Flexible Ablators Char Depths   LHMEL Test Results

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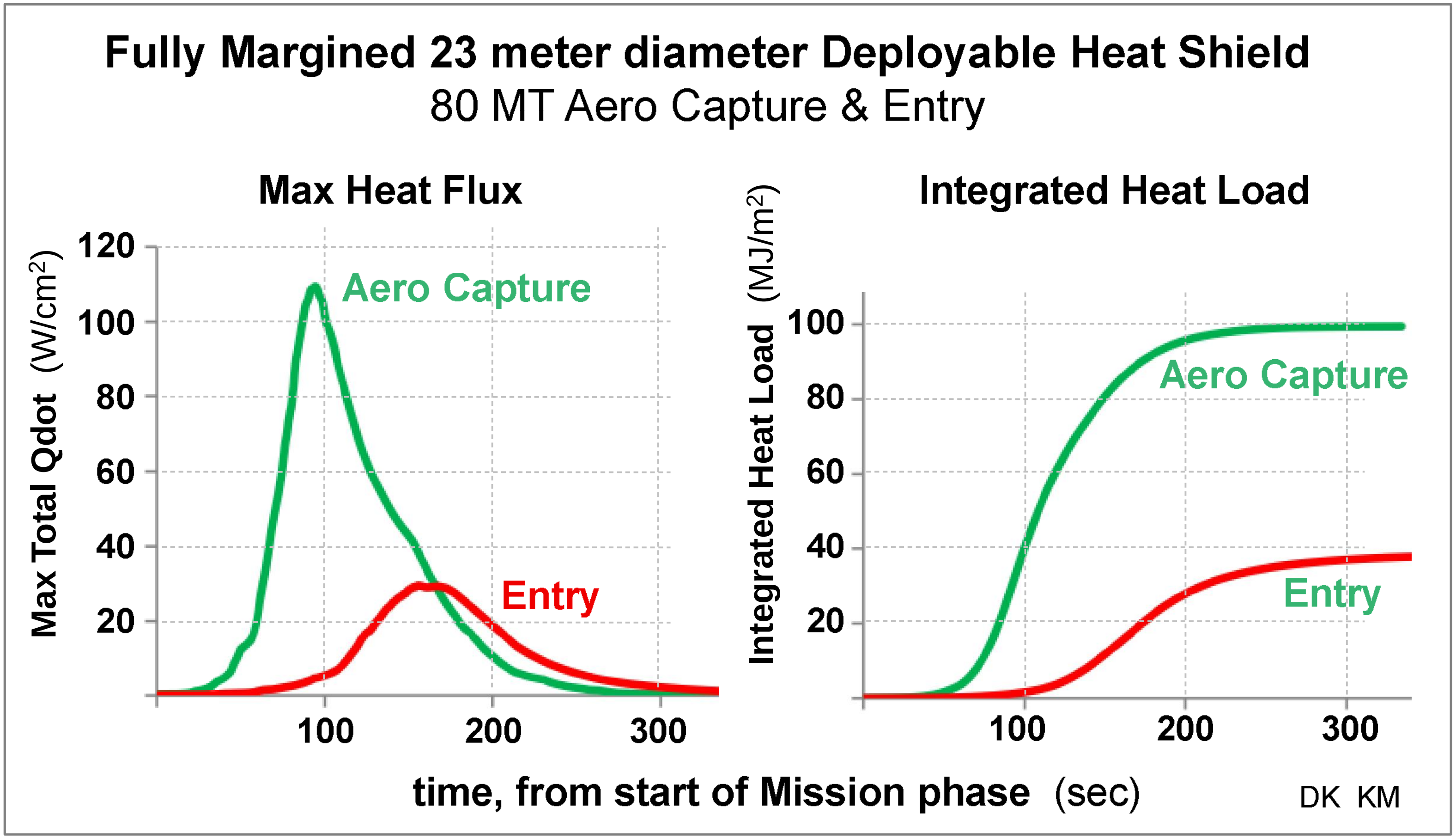
Flexible Ablators Char Depths: *Outline*

- Motivation
- Background
- Approach
- Test Results
- Summary & Conclusions

Flexible Ablators Char Depths: *Motivation*

In 2008, NASA began a 3-year study to establish entry system technologies required to put 40+ metric tons on the surface of Mars* *state-of-the-art ~ 1 MT

Concepts included ablative flexible TPS and dual pulse entry



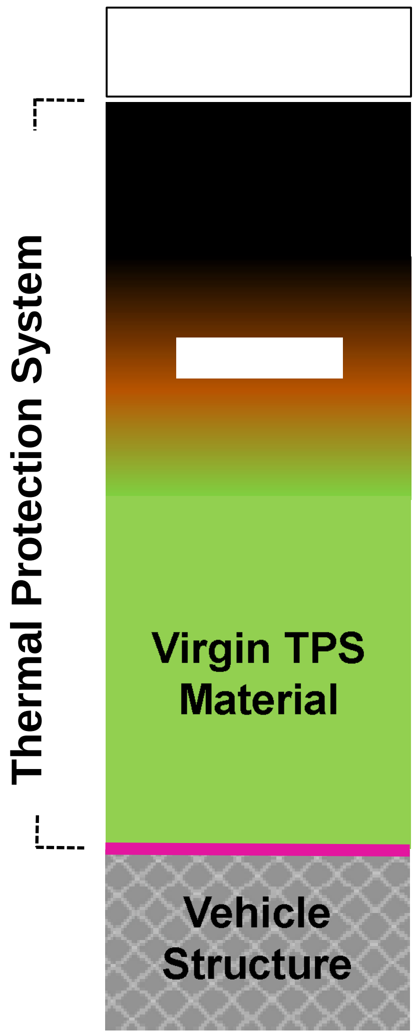
Flexible Ablators Char Depths: *Background*

Flexible Ablator Technology Development, FY 10 - 14

- **Determine evaluation criteria** to define successful development
- **Identify promising materials** with flexible matrices / substrates
  - carbon, silica, and polymer based felts / cloths
  - organic / inorganic blended materials
- **Investigate resins, additives, solvents** for flexible composites
- **Utilize lower cost screening tests** to determine viability
  - Aerothermal screening in NASA Ames X-jet plasma torch
  - Thermal screening in radiant environment at Wright-Patterson AFB Laser Hardened Materials Evaluation Laboratory (LHMEL)
  - Aerothermal screening in NASA Johnson TP2 arc heater
  - Fold testing for stowability effects
- **Downselect materials** for further technology (TRL) maturation

Flexible Ablators Char Depths: *Background*

Accurate thermal prediction and analysis requires proper modeling of the char and pyrolysis zones – including reaction kinetics



Recessed material - vaporization, sublimation, spallation or shrinkage

Highest temperature outer mold line (OML) region.
Coking, other reactions may occur. Organics have burned out, leaving substrate material or a refractory compound (e.g. C, SiO_2, Si-C).

Sufficient temperature for chemical decomposition of pyrolyzing components, which vary by type (e.g. phenolic,silicone) and processing. CMA and FIAT define regions by computed density.

