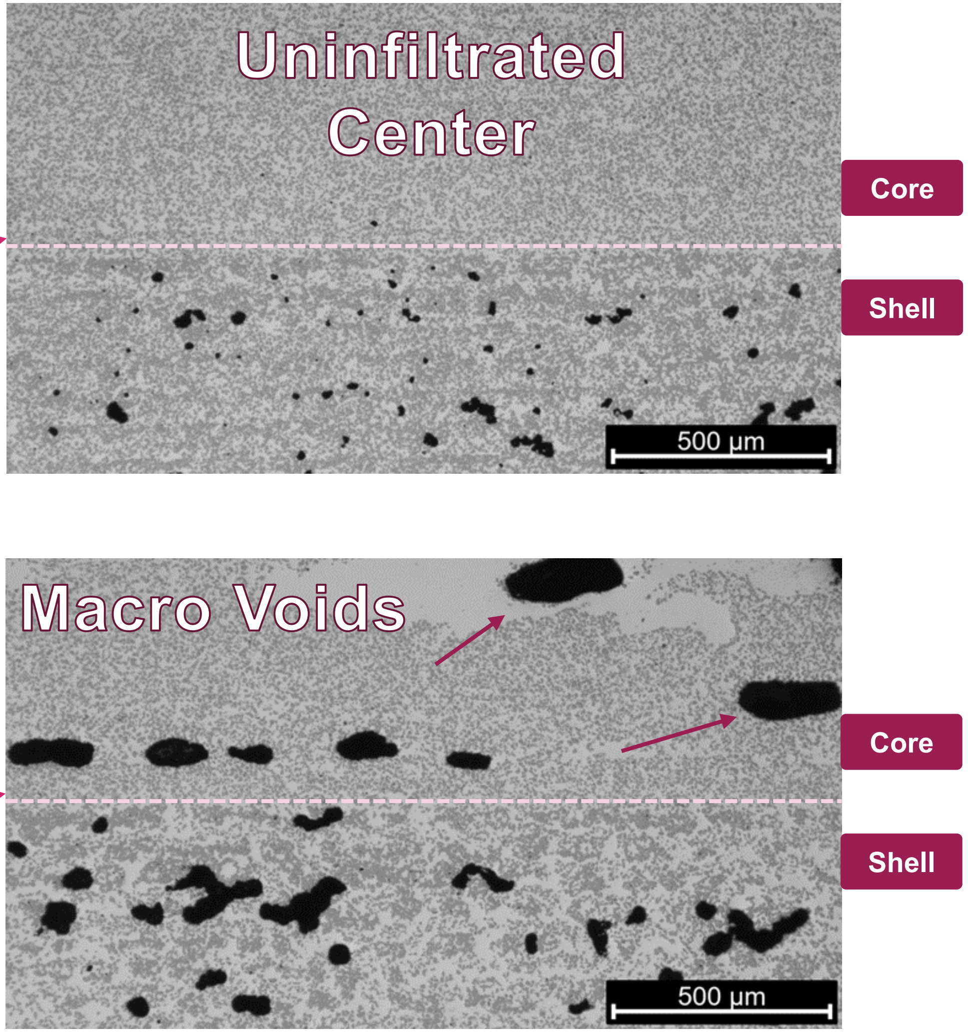
High Flowability/Packability (Large Spherical powder) → Powder bed density close to the higher end (ρ_{tap}) → Decreased density after binder deposition → Shell printing increases green density → **At relatively high LT** → Shell printing decreases green density

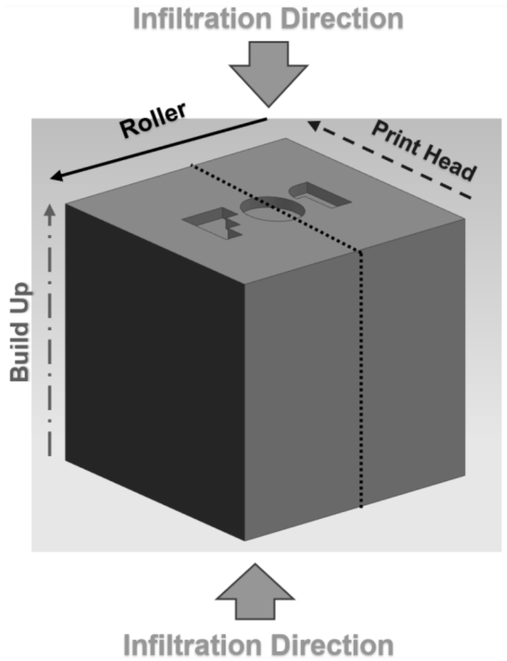
Low Flowability/Packability (Fine Irregular powder) → Powder bed density close to lower end (ρ_{app}) → Increased density after binder deposition → Shell printing decreases green density

Results & Discussion

Optical Microscopy – Shell-printed/ Debinded + LSI (Top & Bottom)





