Silicon Carbide (SiC) Foam for Molten Salt Containment in CSP-GEN-3 Systems

Touchstone Research Laboratory, Ltd.

Contract DE-SC0018678

Project Period: July 02, 2019 – August 18, 2021

Dwayne Morgan (PI)

Phone: (304) 547-5800 Ext. 231

Email: drm@trl.com

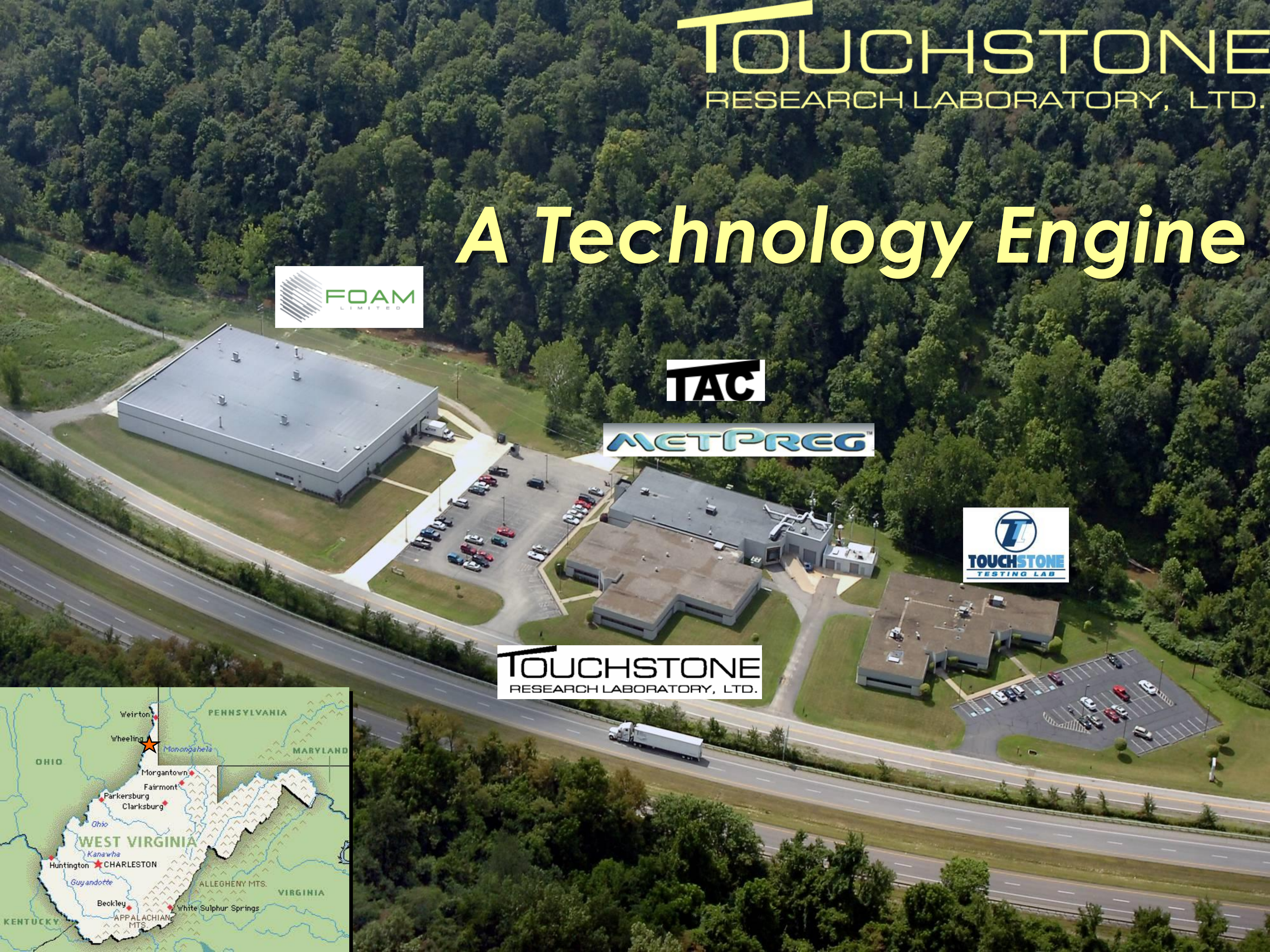
ADVANCED COAL PROCESSING PROJECT REVIEW MEETING
October 19-20, 2020

Outline

I. Background
II. Products Synthesized from Coal
III. Silicon Carbide Foam for Latent Heat Thermal Energy Storage
IV. Phase II Activities
    i. Objectives
    ii. Manufacturing Approach
    iii. Work Plan – Task Summary & Schedule
V. Phase II Accomplishments
    i. Product Improvement
        i. Mix Reformulation
        ii. Lower Ash Impurity
        iii. Engineered Product (Functional by Design)
    ii. Materials Characterization
        i. Mix Formulation Kinetic & Flow Properties
        ii. Bake (Pyrolysis) Optimization to Improve Yield
        iii. Microstructure, Mechanical & Thermal Properties
VI. Summary

Phase II Team

- **Department of Energy (Sponsor)**
  - National Energy Technology Laboratory (Grant Administration & Program Oversight)
    - Ms. Barbara Carney, Program Manager
    - Mr. Walter Strzepka, Contract Management Specialist
- **Touchstone Research Laboratory, Ltd.**
  - Leadership
    - Mr. Brian Joseph, President and CEO
    - Mr. Brian Gordon, R&D Director
  - Project Management
    - Mr. Dwayne Morgan, Sr. Research Scientist, Principal Investigator (PI) on this effort.
    - Mr. J. W. Freeland, Contracting Specialist
  - Technical Support
    - Analytical – Dr. Lou Hart and Mr. Brandon Coates
    - Processing - Mr. Fred Wade and Mr. Jacob Buck
    - Facilities and Equipment Installation – Mr. Jim Witzgall and Mr. Neven Cook
- **TechOpp Consulting Inc. (collaborator in the commercialization effort)**
  - Bob Fielder – President
  - Mary Ann Bonadeo – Project Manager (POC)



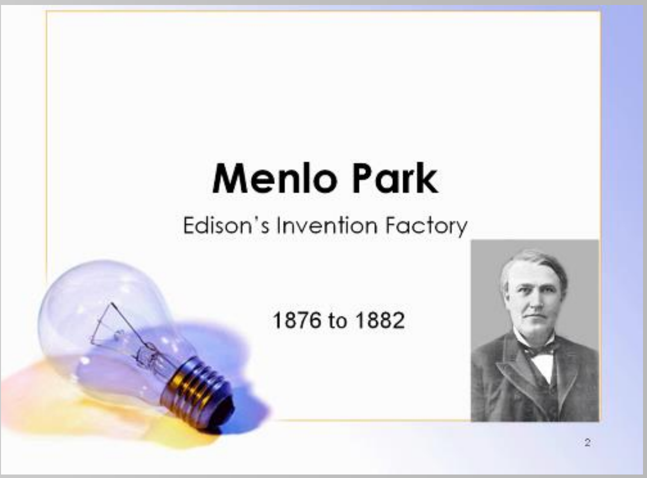
TOUCHSTONE
RESEARCH LABORATORY, LTD.
A Technology Engine
FOAM
LIMITED
TAC
METPREG
TOUCHSTONE
TESTING LAB
TOUCHSTONE
RESEARCH LABORATORY, LTD.
PENNSYLVANIA
OHIO
MARYLAND
Weirton
Wheeling
Monongahela
Morgantown
Fairmont
Parkersburg
Clarksburg
Ohio
WEST VIRGINIA
Kanawha
Huntington
CHARLESTON
Guyandotte
ALLEGHENY MTS.
VIRGINIA
Beckley
White Sulphur Springs
APPALACHIAN MTS.
KENTUCKY

“The Innovation Factory”

Award Winning

- National Blue Chip Enterprise Award
- 3M “Small Business Subcontractor of the Year”
- United Technologies “NASA Subcontractor of the Year”
- Southern Growth Policy Board “Governor's Cup”
- Inc. 500
- 3 Time Winner U.S. Small Business Administration National Tibbetts Award
- 5 Time Winner R&D 100 Awards