Original material, thermally and chemically unchanged, typically consisting of a substrate and resin

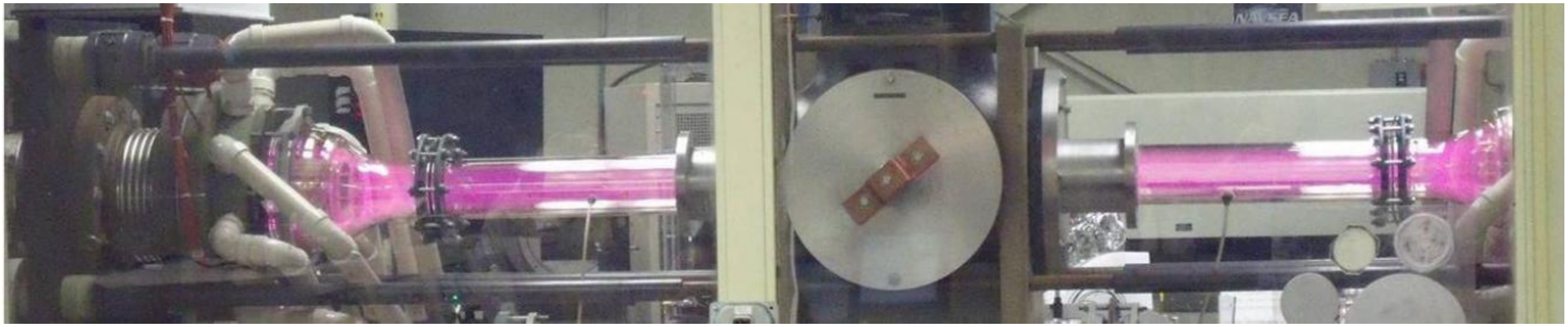
Inner mold line (IML) interface between TPS and vehicle structure

Temperature and heat load limits of the vehicle structure drive TPS requirements and sizing with margins

Flexible Ablators Char Depths: *Background*

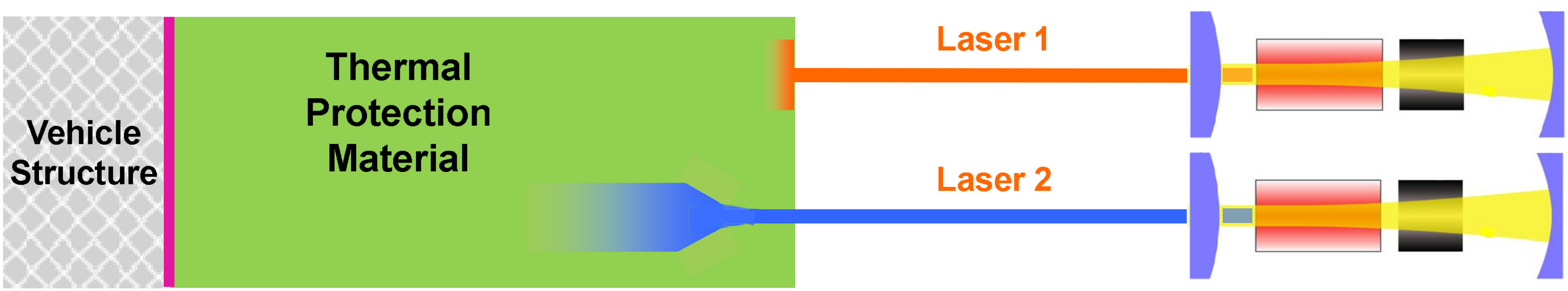
Laser Hardened Materials Evaluation Laboratory (LHMEL)

- Reliable, calibrated, and economical laser test facility located at Wright-Patterson AFB and operated by the U.S. Air Force Research Lab
  - Predictable high quality beam, NIST traceable diagnostics
  - High test rate (to 50/day) with purged test box - inert N_2 environment
- LHMEL I is a 15 kW CO_2 laser @ 10.6 micron wavelength
- Newer 10kW IPG Photonics Fiber Laser (1.07 micron) also used
- LHMEL II is a 100 kW CO_2 laser (not part of screening tests)



Flexible Ablators Char Depths: *Background*

- **Primary laser used in these screening tests in order to simulate Mars aero capture and entry heating was the 10.6 micron CO_2 laser – LHMEL I**
- **However, a second laser with a shorter wavelength - 10kW IPG Photonics Fiber Laser at 1.07 micron - was also used**
- **WHY? - hypersonic entry can produce significant shock layer radiation, with energy concentrated in characteristic wavelengths** (e.g. vacuum ultra-violet and near infrared)
- **Therefore, 2 lasers with a factor of 10 difference in wavelength used to assess effects from wavelength, with the IPG laser operating near infrared**



Example: Laser 1 radiant energy absorbed at or near surface.
Radiant energy from Laser 2 travels further in-depth.

Flexible Ablators Char Depths

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Flexible Ablators Char Depths: *Objective*

OBJECTIVE: Screening a broad array of flexible ablative TPS materials

- **Comparing within classes**
- **Comparing across classes**
- **Assessing response to different wavelengths** (near infrared vs. mid infrared)

Test Material	Type	Fiber Substrate	Pyrolyzing Component	Characteristics
Morgan-Phenolic FMI-Phenolic	Carbon Phenolic	Carbon Felt	Phenolic	High heat flux/ heat load capabilty
Morgan-S-T FMI-S-T FMI-S-B FMI-S-T-A1 FMI-S-T-A2 BFFAB Carbon Felt-silicone	Carbon Felt Silicone	Carbon Felt	Silicone resin	Oxidation protection
AFRSI-S-B Refrasil-S-B SIRCA (as Reference)	Glass Fiber w/ Silicone	Silica or Glass Fiber Felt	Silicone resin	Oxidation resistent, efficient at ~ 100 W/cm^2
Carbon/PBI-P-T PBI-S-B	Flex Polybenzi-midazole (PBI) fiber	Carbon or PBI Needled Felt	PBI fiber	Exploit high temp pyrolysis of charring fibers
LM3S Silica Felt Blanket LM2Z Zirconia Felt Blanket BFFA Impregnated Nextel	Layered	Carbon or Silica or Zirconia Felt layers	Silicone resin	Thin, tailorable, flexible. Integrate insulation layer by stictching.

KEY: S – silicone P – phenolic F, A* - additive B, T – solvents

Flexible Ablators Char Depths: *Approach*

LHMEL I Test Matrix Dual Pulse for Nominal, High Heating Conditions			
Mission Type	**Phase**	**Max Heat Flux** W/cm^2	**Duration** sec
Nominal	Aero Capture	115	30
	Entry	30	100
High Heating	Aero Capture	450	25
	Entry	115	50

- **Bondline thermocouples used**
- **Exposed area spot size**
  - 2.8” (7.1 cm) diameter for nominal conditions
  - 1.8” (4.6 cm) diameter for higher flux conditions

Flexible Ablators Char Depths: *Approach*

Test matrix of materials
by laser, mission type, and mission phase

Flexible Ablator Screening Test Test Matrix	NOMINAL HEATING			HIGH HEATING	
	Fiber Laser	CO2 Laser		CO2 Laser	
Test Material	Aero Capture	Aero Capture	Entry	Aero Capture	Entry
Morgan-Phenolic	■	■		char depth?	char depth?
FMI-Phenolic-F		■		■	■
FMI-Phenolic		■		■	■
Morgan-S-T	■	■		■	■
FMI-S-T	■	■		■	■
FMI-S-B	■	■		■	■
FMI-S-T-A1	■	■		■	■
FMI-S-T-A2	■	■		■	■
BFFAB Carbon Felt-silicone	■	■		■	■
AFRSI-S-B	■	■	■		
Refrasil-S-B	■	■	■		
SIRCA (as Reference)	■	■	■		
Carbon/PBI-P-T	■	■	■	■	char depth?
PBI-S-B		■	■	■	■
LM3S Silica Felt Blanket	■	■	■		
LM2Z Zirconia Felt Blanket	■	■	■		
BFFA Impregnated Nextel	■	■	■		

Expectations & Terminology

'*Well behaved*' or '*good*' means

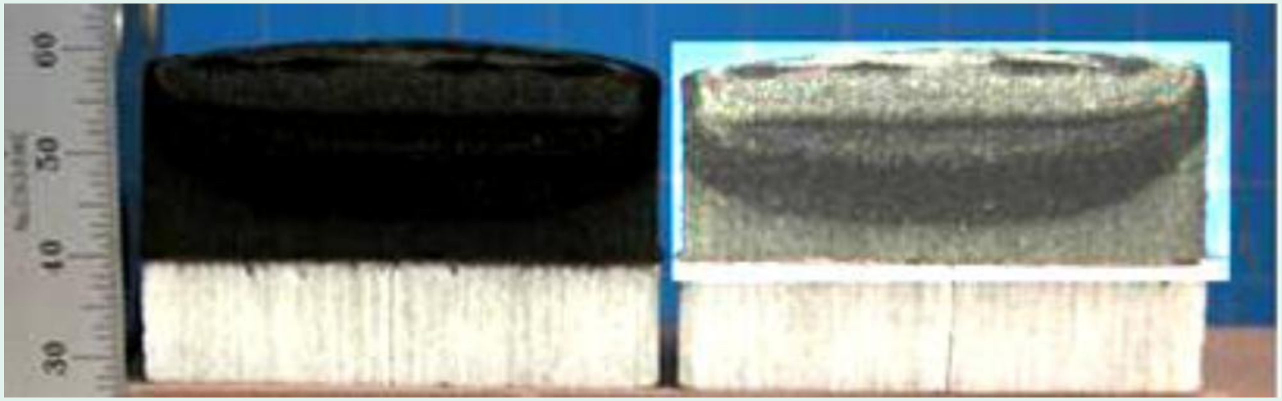
- Maintain structural integrity
- Surviving virgin material
- Recession is relatively even and moderate
- Limited shrinkage, swelling
- No excessive pressure buildup leading to char blowoff
- Stable char: no spall, delamination