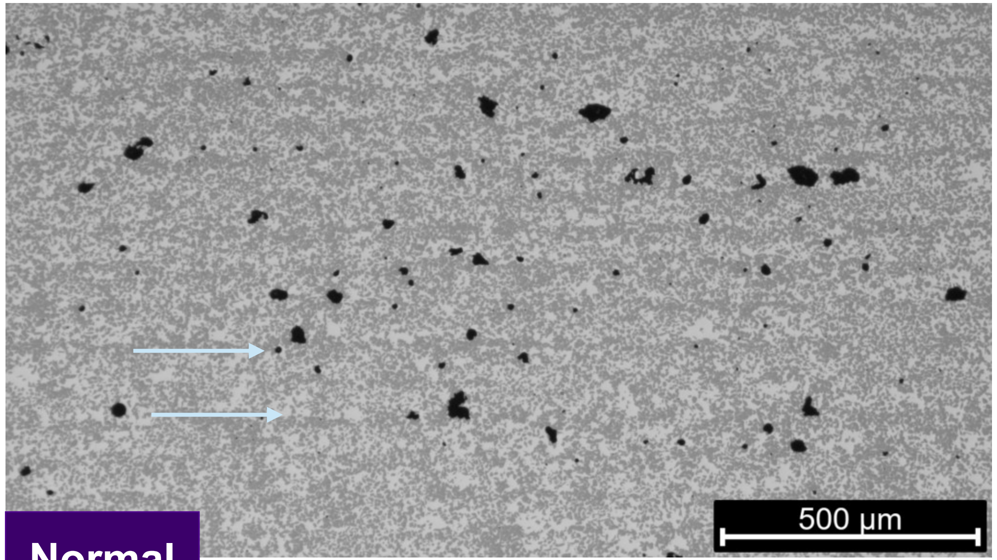


Resolved after I&P

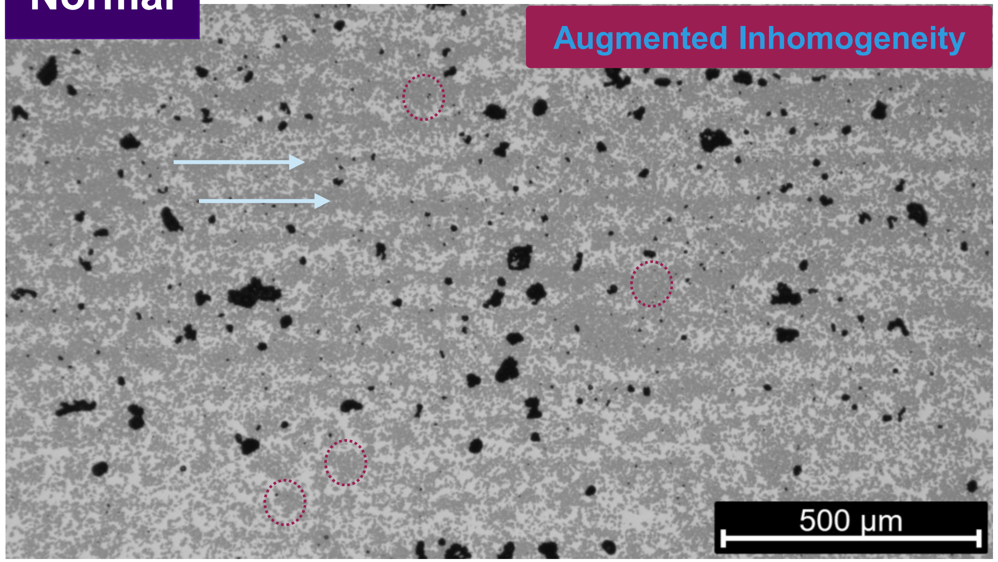
- Fixed particles- No rearrangement during infiltration
- Reactive wetting
- Temp. spikes due to exothermic reaction