Unique R&D Technology & Patent Development Strategy



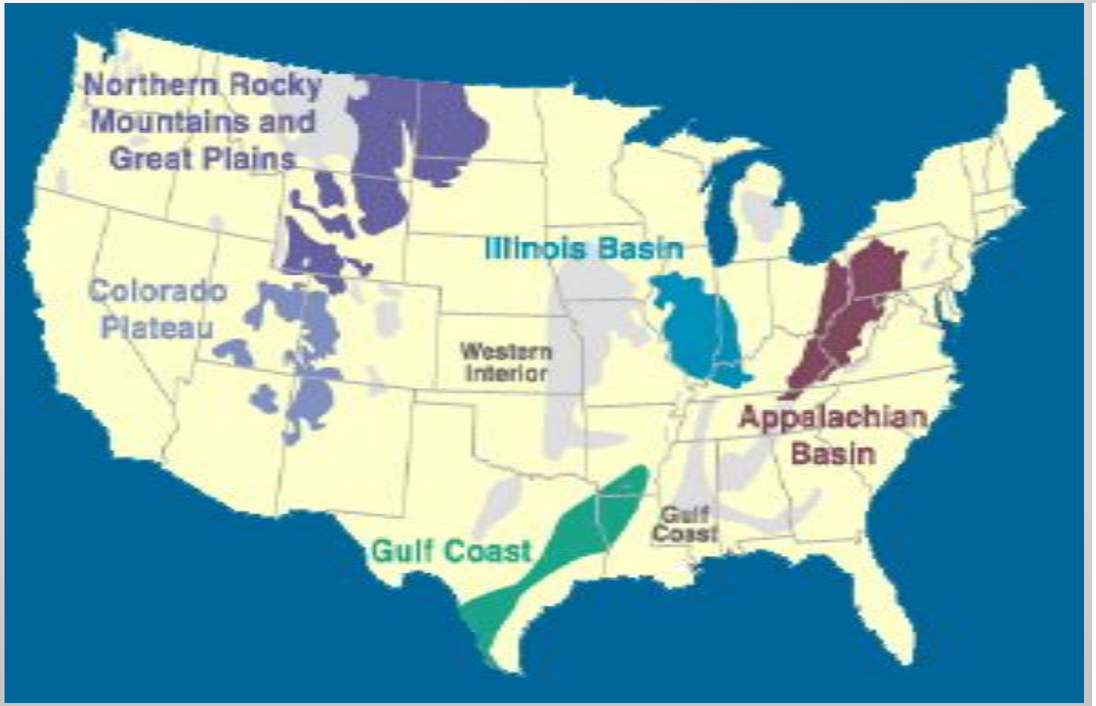
2020 – Navy Ship Repair

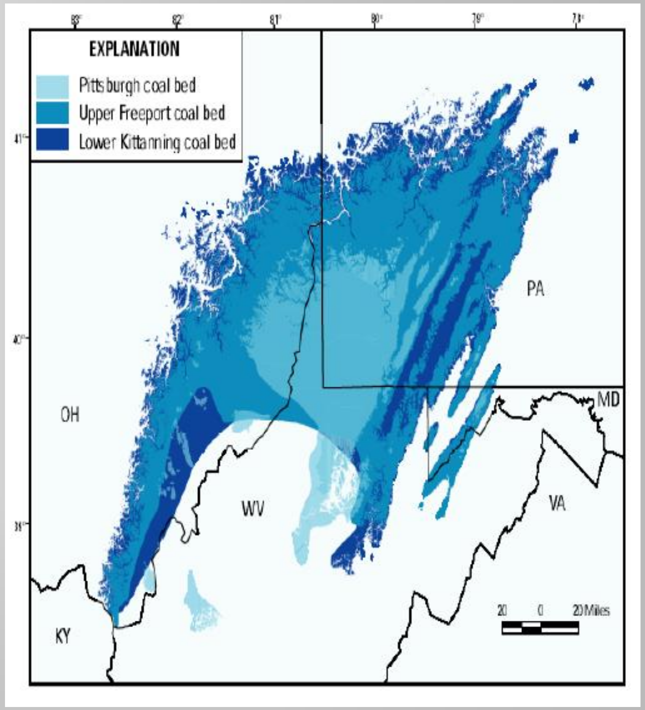
Touchstone’s successful R&D and Innovation processes are patterned from Thomas Edison’s Menlo Park model. This approach has enabled Touchstone to win multiple awards, including the prestigious R&D 100 award, using coal feedstock.

Products Synthesized from Coal

Coal Feedstock

Abundant Domestic Supply of Raw Materials

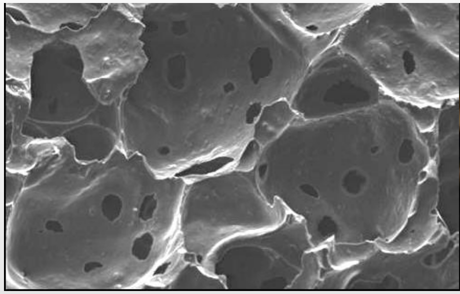




Largest Hydrocarbon Deposit in the World

CFOAM®

2004



Magnification 50X

CFOAM Exposed to 3000°F Acetylene Torch

Easily Machined

CFOAM® Physical Properties

Property	Unit	CFOAM
Density	g/cm^3	0.48
	lbs/ft^3	30
Porosity	%	79
Compressive Strength	MPa	16.04
	ksi	2.3
Tensile Strength	MPa	5.2
	ksi	0.76
Coefficient of Thermal Expansion	ppm/°C	5.0
Thermal Conductivity	W/m·K	0.5

CFOAM® is a next-generation structural carbon foam designed to meet growing demand for ultra high-performance engineering materials in the military, industrial, aerospace, and commercial product markets.

CSTONE®

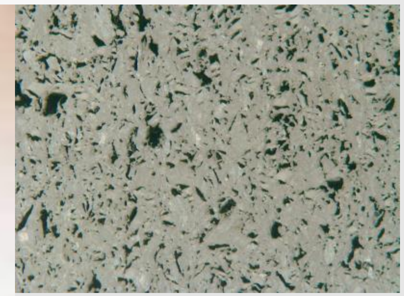
2007

R&D 100

CSTONE® Physical Properties

Property	Unit	CSTONE
Density	g/cm³	1.52
	lbs/ft³	95
Porosity	%	33
Compressive Strength	MPa	137.89
	ksi	20
Tensile Strength	MPa	20.68
	ksi	3.0
Coefficient of Thermal Expansion	ppm/°C	3.5
Thermal Conductivity	W/m·K	5-70

CSTONE® Heat Exchanger



Magnification 100X

CSTONE® Panels

CSTONE® is an extremely hard, high strength, high density carbon (95 lbs/cubic ft.). CSTONE has the potential to be used as an engineering material for rocket nozzles, high temperature/high impact surface material for vertical take-off and landing pads, furnace floors or other industrial application requiring excellent high temperature performance and chemical resistance.

Silicon Carbide (SiC) Foam for Thermal Energy Storage

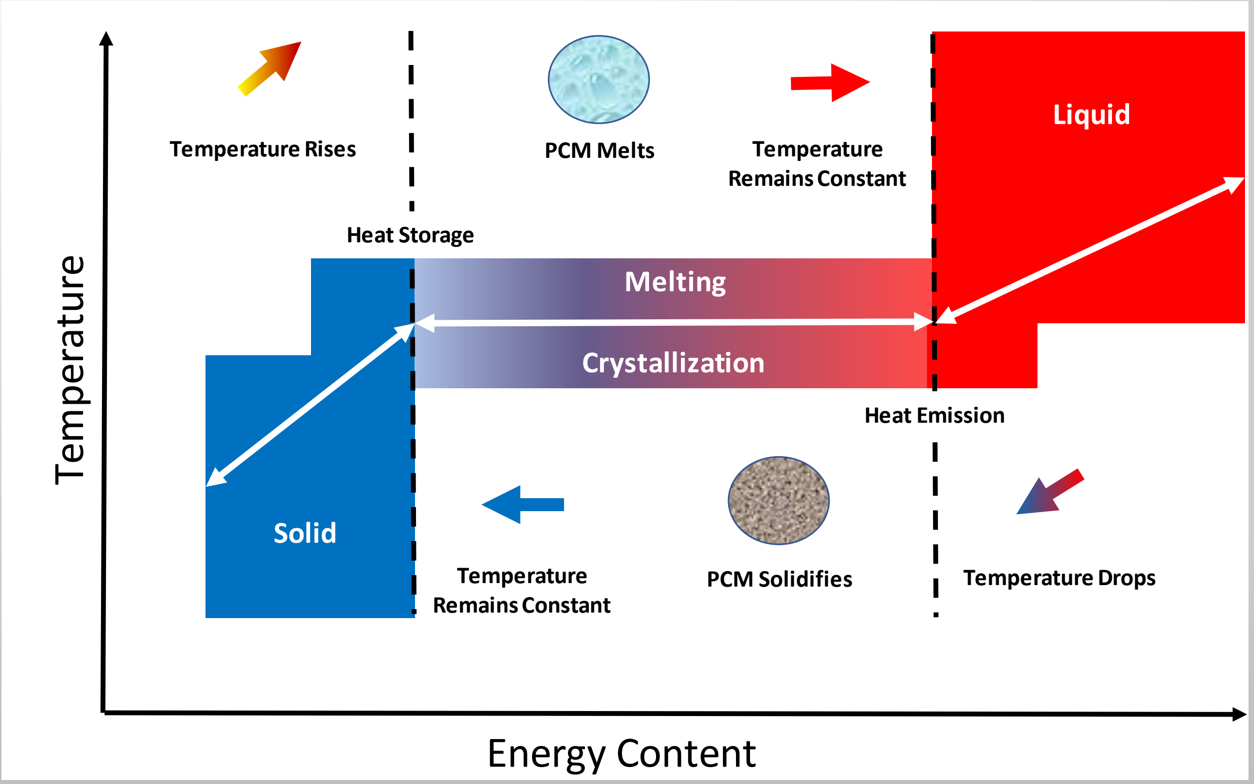
ADVANCED COAL PROCESSING PROJECT REVIEW MEETING
October 19-20, 2020

Silicon Carbide (SiC) Foam from Coal Feedstock

- High conductivity PCM (molten salt) substrate (SiC) that will make high efficiency CO_2 Brayton cycles (100 kW – 5 MW) possible.
- Suitable for application temperature > 1000°C.
  - Peak temperature for sCO2 Brayton Cycle 750 °C.
- Enhances heat transfer rate of molten salt
- High oxidation resistance up to 1200 °C.
- High corrosion resistance

Thermal Energy Storage

Sensible heat and Latent heat are not special forms of energy. Rather, they describe exchanges of heat under conditions specified in terms of their effect on a material or a thermodynamic system.