Flexible Ablators Char Depths: *Background*

Char and pyrolysis depth measurement from digital cross-section photographs

- Simple, efficient, low cost approach
- Black on black materials pose a challenge for identifying char region
- Many image processing tools available: pixel counting, color tweaking

Post Test Sample



Untouched, raw Image **with Image Processing**

- Other approaches (in depth thermocouples, XRD, FTIR, x-ray tomography…) are more expensive or appropriate for higher TRL

Flexible Ablators Char Depths

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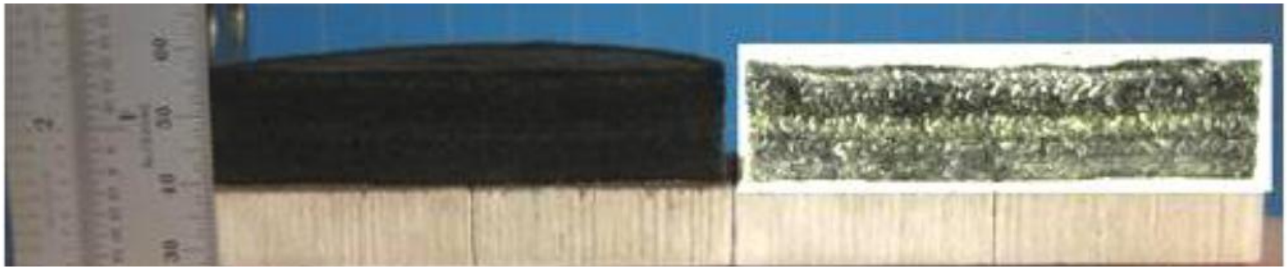
Carbon fiber reinforced materials

Nominal Aero Capture: 115 W/cm^2 for 30 sec

Test Material	Type	Fiber Substrate	Pyrolyzing Component	Characteristics
Morgan-Phenolic FMI-Phenolic	Carbon Phenolic	Carbon Felt	Phenolic	High heat flux/ heat load capabilty

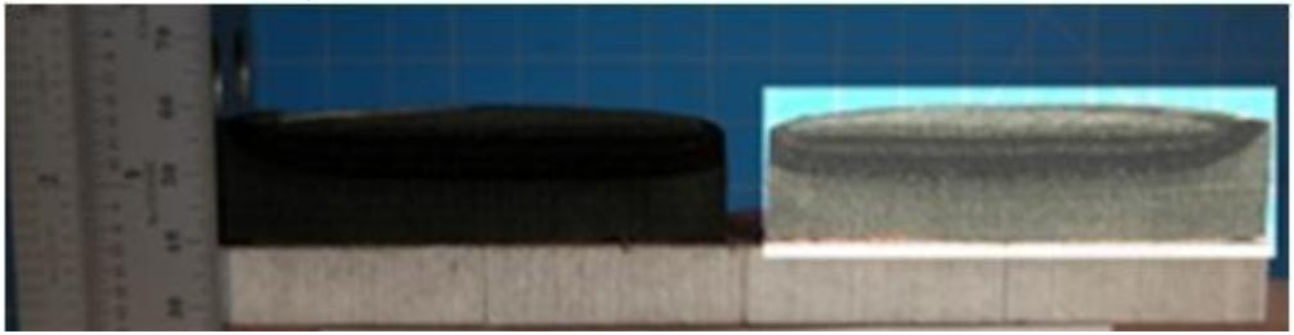


Single Pulse 115 W/cm^2 for 30 seconds

Carbon fiber reinforced materials

Nominal Aero Capture: 115 W/cm^2 for 30 sec

Test Material	Type	Fiber Substrate	Pyrolyzing Component	Characteristics
Morgan-S-T	Carbon Felt Silicone	Carbon Felt	Silicone resin	Oxidation protection
FMI-S-T				
FMI-S-B				
FMI-S-T-A1				
FMI-S-T-A2				
BFFAB Carbon Felt-silicone				

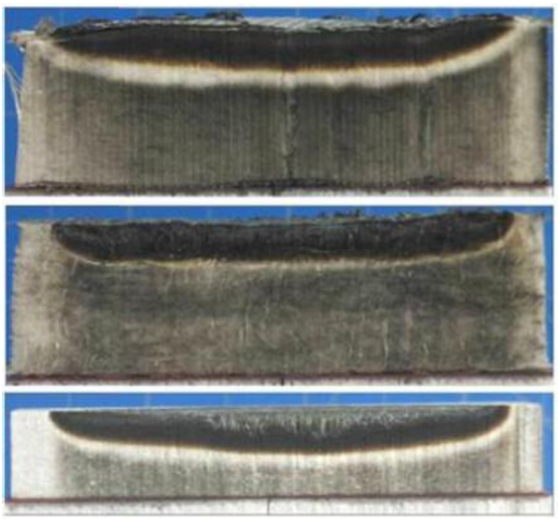


Single Pulse 115 W/cm^2 for 30 seconds

Glass fiber reinforced materials

Nominal Aero Capture: 115 W/cm^2 for 30 sec **Entry**: 30 W/cm^2 for 100 sec

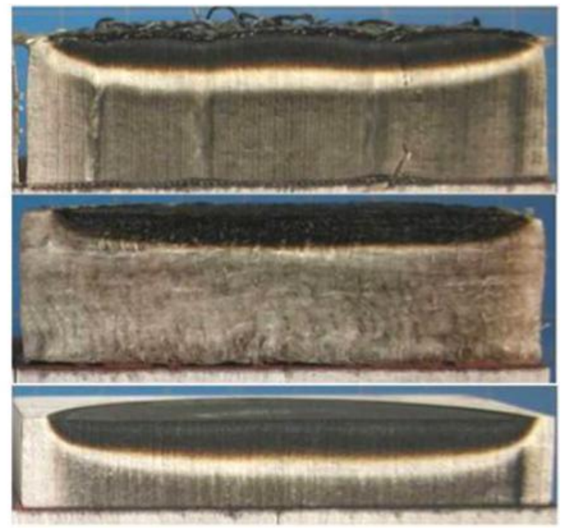
Test Material	Type	Fiber Substrate	Pyrolyzing Component	Characteristics
AFRSI-S-B	Glass Fiber w/ Silicone	Silica or Glass Fiber Felt	Silicone resin	Oxidation resistent, efficient at ~ 100 W/cm^2
Refrasil-S-B				
SIRCA (as Reference)				



AFRSI / Silicone

Refrasil/ Silicone

SIRCA



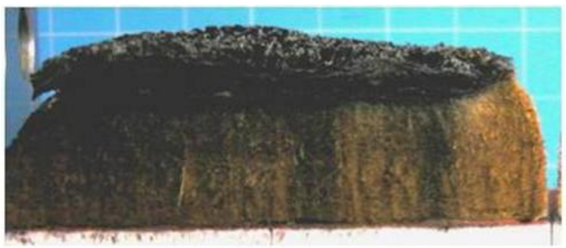
Aerocapture (1 pulse) **Entry (dual pulse)**