Results & Discussion

OM - LT35 µm / Debinded vs. I&Ped (LSI Top) / Center



Normal



Debinded

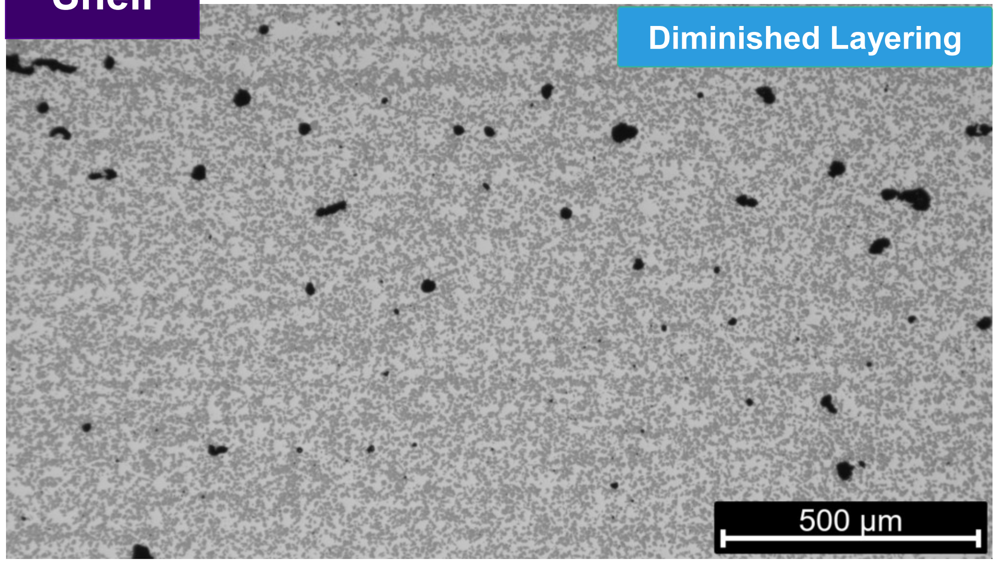
I&Ped

Layering

Isolated Primitives

Uninfiltrated Center

Shell



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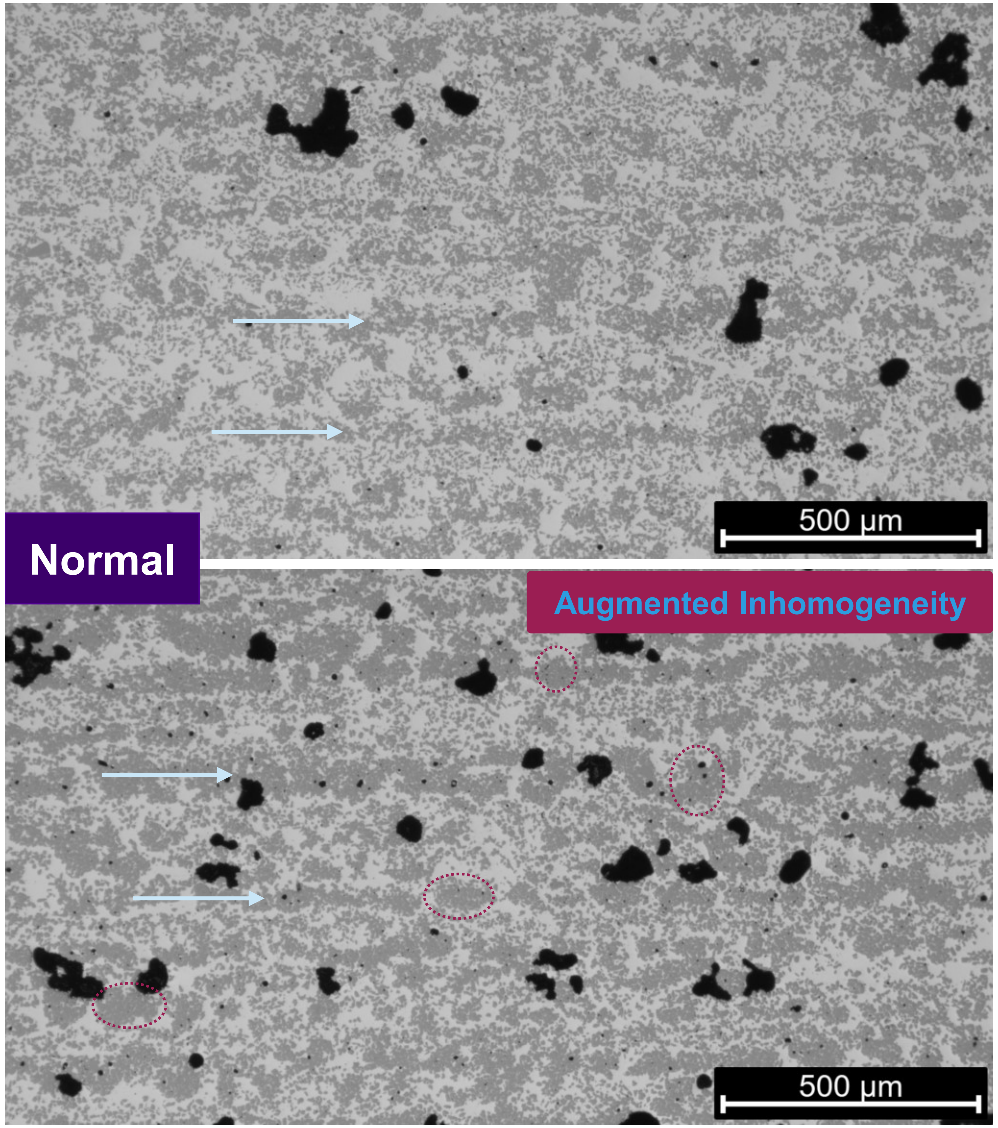
Results & Discussion