Sensible heat $\Rightarrow Q_{sensible} = mc\Delta T$

- Heat exchanged by a body or thermodynamic system
- Exchange of heat results in change of the temperature of the body or system
- <u>Change of heat does not result in volume or pressure change (i.e. constant).</u>

Latent heat $\Rightarrow$ **Q = mL, where** $L=L_f$ **or** L_v

- Heat exchanged by a body or thermodynamic system
- Exchange of heat results in no change of temperature of the body or system
- <u>Change of heat does result in volume or pressure change (Phase Change)</u>

Phase II Objectives

- **Focus on Process Optimization, Manufacturing Scaleup, and Systems Integration**
  - Demonstrate manufacturability of large panels that meets product & project definitions.
  - Verify product through internal testing and external customer trials.
- **Detailed product specifications defined and available in product technical data sheets (TDS).**
- **Technology defined and documented in Best Practice.**
- **Develop Phase III Plan.**

Silicon Carbide (SiC) Foam

General Goals:

- SAFETY, QUALITY, PRODUCTIVITY ...
- Scaleup and Optimize SiC-FOAM processes
- Data-driven

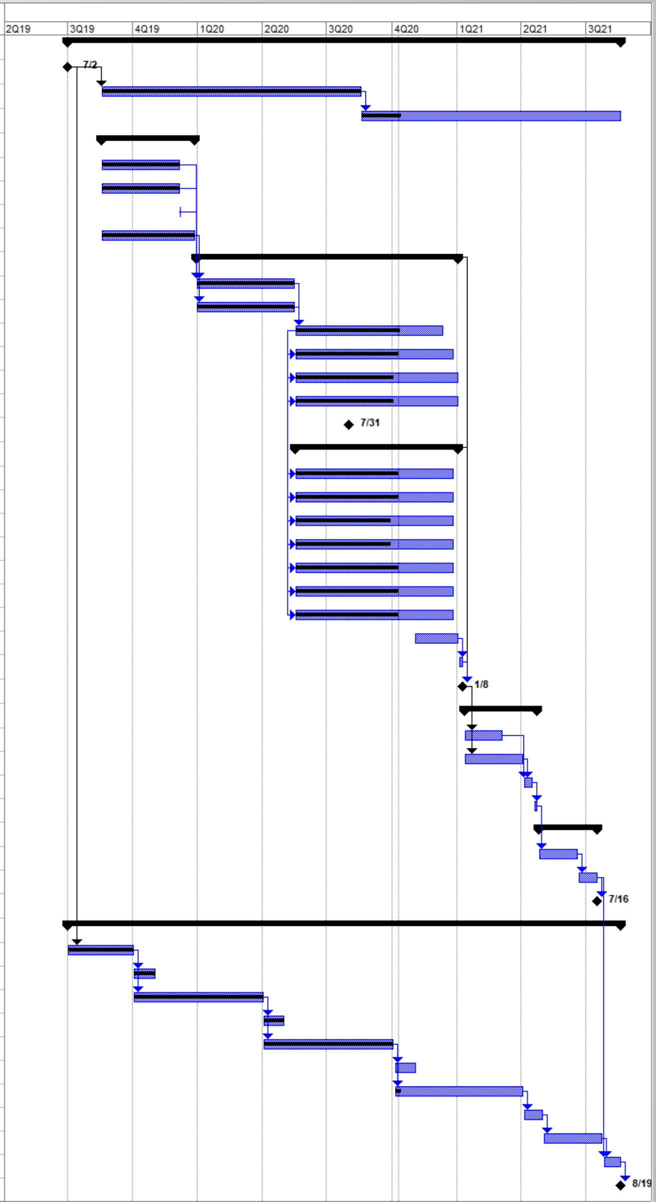
2018	2019-2020	2020-2021	
R&D	Small-Scale Production	Med-Scale Production	**Full-Scale Production**
		* Optimize Current Equipment for 24/7 ops	* Capital Investment – New Equipment

Phase II Work Plan
Task Summary

- Task 1: Project Definition, Startup, & Kickoff
- Task 2: Process Optimization and Scale-up
- Task 3: Testing & Validation
- Task 4: Update Analytical Models
- Task 5: Prototype Demonstration
- Task 6: Customer Evaluation – Phase III Plan
- Task 7: Reporting & Program Management

Phase II Schedule

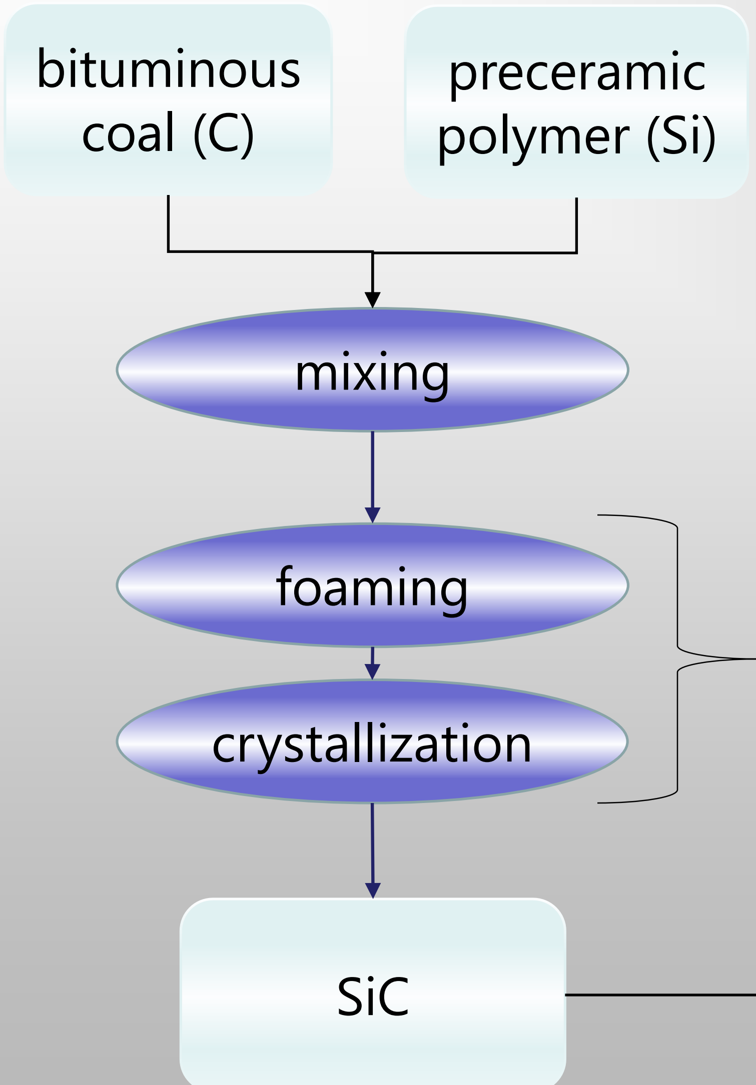
ID	Task Name
1	**DOE Topic 18-c SiC Foam for Gen3 PCM**
2	**Project Start Date**
3	**Budget Period 1**
4	**Budget Period 2**
5	**Task 1-Project Definition and Startup-Phase II Kickoff Meeting**
6	Establish Requirements
7	Update CSP and Commercial Applications
8	Kickoff Meeting
9	Equipment Procurement and Installations
10	**Task 2-Process Optimization and Scaleup**
11	Cold Mix Trials
12	Hot Mix Trials
13	Foaming with accepted mix design
14	beta-SiC Processing
15	alpha-SiC Processing
16	Phase Chase Material (PCM) Infusion Trials
17	***Milestone Review - Optimized Process***
18	**Task 3-Testing and Validation**
19	X-ray Methods
20	Microscopy Methods
21	Mechanical Testing
22	Thermal Conductivity ASTM C201
23	Thermal Expansion
24	Oxidation Resistance of SiC
25	Thermal Energy Storage (TES) Product Evaluation
26	Molten Salt Testing (corrosion)
27	**Task 4-Update Analytical Models**
28	***Milestone Review - Thermal Energy Storage Capacity***
29	**Task 5-Prototype Demonstration**
30	Manufacture PCM Panels
31	Infuse Panels with PCM
32	Document Best Practices
33	Send out Prototypes for Customer Validation
34	**Task 6-Customer Evaluation-Phase III Plan**
35	PCM Customer Product Evaluation
36	Phase III Plan
37	***Milestone Review - VOC***
38	**Task 7-Reporting/Program Mgmt**
39	Project Period No. 1
40	RPPR No. 1
41	Project Period No. 2
42	RPPR No. 2
43	Project Period No. 3
44	RPPR No. 3
45	Project Report No. 4
46	RPPR No. 4
47	Project Report No. 5
48	RPPR No. 5
49	End



Phase II Accomplishments

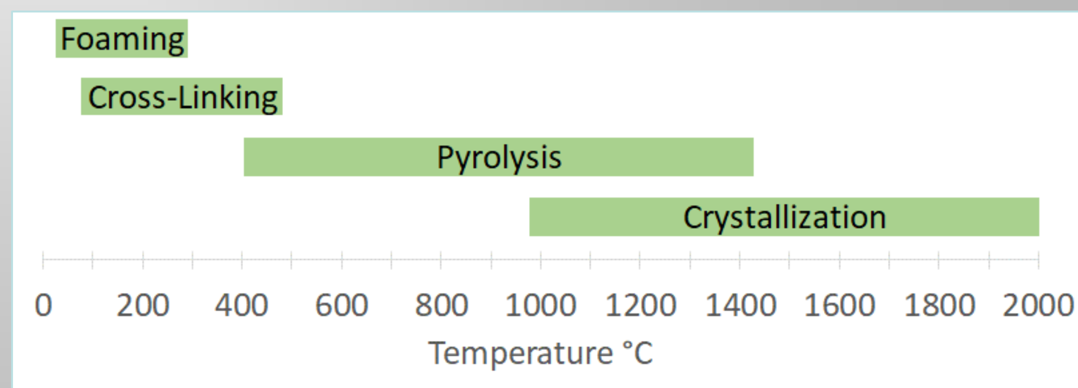
- **Early Production Trials**
  - Production scale prototype panels were successfully formed in Foaming via the Phase-I mix formulation:
    - 2”x24”x24” square panels
    - 3” (H) x 10” (ϕ) trim disk form factor
  - High shrinkage during pyrolysis impacts overall product yield and properties
  - Lack of capability in optimizing porosity with Phase I mix design
- **Developed a modified mix design for product improvement**
  - Indicates half the volumetric shrinkage over previous mix design.
  - Will have coal precursor with significantly lower ash and sulfur content, trials are in process.
  - It is anticipated that the modified mix design will enable Touchstone to engineer products for a range of density and porosity grades