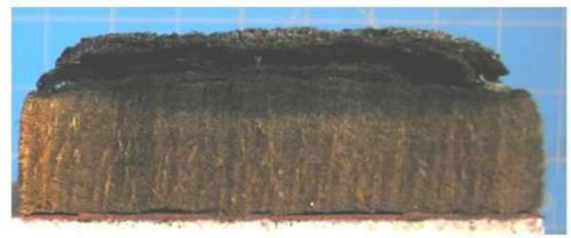
- Limited char growth during Entry (second pulse) simulation
- Silica fiber materials are limited by glass melt to lower heat flux ranges than carbon fiber materials, but gave lower bondline temperatures than carbon

Pyrolyzing fiber reinforced materials

Nominal Aero Capture: 115 W/cm^2 for 30 sec **Entry**: 30 W/cm^2 for 100 sec

Test Material	Type	Fiber Substrate	Pyrolyzing Component	Characteristics
Carbon/PBI-P-T	Flex Polybenzimidazole (PBI) fiber	Carbon or PBI Needled Felt	PBI fiber	Exploit high temp pyrolysis of charring fibers
PBI-S-B				





PBI with Silicone Resin

Carbon fiber/ PBI fiber blend

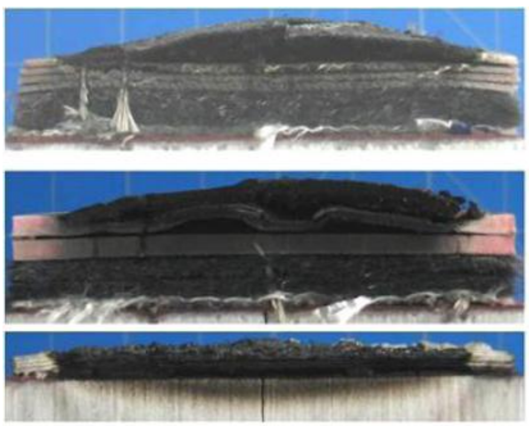
Aerocapture (1 pulse) **Entry (dual pulse)**

- PBI decomposes at a higher temperature than phenolic resin, so these materials exploit pyrolysis and charring of the fibers themselves.
- PBI fibers alone shrink upon charring as expected, but the carbon PBI fiber mixture had limited shrinkage due to the carbon fibers

Layered materials

Nominal Aero Capture: 115 W/cm^2 for 30 sec **Entry**: 30 W/cm^2 for 100 sec

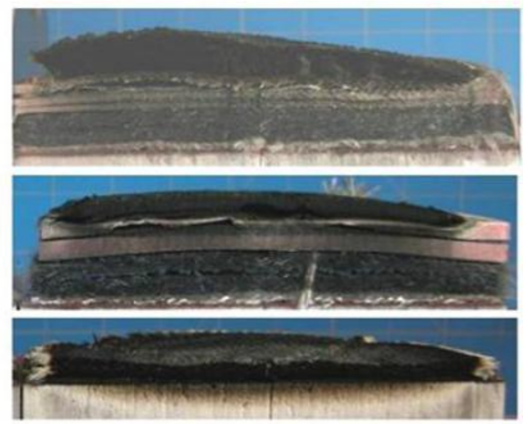
Test Material	Type	Fiber Substrate	Pyrolyzing Component	Characteristics
LM3S Silica Felt Blanket LM2Z Zirconia Felt Blanket BFFA Impregnated Nextel	Layered	Carbon or Silica or Zirconia Felt layers	Silicone resin	Thin, tailorable, flexible. Integrate insulation layer by stictching.



Lockheed LM3S layered Silica felt

Lockheed LM2Z layered Zirconia felt

BFFA Boeing Impregnated Nextel



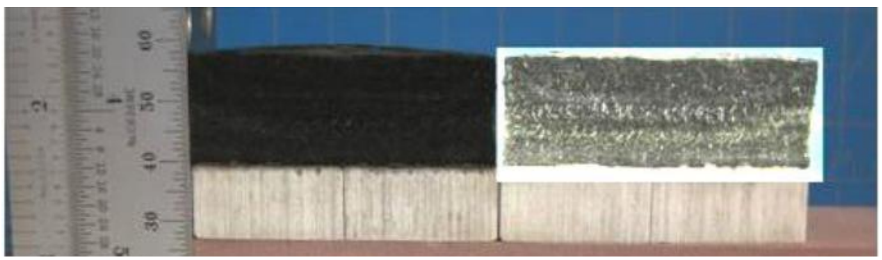
Aerocapture (1 pulse) **Entry (dual pulse)**

- Silica and Zirconia felts were layered, stitched together with ceramic thread
- Nextel fabric loaded with silicone resin
- These materials were relatively thin and gave high bondline temperatures.

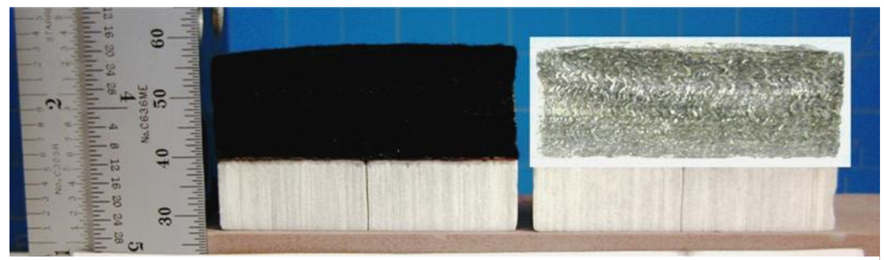
Carbon fiber reinforced materials

High Heating Aero Capture: 450 W/cm^2 for 25 sec **Entry**: 115 W/cm^2 for 50 sec

Test Material	Type	Fiber Substrate	Pyrolyzing Component	Characteristics
Morgan-Phenolic	Carbon Phenolic	Carbon Felt	Phenolic	High heat flux/ heat load capabilty
FMI-Phenolic				



Single Pulse 450 W/cm^2 for 25 seconds



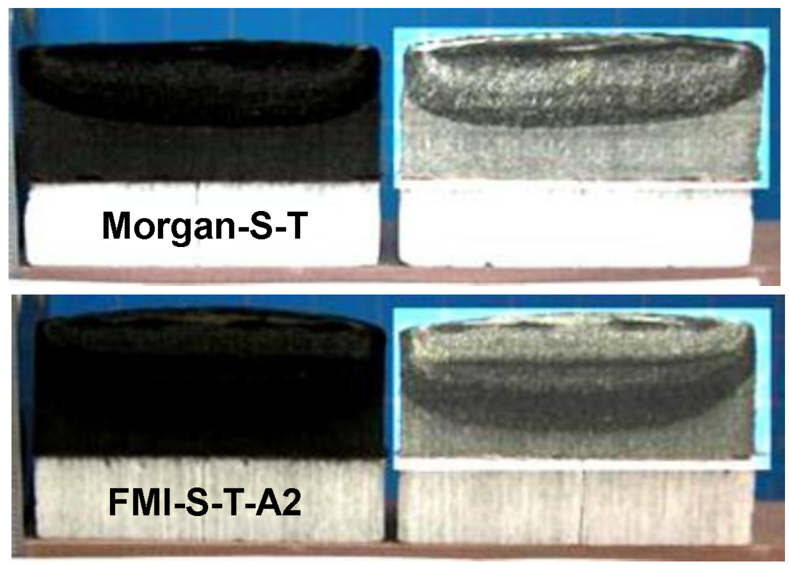
First Pulse: 450 W/cm^2 for 25 seconds
Second Pulse:115 W/cm^2 50 seconds

Carbon fiber reinforced materials

High Heating Aero Capture: 450 W/cm^2 for 25 sec **Entry**: 115 W/cm^2 for 50 sec

Test Material	Type	Fiber Substrate	Pyrolyzing Component	Characteristics
Morgan-S-T	Carbon Felt Silicone	Carbon Felt	Silicone resin	Oxidation protection
FMI-S-T				
FMI-S-B				
FMI-S-T-A1				
FMI-S-T-A2				
BFFAB Carbon Felt-silicone				