OM - LT70 µm / Debinded vs. I&Ped (LSI Top) / Center

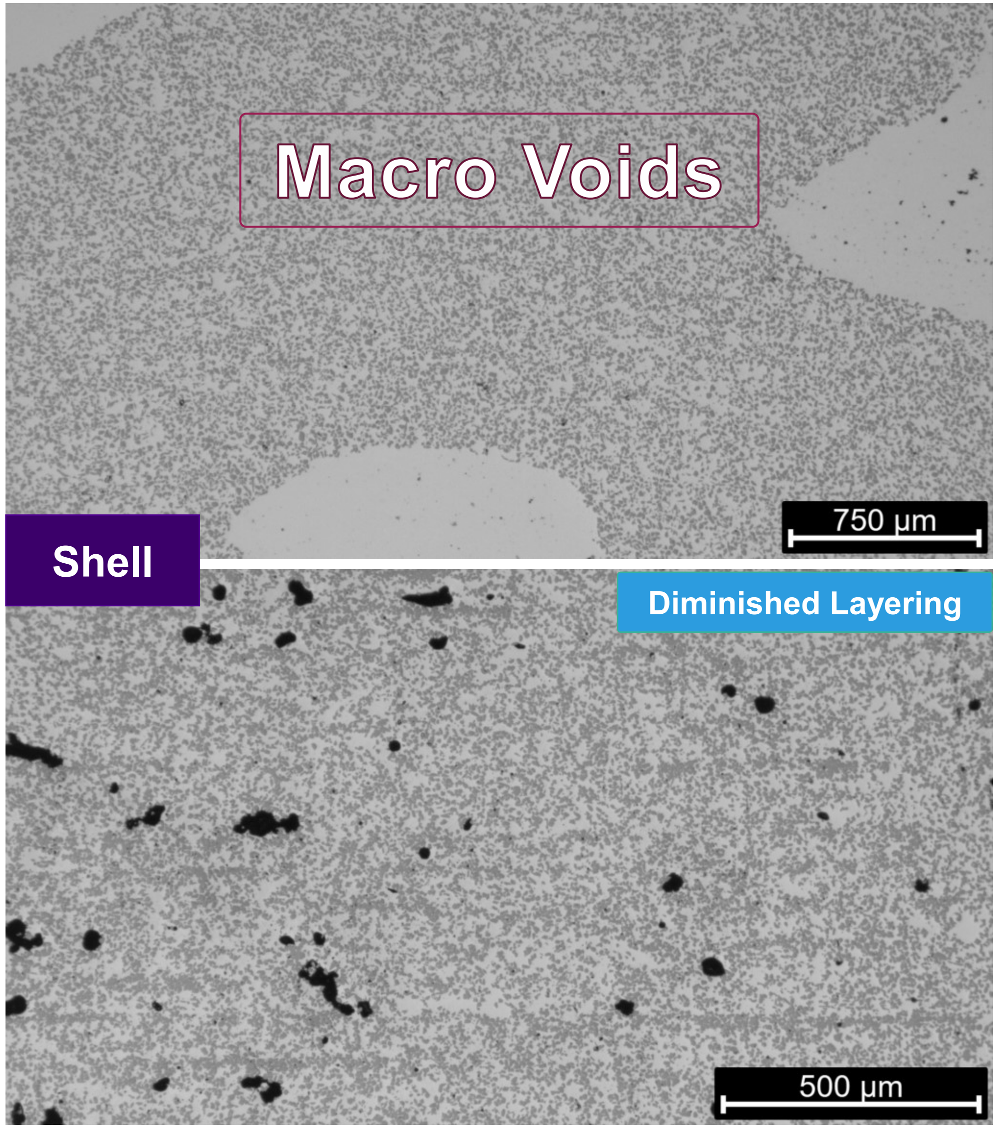


Debinded

I&Ped

Layering

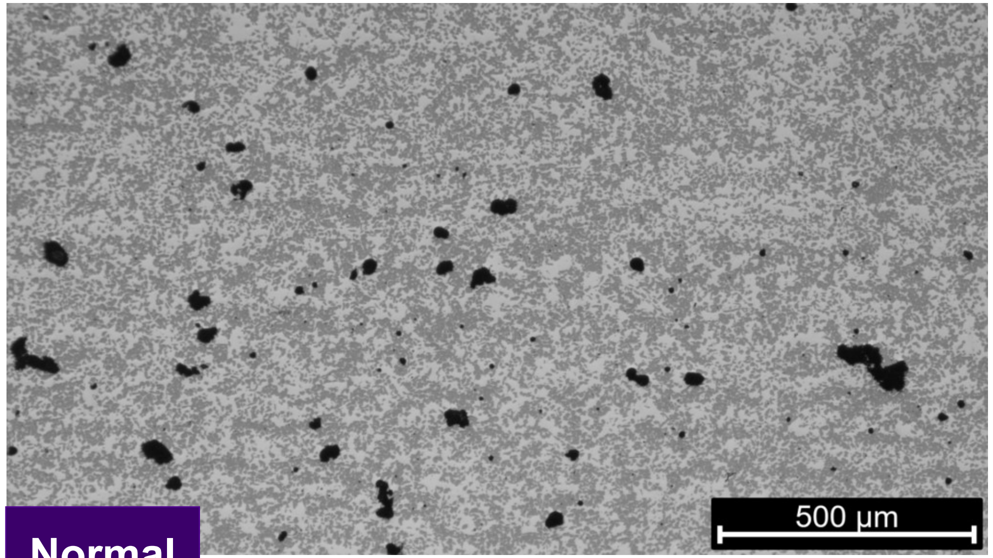
Isolated Primitives



Results & Discussion

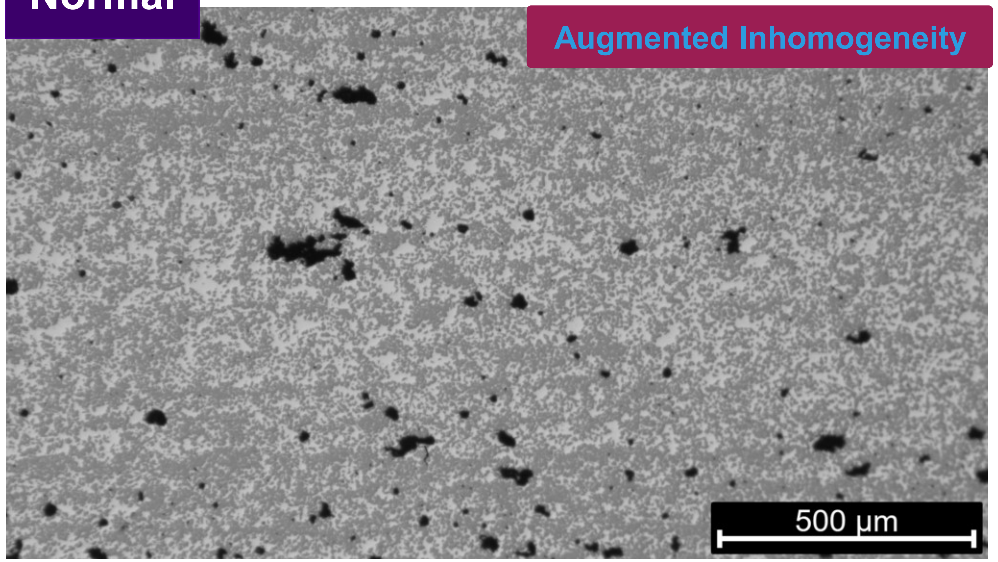
OM - LT35 µm / Debinded vs. I&Ped (LSI Bottom) / Center