Direct Synthesis Route
Silicon Carbide Foam from Coal



PH-I Research

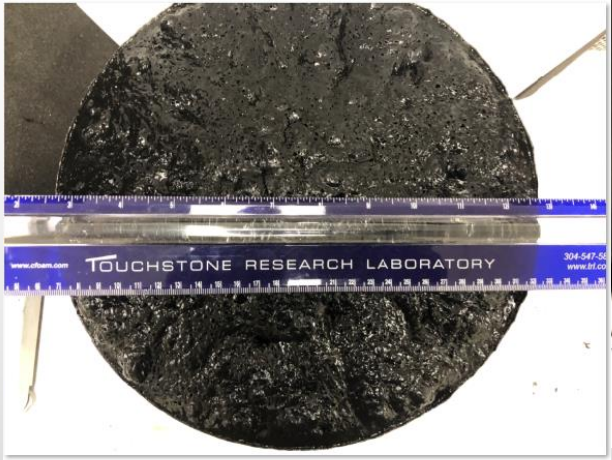
PH-II Development

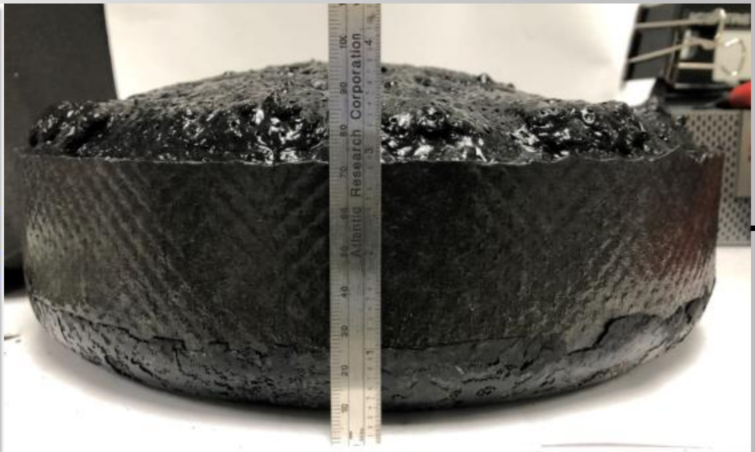


Polytype	Crystal Structure	Wt. %
3C (β-SiC)	**Zinc blende (cubic)**	**90 (±2.1)**
6H (α-SiC)	**Wurtzite (hexagonal)**	**10 (±1.6)**

3” (H) x 10”ϕ Disk Trimmed

Green – As Formed



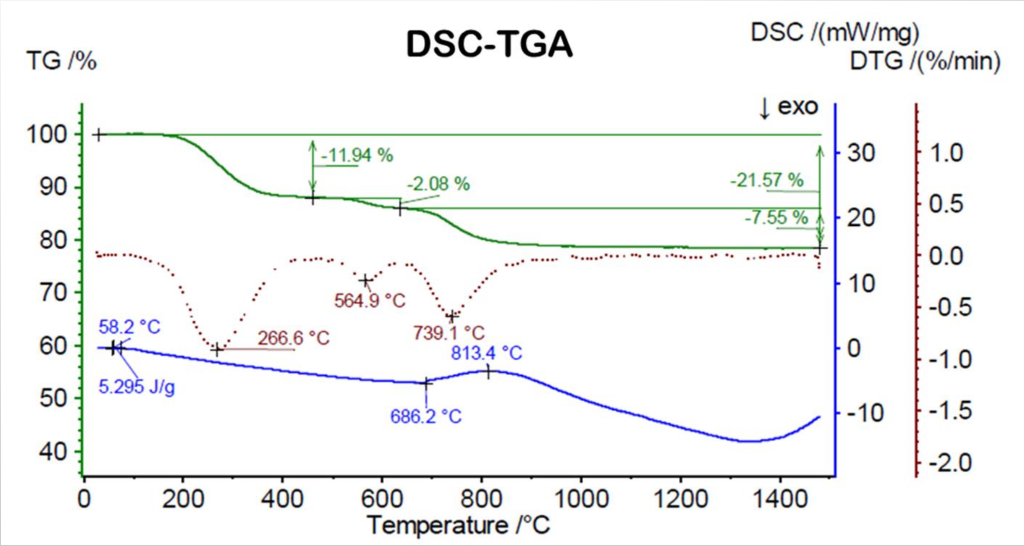


Post Pyrolysis

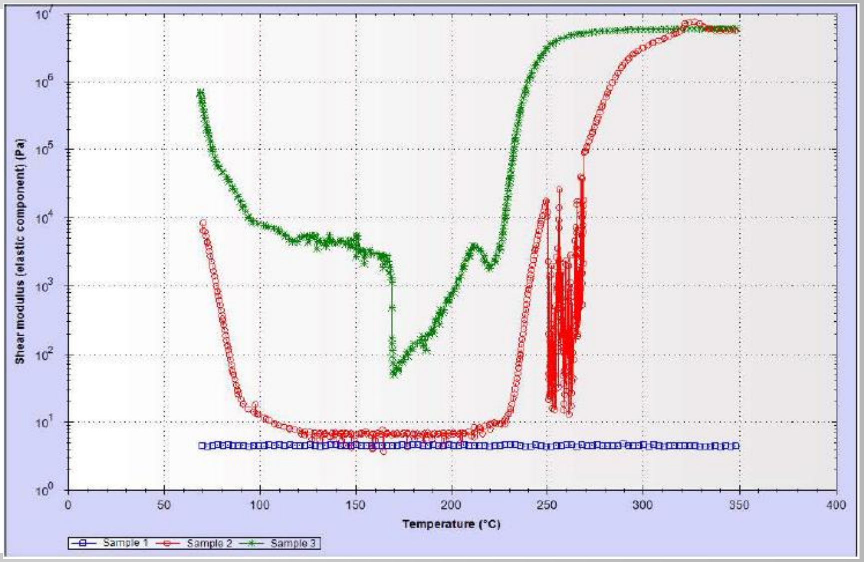
Green – Outer Skin Removed

Reaction Kinetics (DSC-TGA) Viscoelasticity (Rheology)

Differential Scanning Calorimeter (DSC)
Thermogravimetric Analysis (TGA)
Rate of Weight Loss (DTG)



Rheology (Shear "Storage & Loss" Modulus)



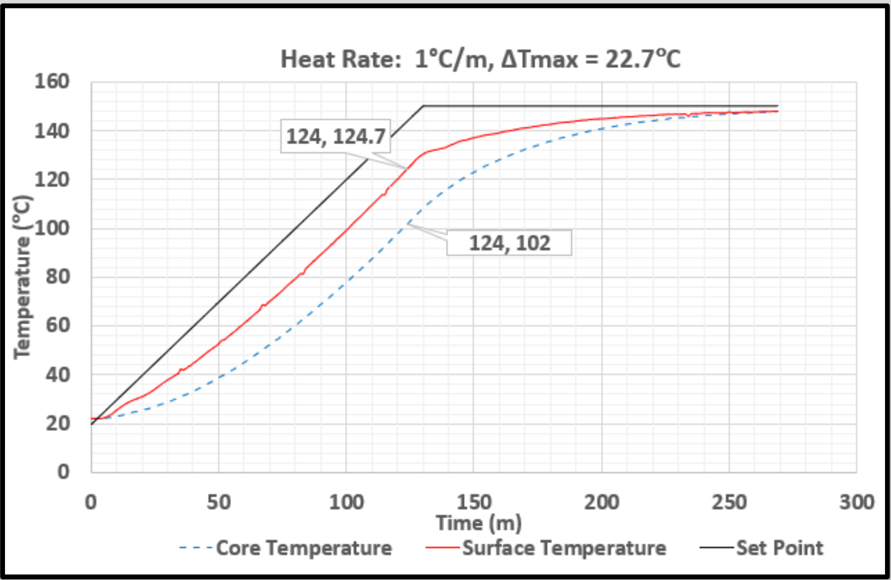
Thermal Stress Analysis (Bake Modeling)

Optimize heat schedules where thermal stress less than ultimate tensile stress:

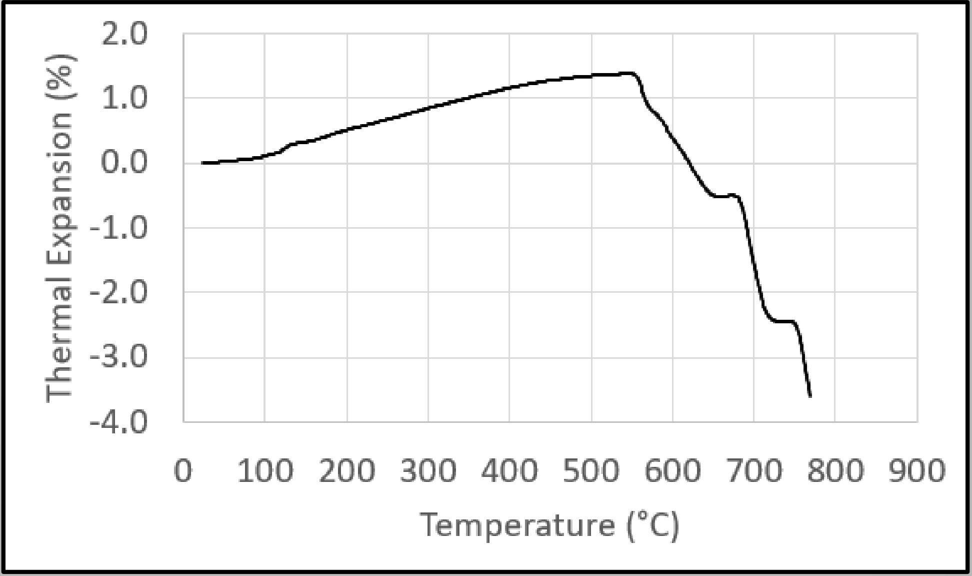
$$\sigma_{Tensile} > \sigma_{TH} \quad \therefore \ \sigma_{TH} = \varepsilon_{TH} \cdot E = \alpha \cdot \Delta T \cdot E$$