- Images are of the materials after both pulses
- Silicone resin offers significant oxidation protection
- High-temperature reactions can produce useful SiC, SiOC in the char layer.
- Photos show after dual pulse



raw, untouched image processed image

First Pulse: Nominal Aerocapture Heating Level

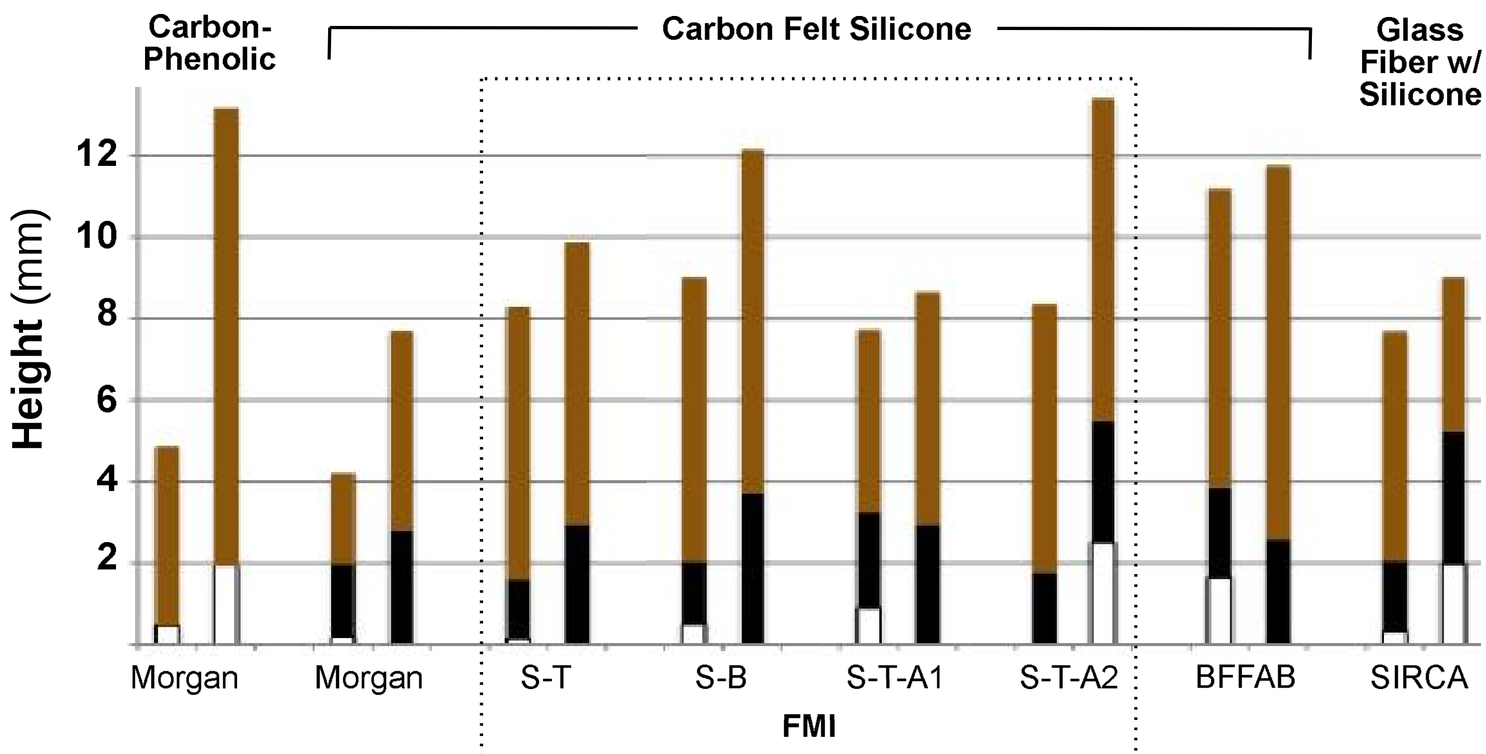
Nominal Heating: CO_2 vs. Fiber lasers

Aero Capture: 115 W/cm^2 for 30 sec

Shorter wavelength NIR Fiber laser resulted in deeper in depth thermal penetration