Uninfiltrated Center



Normal

Augmented Inhomogeneity

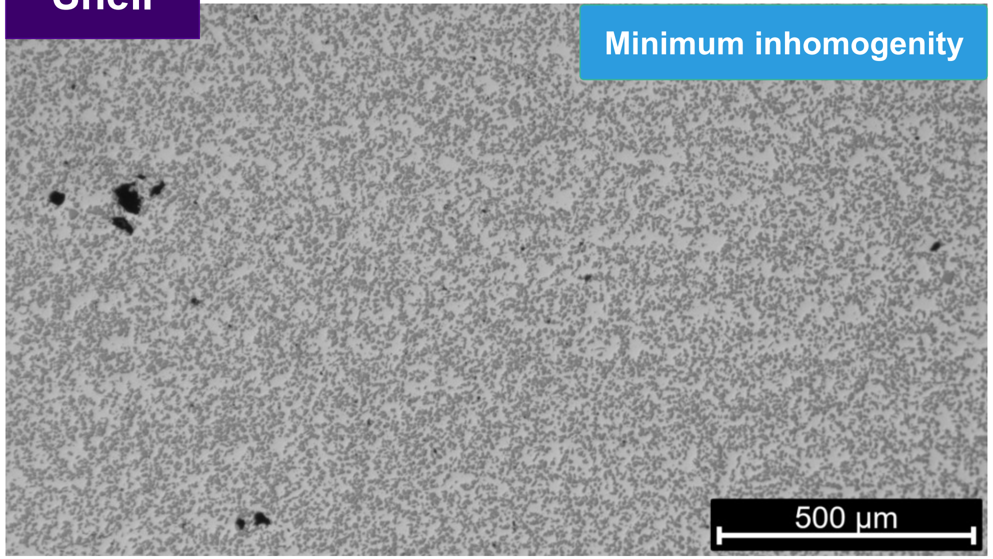


Debinded

I&Ped

Shell

Minimum inhomogenity



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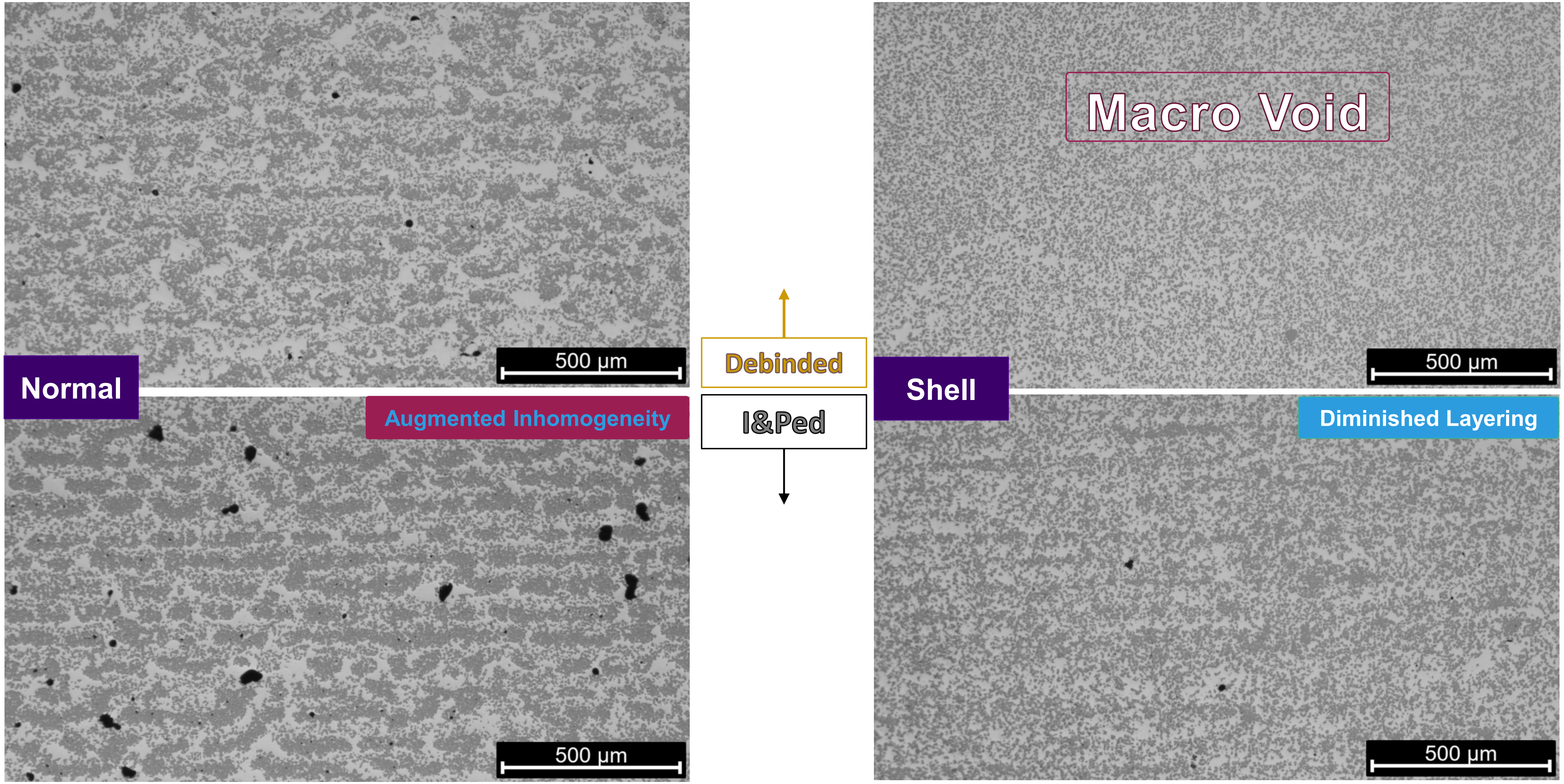
Results & Discussion