Where: σ_{TH} ***= Thermal Stress***
ε_{TH} ***= Thermal Strain***
E ***= Modulus of Elasticity***
α ***= Coefficient of Thermal Expansion***
ΔT ***= Temperature Differential***

Surface to Core Temperature Differential (ΔT)

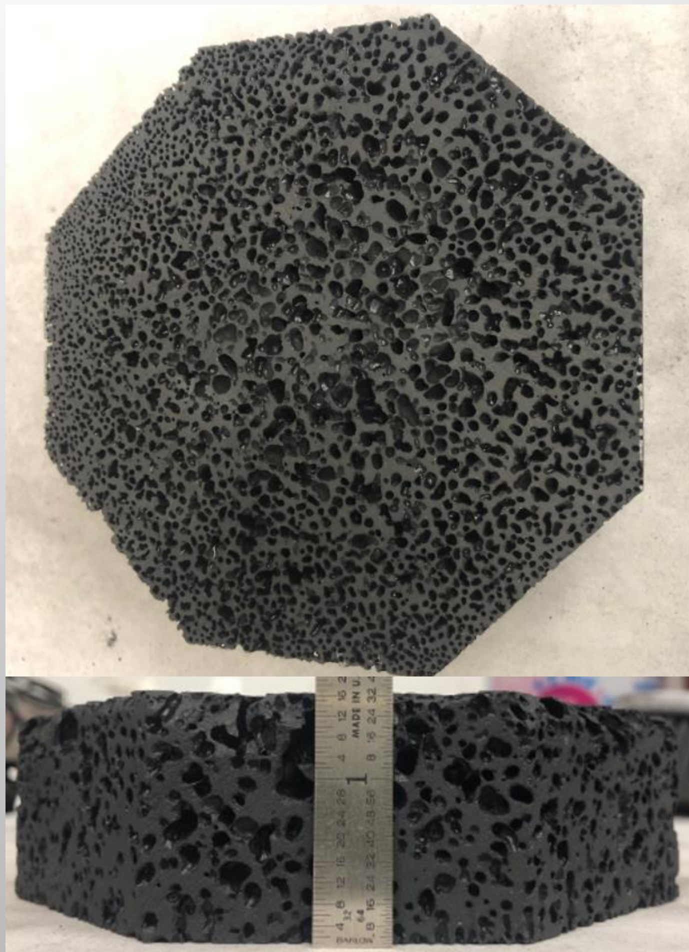


Thermal Strain (ε_{TH}) via Dilatometry (α)

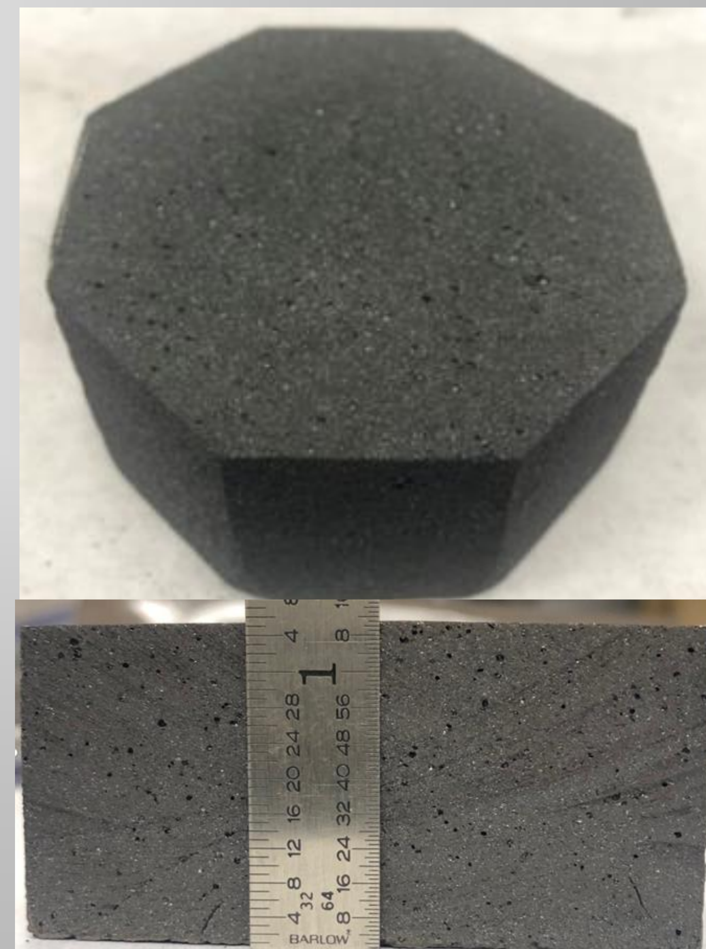


Macrostructure Comparison Pressurless Foam

Typical PH-I Porosity



PH-II Development



Silicon Carbide Foam Fine Porosity

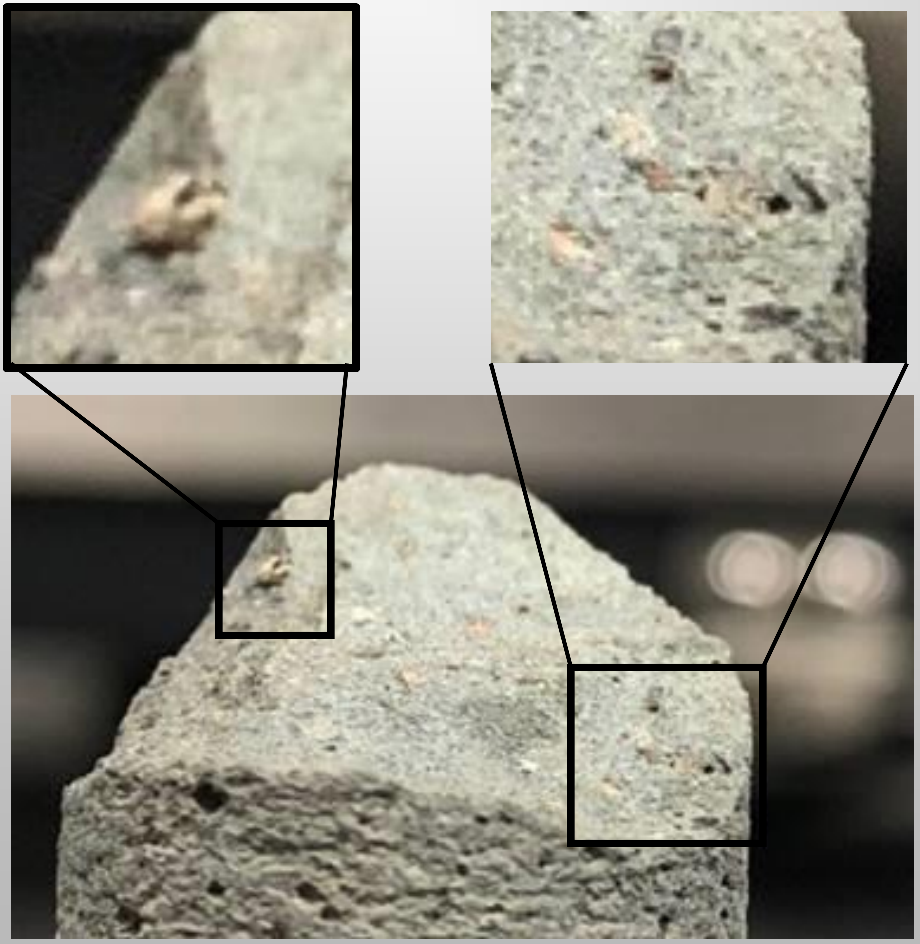
Recipe	Shrinkage
PH-I*	38.64%
PH-II**	22.66%