LEGEND
- Pyrolysis Zone
- Char
- Recession

left bar: CO_2 laser
right bar: Fiber laser

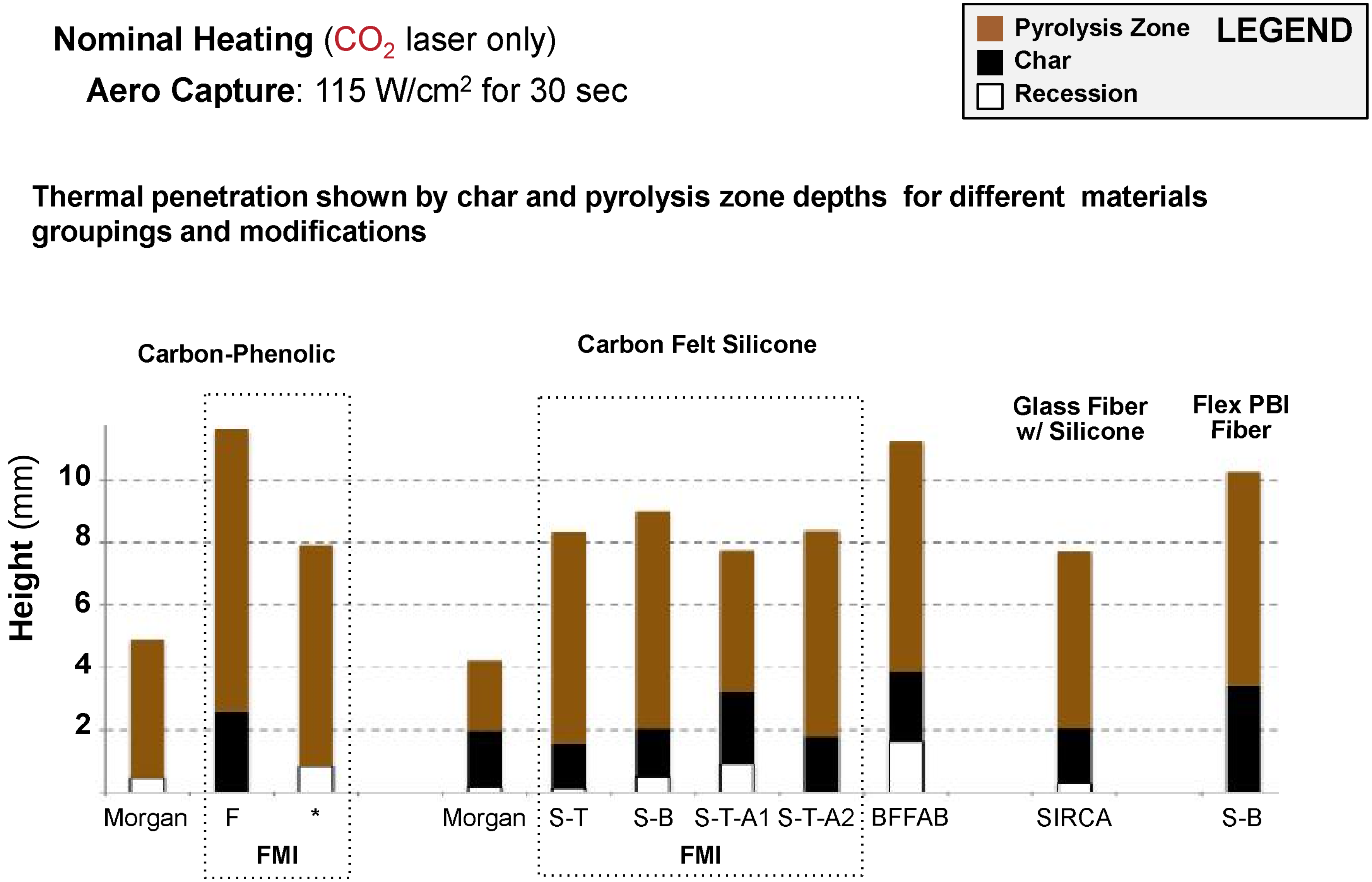


First Pulse: Nominal Aerocapture Heating Level

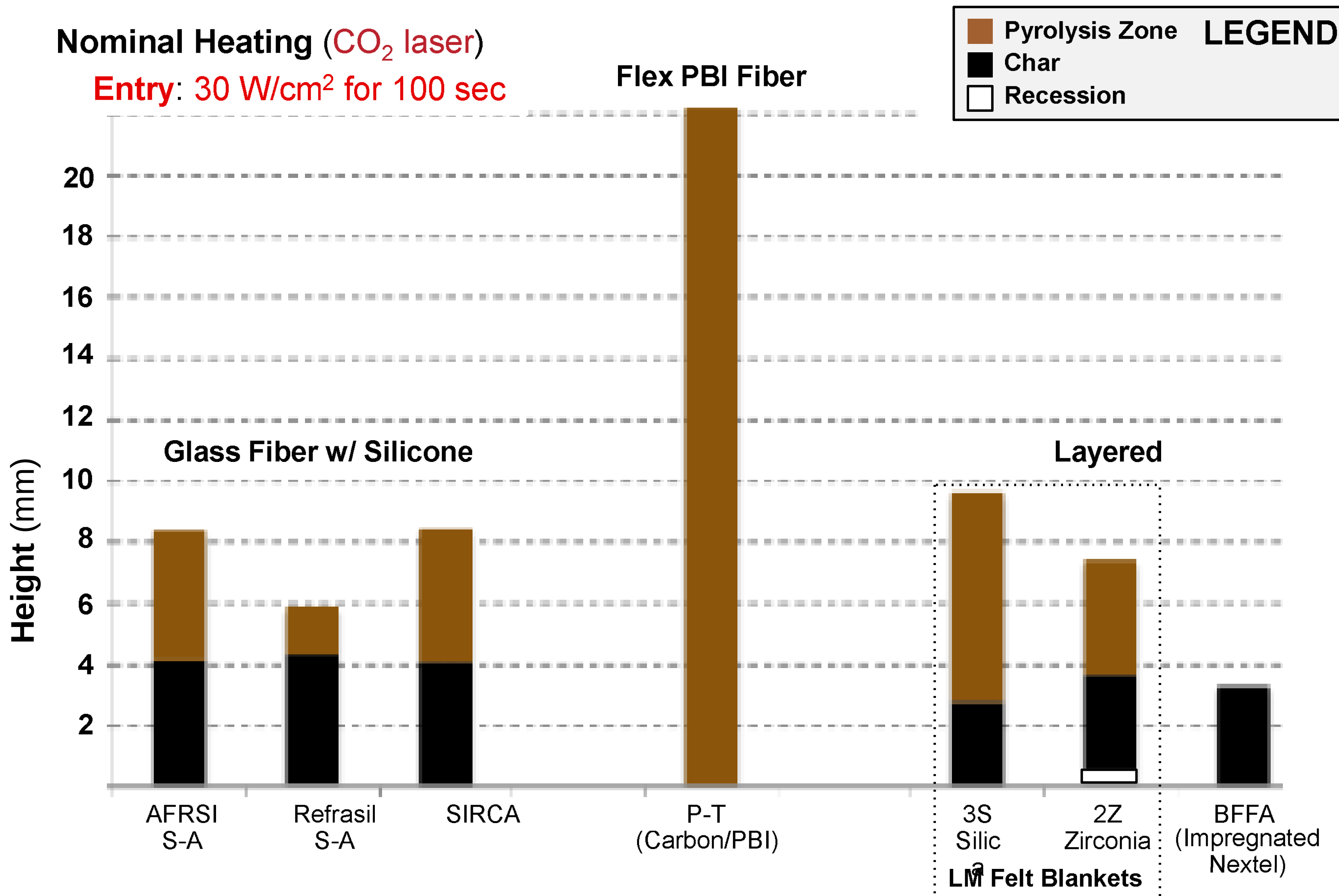
Nominal Heating (CO_2 laser only)

Aero Capture: 115 W/cm^2 for 30 sec

Thermal penetration shown by char and pyrolysis zone depths for different materials groupings and modifications



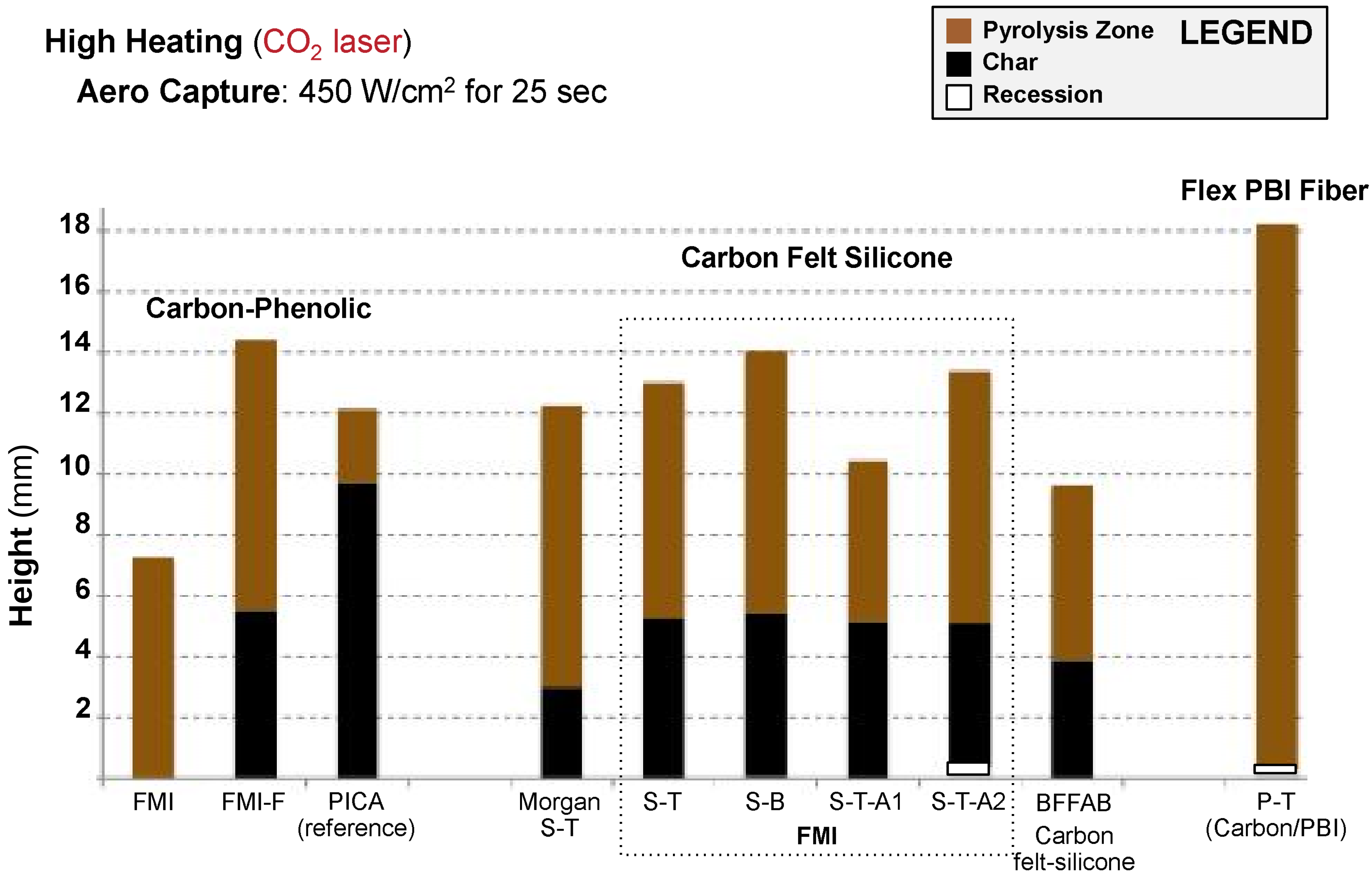
Second Pulse: Nominal Entry Heating



First Pulse: High Option Aerocapture Heating Level

High Heating (CO_2 laser)