OM - LT70 µm / Debinded vs. I&Ped (LSI Bottom) / Center



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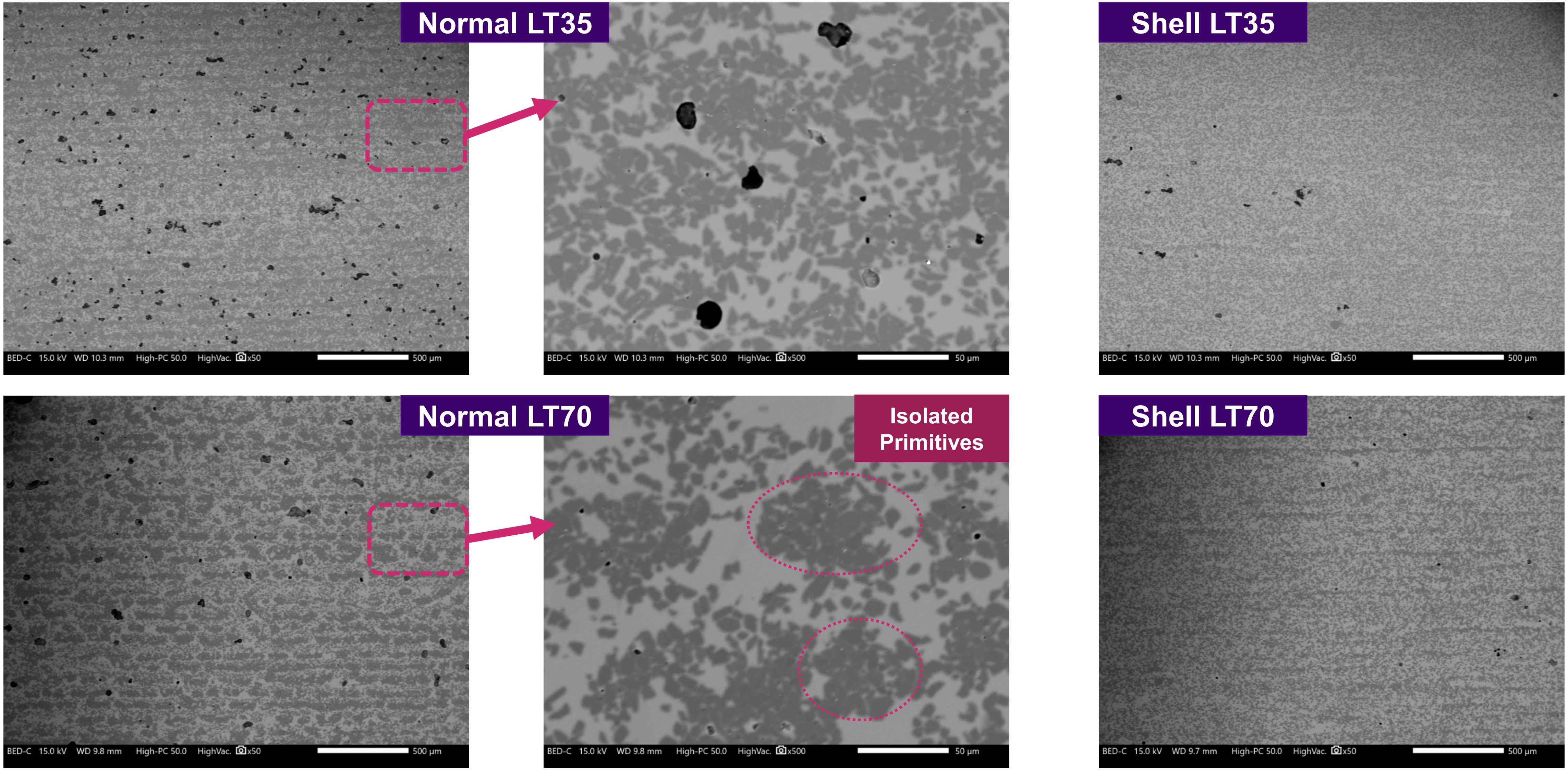
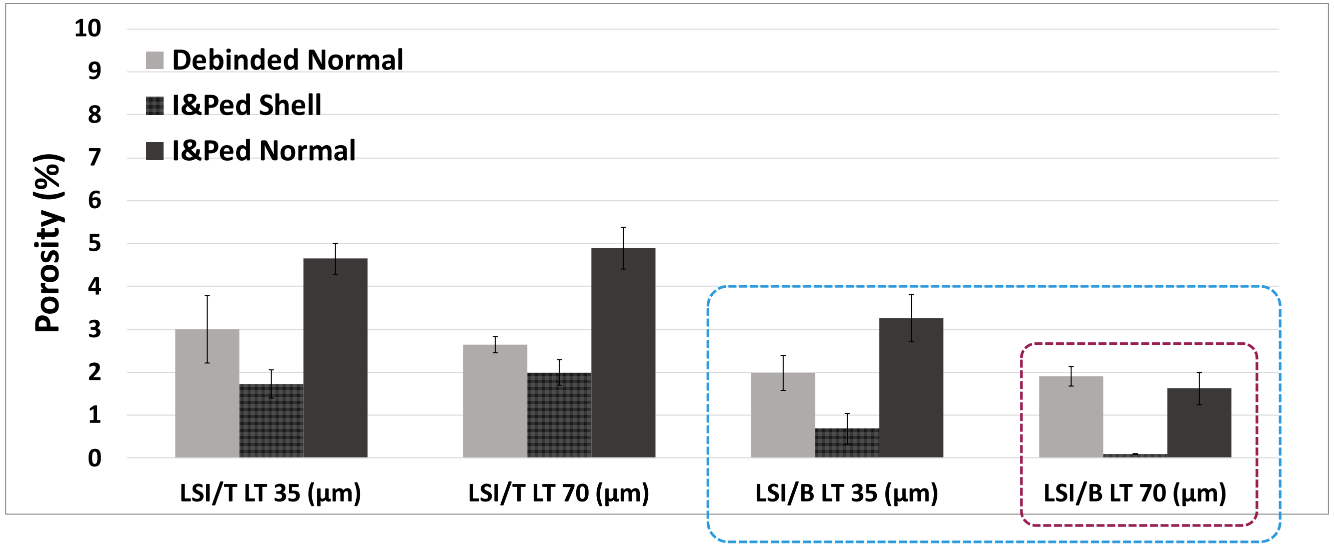
Results & Discussion