Phase II Modified Process Helium Density Results

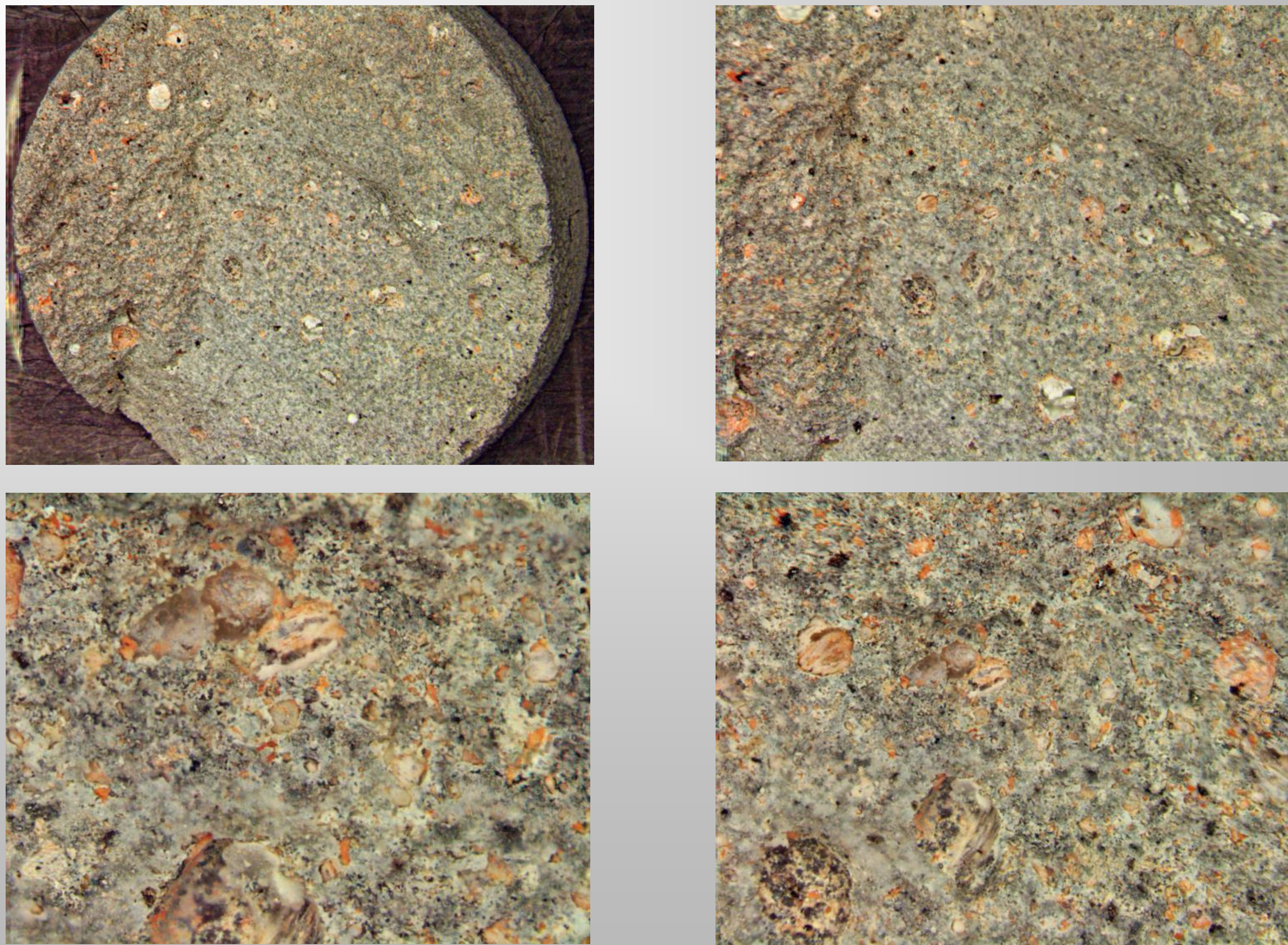
Description	Measurement	Unit
Sample Diameter	16	mm
Sample Length	35	
Sample Diameter	1.6	cm
Sample Length	3.5	
Area	2.011	cm^2
Bulk Volume	7.037	cm^3
Weight	4.8256	g
SiC Theoretical Density	3.21	g/cm^3
Apparent Bulk Density	**0.686**	**g/cm^3**
Porosity	**79%**	**%**
Skeletal Volume	1.5662	cm^3
Skeletal Density	***3.081***	***g/cm^3***
% Theoretical Density	***96%***	***%***

Phase II Modified Process Core Drilled Specimen 5/8”ϕ

Ash Agglomerate Fractography Detection Method

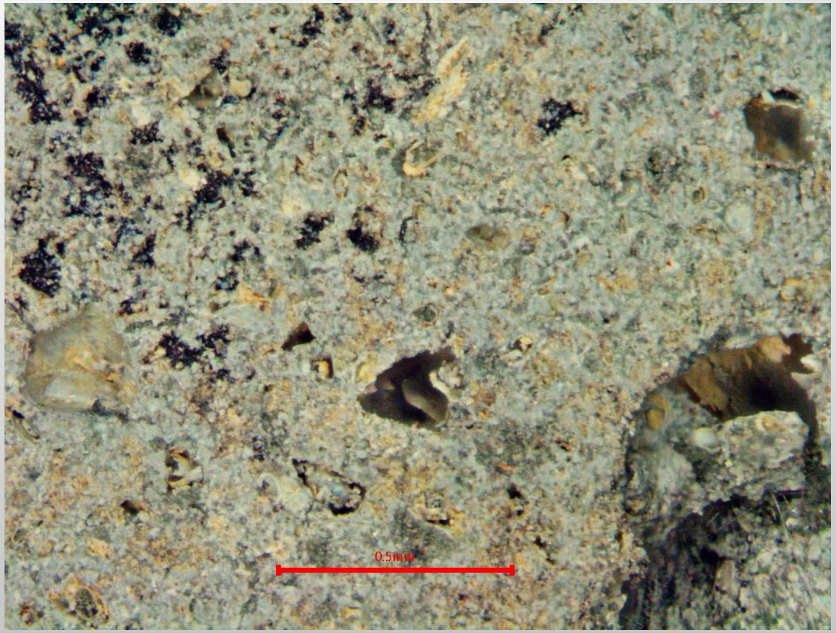


Stereomicroscopy Fracture Surface

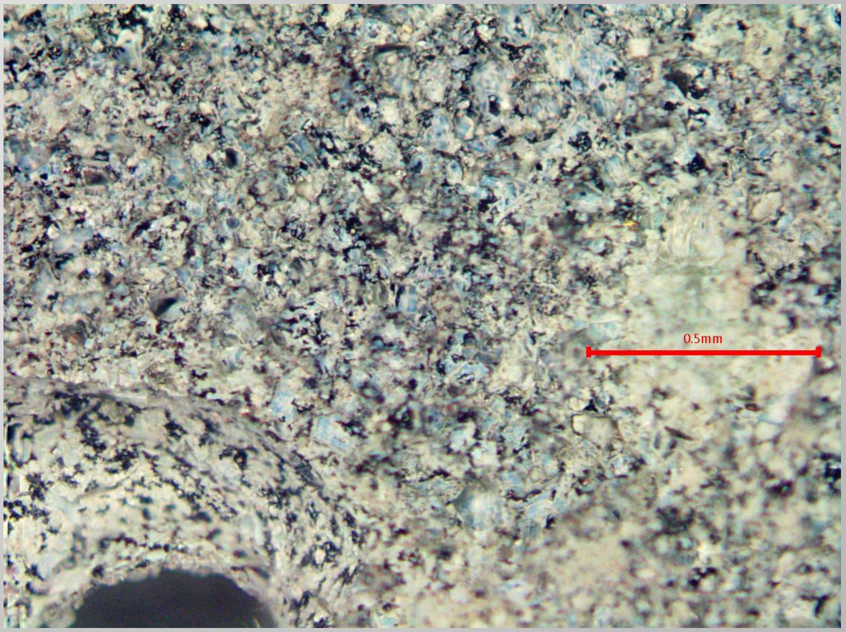


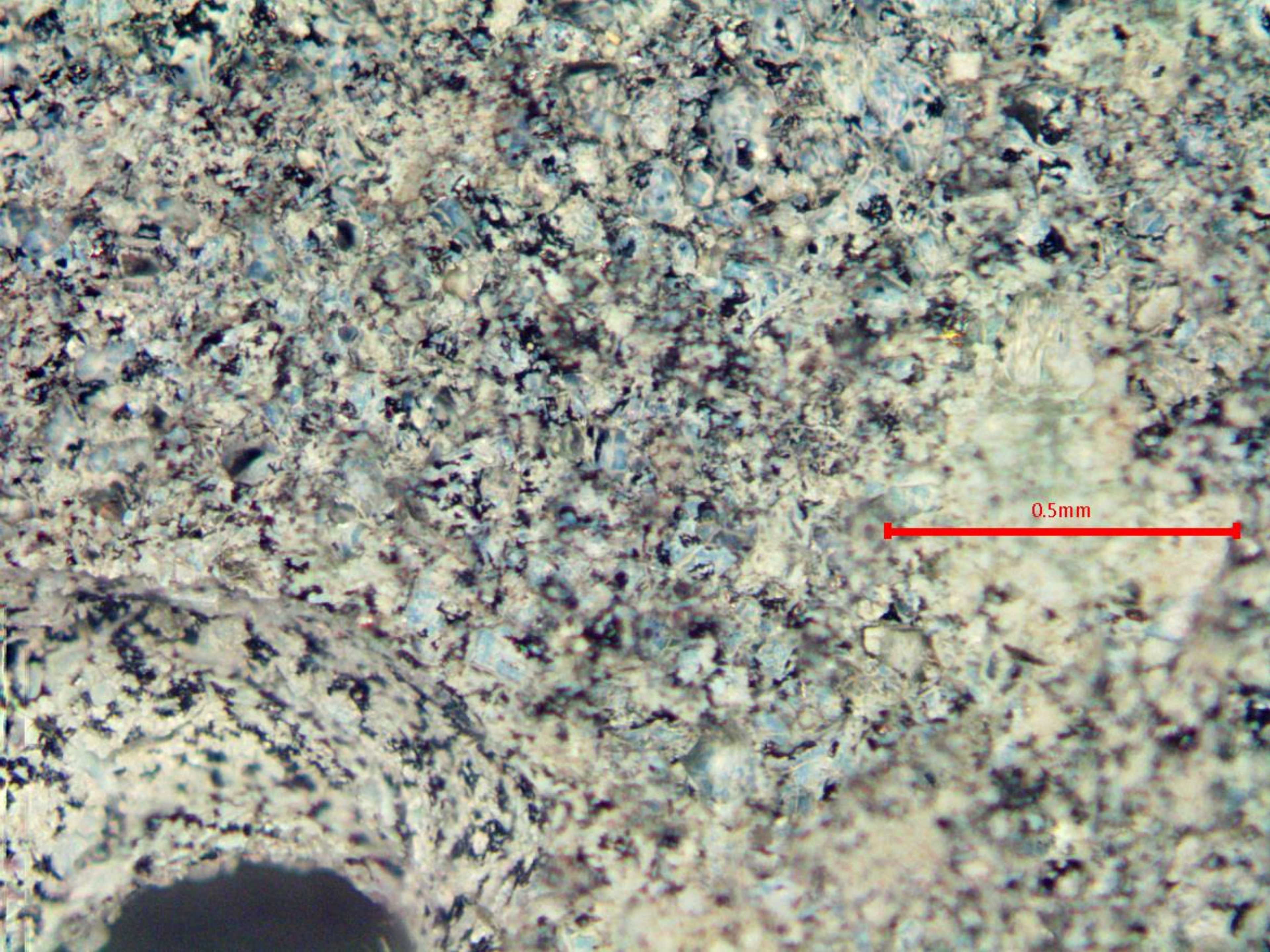
Stereomicroscopy
Magnification 63x, Extended Depth of Field