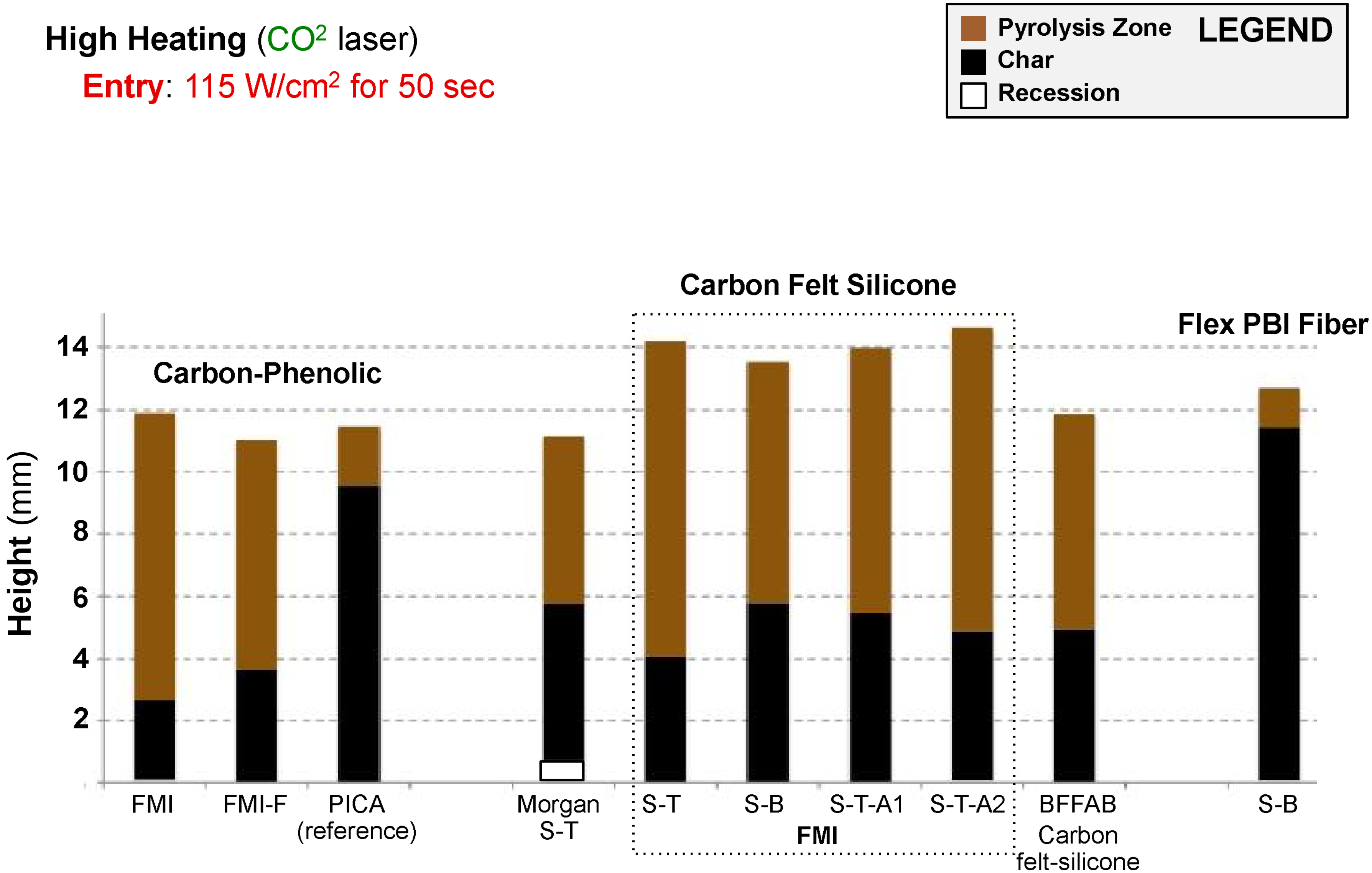
Aero Capture: 450 W/cm^2 for 25 sec



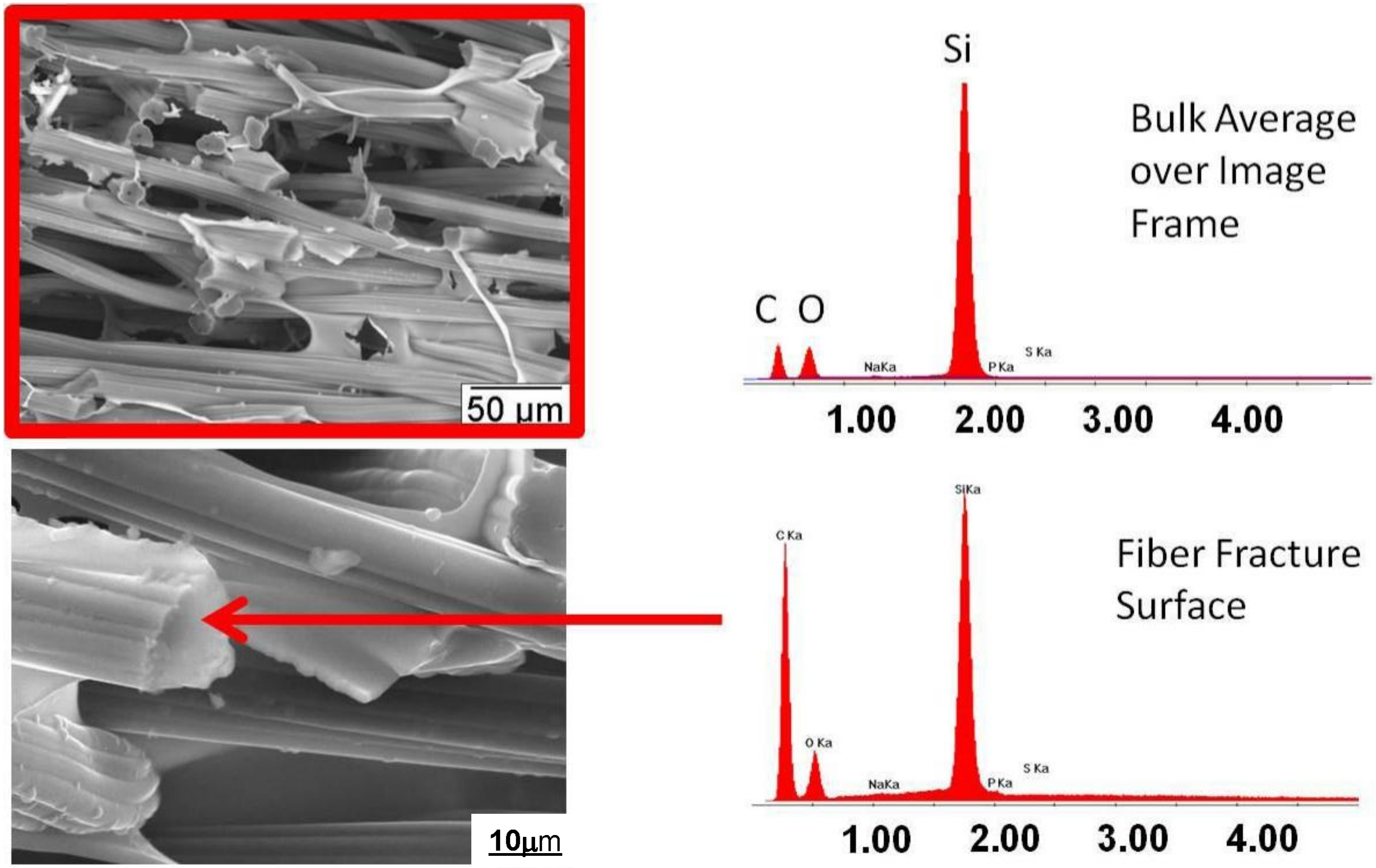
Second Pulse: High Option Entry Heating

High Heating (CO^2 laser)