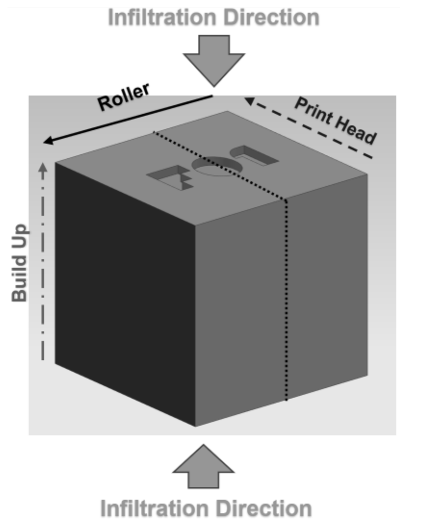
SEM-Normal vs. Shell / I&Ped + (LSI Bottom)



Results & Discussion

Comparing Porosity and Residual Si





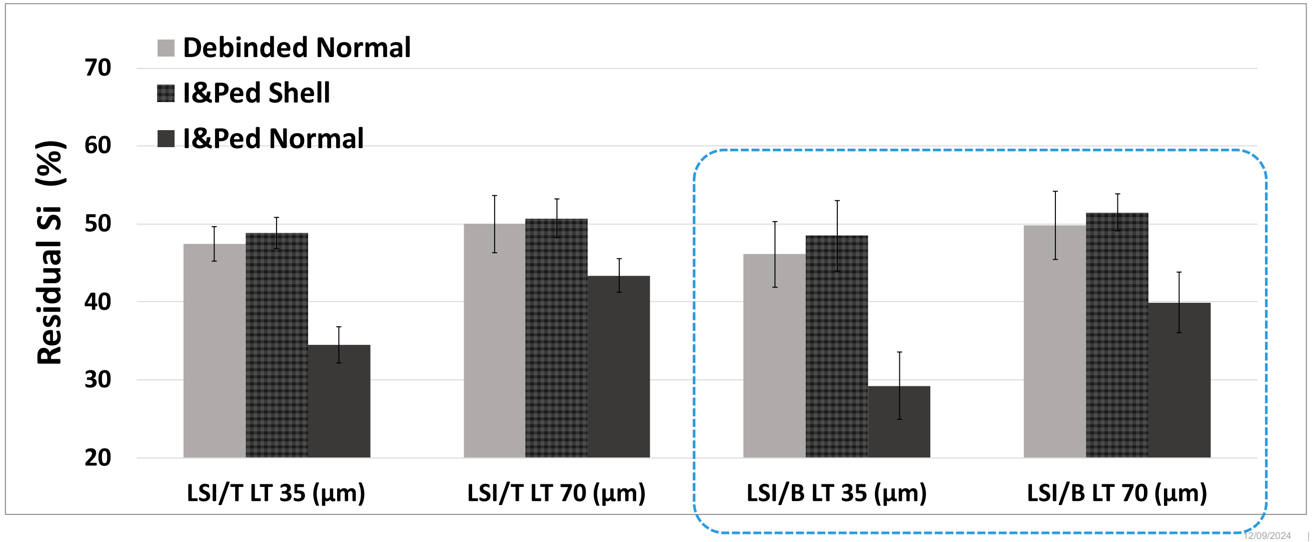
Enhanced balance between reaction and infiltration rate