Typical Coal
Ash 8%, Sulfur 2%



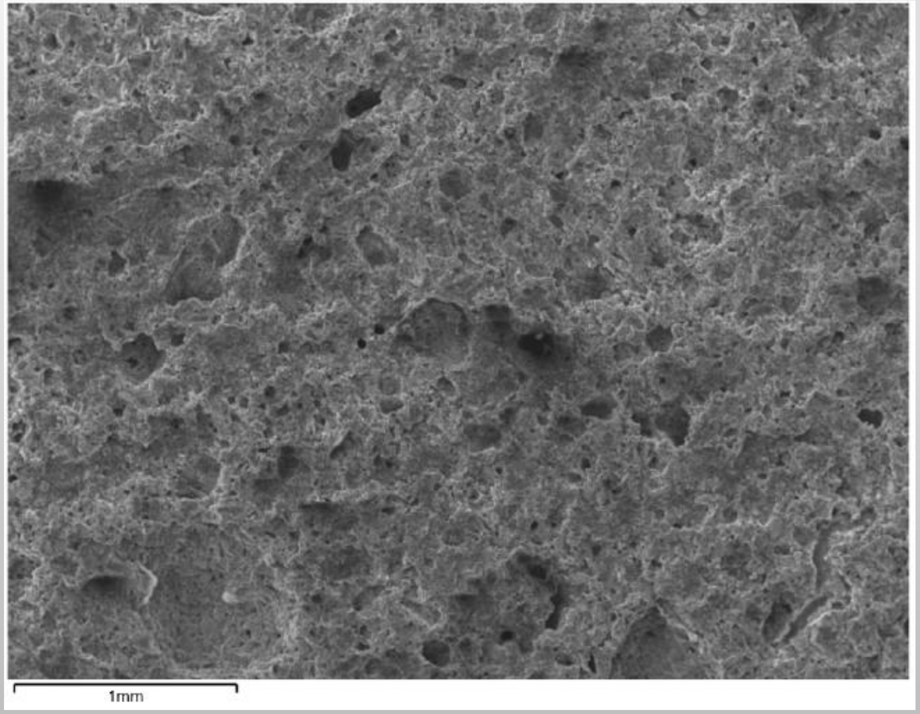
Cleaner Coal
Ash 1.34%, Sulfur 0.81%

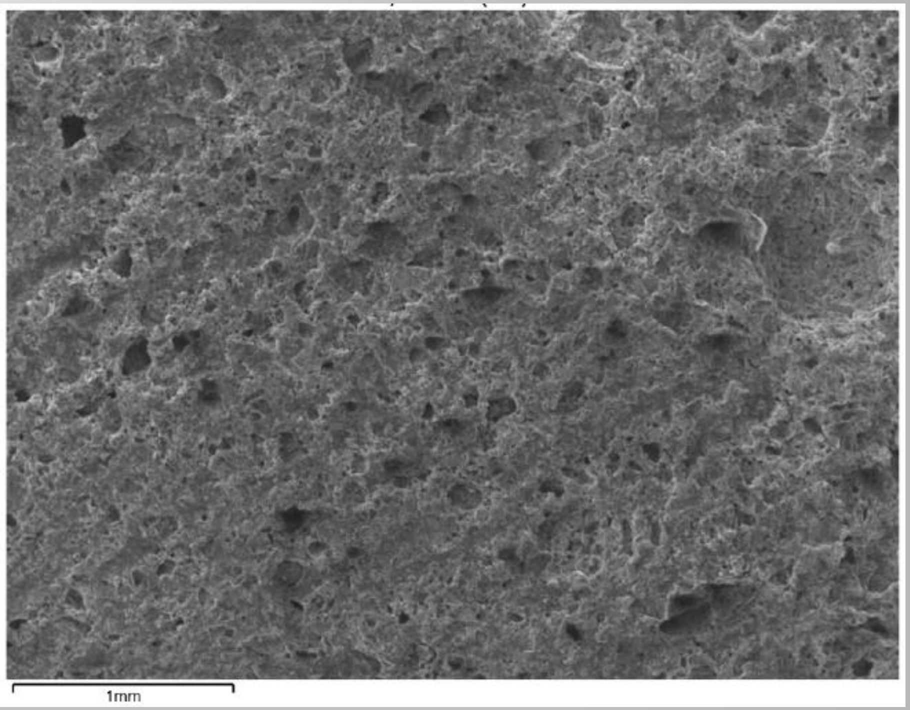



0.5mm

Scanning Electron Microscopy (SEM) at 32X Secondary Emission (SE)

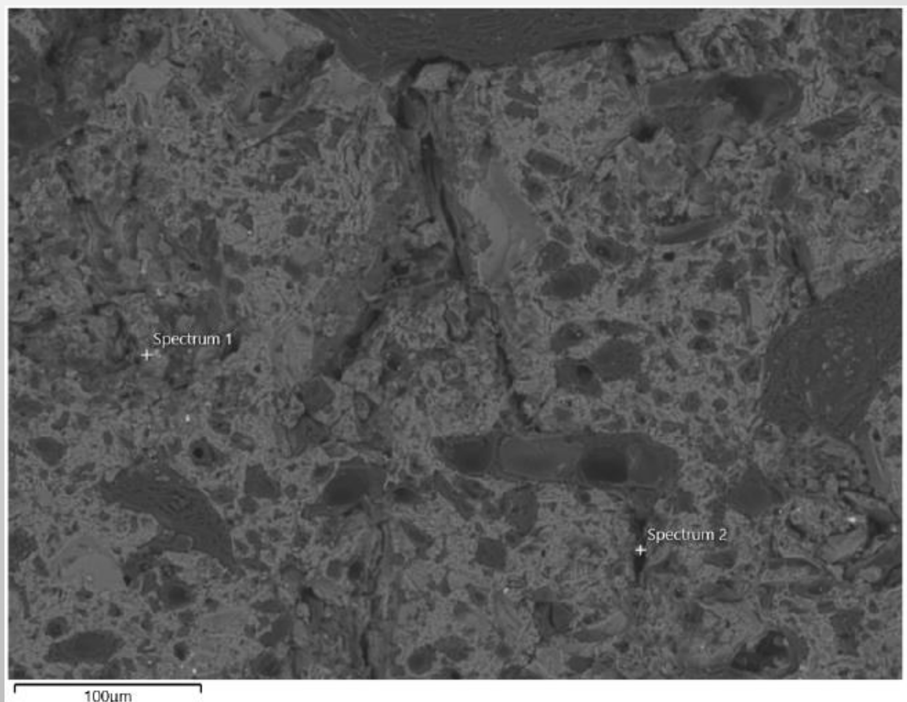
Phase II – Modified



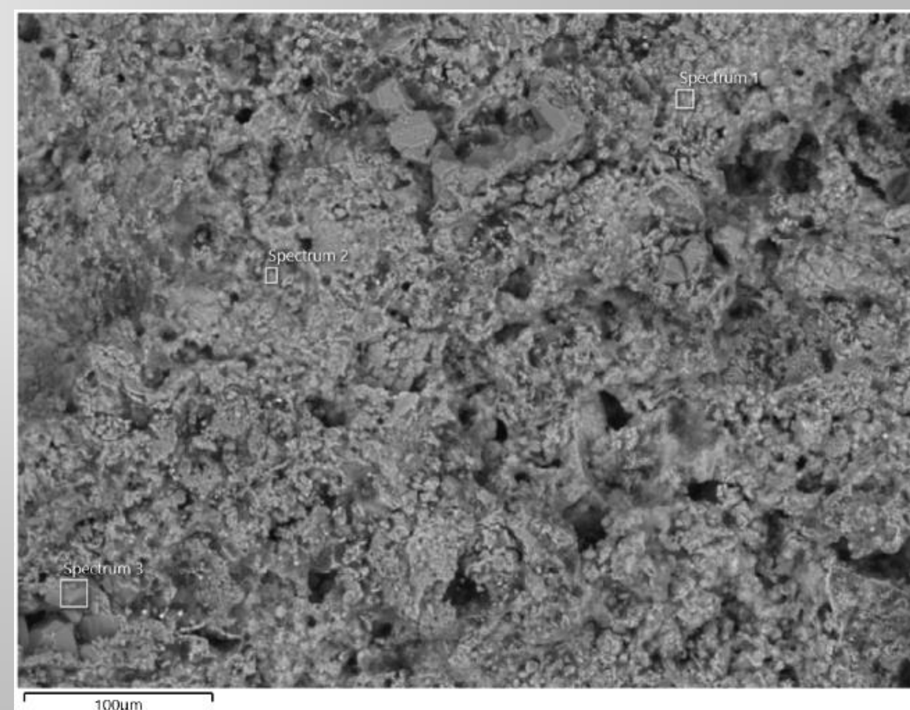


Scanning Electron Microscopy (SEM) at 270X Backscatter Emission (BSE)

Phase I - Structure

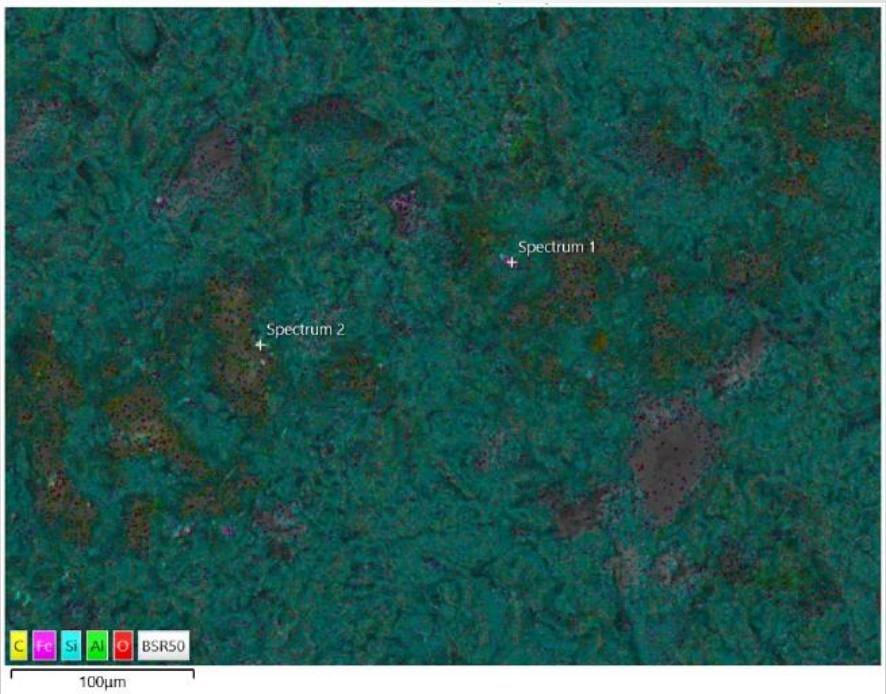


Phase II – Modified



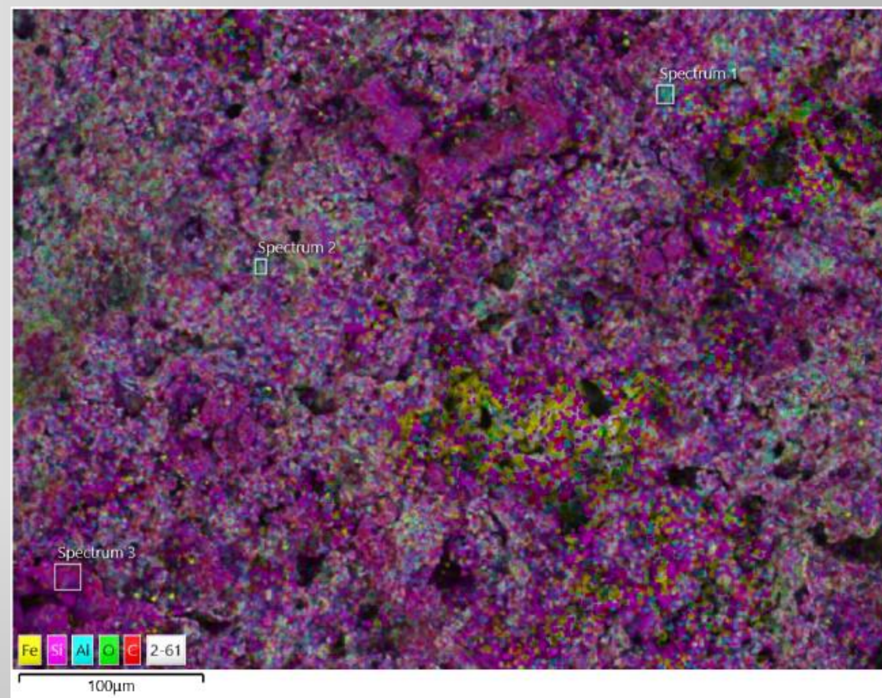
Bit Map Image 270X

Phase I Structure



Element	Line Type	Wt%	Wt% Sigma	Atomic %
Si	K Series	56.52	0.47	40.61
C	K Series	20.41	0.63	34.29
O	K Series	17.76	0.23	22.41
Fe	K Series	3.07	0.11	1.11
Al	K Series	1.85	0.04	1.39
Ca	K Series	0.39	0.04	0.20
Total		100.00		100.01

Phase II – Modified



Element	Line Type	Wt%	Wt% Sigma	Atomic %
Si	K Series	55.30	0.26	38.41
C	K Series	25.17	0.32	40.89
O	K Series	15.32	0.14	18.68
Fe	K Series	2.74	0.07	0.96
Al	K Series	1.46	0.03	1.06
Total		99.99		100.00

Spectrum Elements

Phase I Structure – Bit Map @270x

Spectrum 1

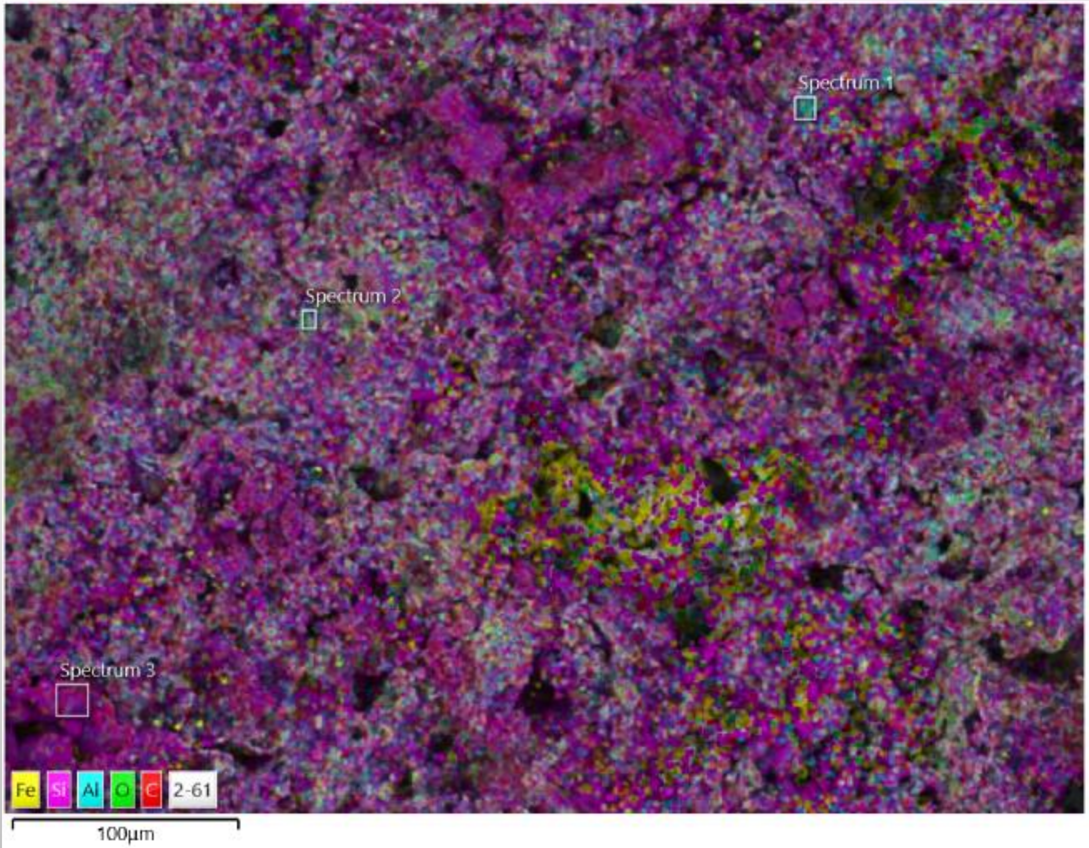
Element	Line Type	Wt%	Wt% Sigma	Atomic %
Fe	K Series	66.26	1.18	35.15
B	K Series	11.75	1.55	32.21
C	K Series	9.23	0.29	22.77
Cr	K Series	7.50	0.14	4.27
O	K Series	1.80	0.09	3.33
Ni	K Series	1.57	0.08	0.79
Cu	K Series	0.88	0.1	0.41
Si	K Series	0.75	0.02	0.80
Al	K Series	0.25	0.02	0.27
Total		99.99		100.00

Spectrum 2

Element	Line Type	Wt%	Wt% Sigma	Atomic %
Cu	K Series	54.16	0.42	22.69
C	K Series	26.07	0.56	57.79
Si	K Series	11.35	0.1	10.76
O	K Series	3.81	0.11	6.33
Sn	K Series	1.88	0.08	0.42
Al	K Series	1.21	0.04	1.19
Fe	K Series	1.12	0.06	0.54
Ca	K Series	0.31	0.03	0.21
S	K Series	0.08	0.02	0.07
Total		99.99		100.00

Bit Map Image 270X

Phase II – Modified



Spectrum 1

Element	Line Type	Wt%	Wt% Sigma	Atomic %
Si	K Series	52.03	0.15	47.38
O	K Series	19.53	0.18	31.23
Al	K Series	14.69	0.07	13.93
Fe	K Series	7.33	0.11	3.36
Ca	K Series	4.30	0.05	2.74
Ti	K Series	1.29	0.05	0.69
P	K Series	0.56	0.04	0.46
S	K Series	0.28	0.03	0.22
Total		100.01		100.01

Spectrum 2

Element	Line Type	Wt%	Wt% Sigma	Atomic %
Si	K Series	52.67	0.12	43.23
O	K Series	32.99	0.13	47.54
Al	K Series	6.74	0.04	5.76
Fe	K Series	4.21	0.07	1.74
Ti	K Series	2.22	0.04	1.07
Ca	K Series	1.17	0.03	0.67
Total		100.00		100.01

Summary

- **Process Optimization:**
  - Current mix/process methodology creates high shrinkage during pyrolysis and subsequently lowers product yields.
    - Preliminary results using alternative formulation and processes indicate reduction in shrinkage by 50%.
    - Alternative formulation produces fine and well distributed porosity without sacrificing high void content (79%).
  - Coals with high ash impurity produce inferior SiC product.
  - Implementing the alternative mix design with cleaner coal, i.e. ash and sulfur ≤ 1%, is believed to allow microstructures to be engineered for optimal density, porosity, and properties.
- **Path Forward**
  - Cleaner coal formulation trials.
  - Characterize materials.
    - Microstructures.
    - Phase identification and crystallinity via XRD.
    - Physical and mechanical properties.
    - Thermophysical properties and heat transfer and storage using PCM.
- **Manufacture medium to large prototype panels for customer evaluation.**

Thank You