↓

Improved carbon accessibility by Si

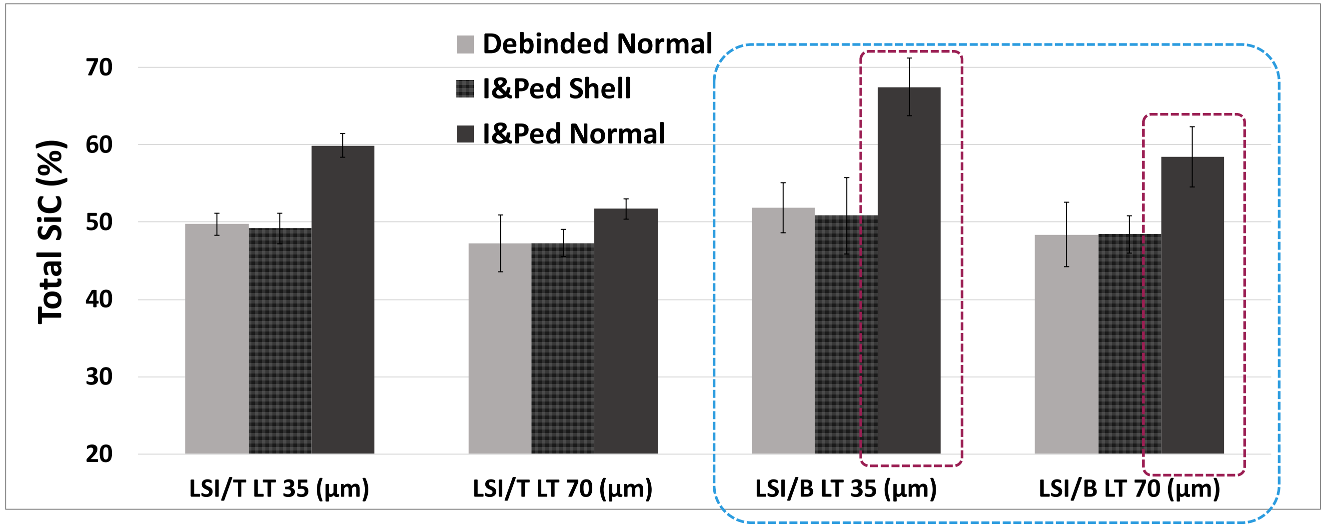
↓

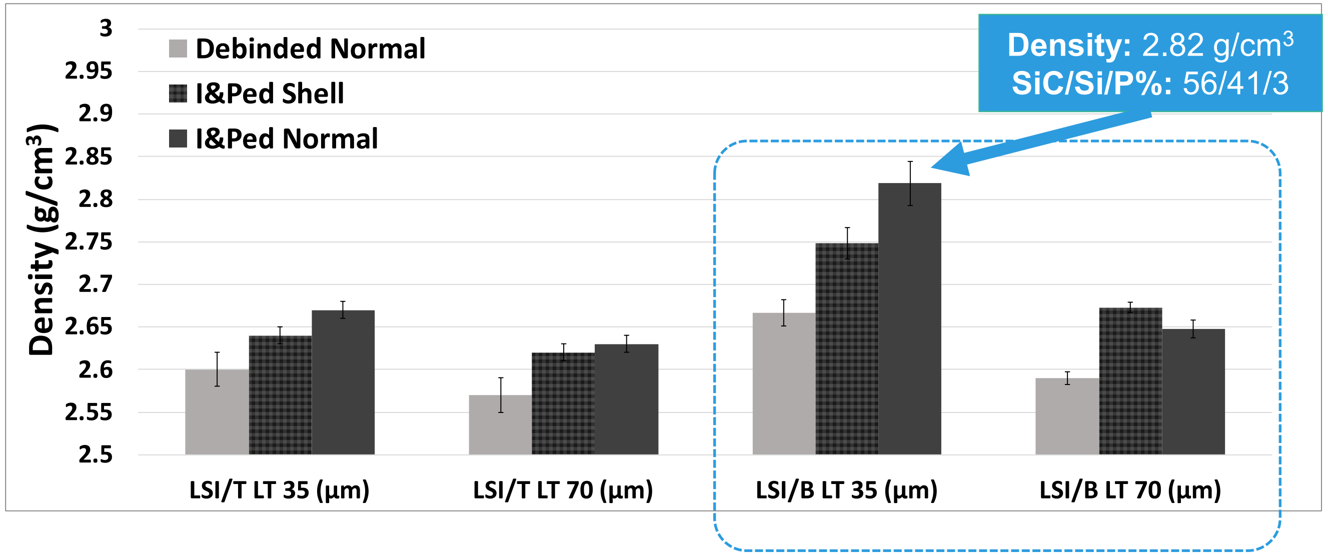
Reduced reaction choking



Results & Discussion

Comparing SiC content and Density





Contribution to final density

Higher Green Density (Lower LT & Normal Printing)

Lower Residual Si (Lower LT & Normal Printing) in LSI/B

Lower Final Porosity (Higher LT & Shell Printing) in LSI/B

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Conclusion

Enhancing Microstructure of BJTed-RBSiC

- ✓ Higher layer thickness leads to higher porosity, both in shell & normal printed samples
- ✓ Shell printing with low flowability/packability powder leads to higher green porosity/lower green density
- ✓ A fringe opportunity working on BJT-RBSiC, freeze the green microstructure to study effects of printing

- ✓ Imperfect infiltration of Si melt in the core of debinded shell printed samples (loose non-bound powder)
- ✓ Successful densification of shell printed samples with one cycle of I&P
- ✓ Normal printing of fine SiC powder creates layering and isolated primitives (augmented after I&P)
- ✓ Diminished layering and no primitive isolation achieved in shell printing of fine SiC powder
- ✓ While normal printing yielded higher SiC fraction, shell printing resulted in minimized porosity.
- ✓ LSI effectiveness is directional in BJTed SiC influencing phase fractions.

Enhanced microstructure can be achieved in BJT-RBSiC by selecting <u>fine</u> SiC particle size <u>low LT</u>, <u>shell</u> printing and <u>LSI/B</u>

Thank you for your attention!