Entry: 115 W/cm^2 for 50 sec

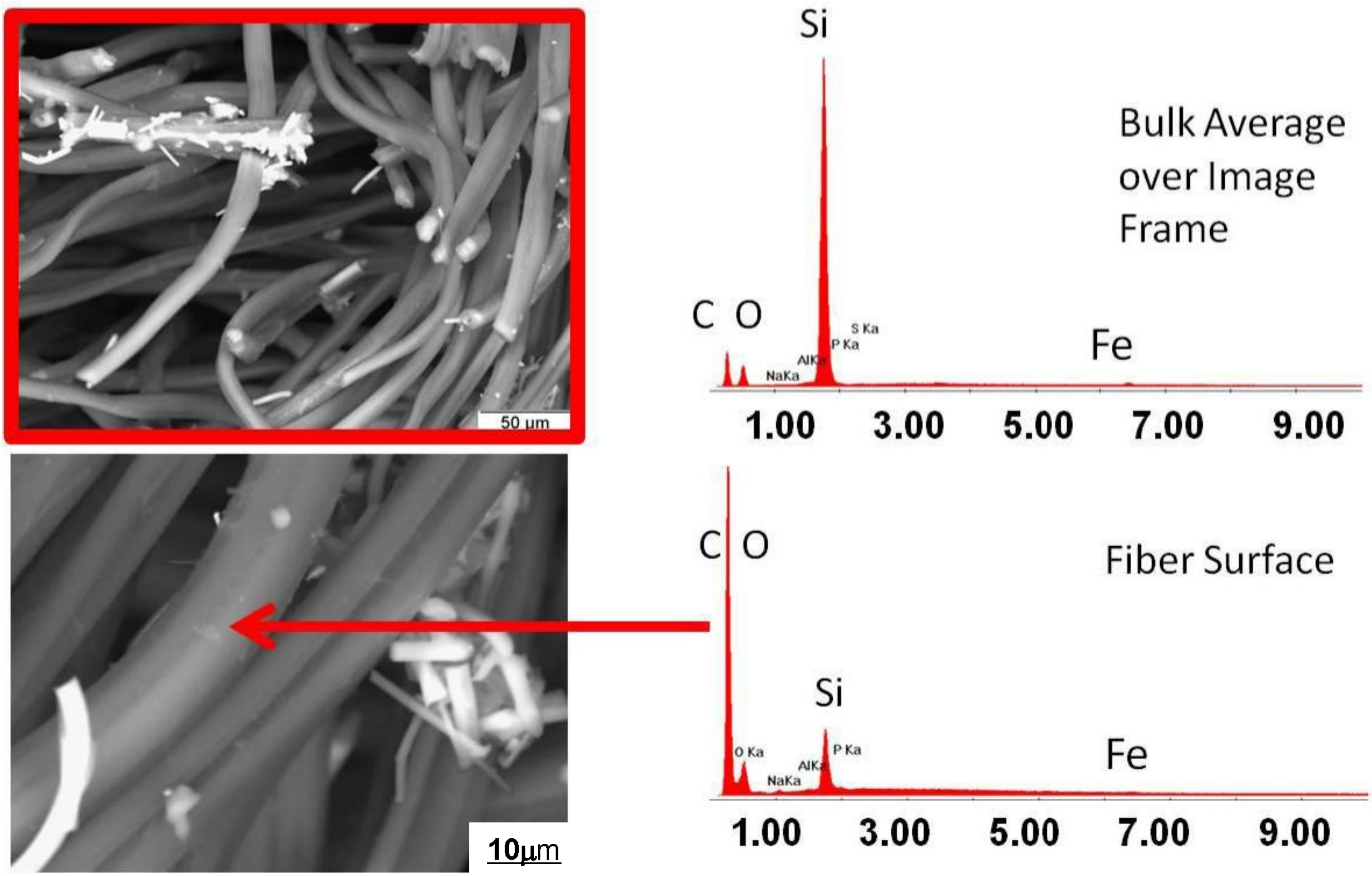


High resolution images :SEM / EDX: Char Layer



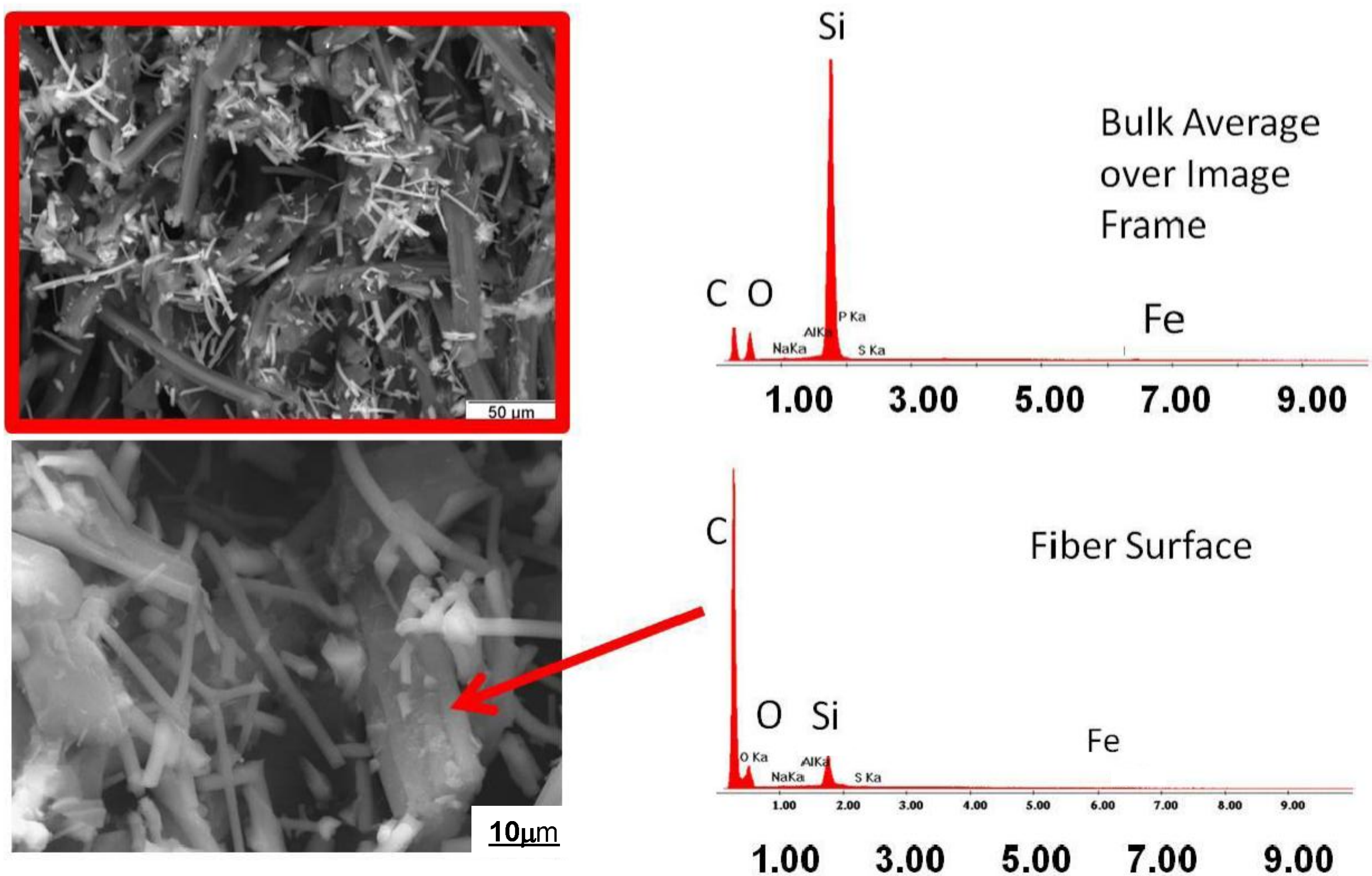
Scanning Electron Micrograph (SEM) shows the char zone fibers with a coating, flaking off. Carbon, Oxygen Silicon measureable with EDS – more carbon at the fiber fracture surface. (SEM with backscattered electron detector (*BSD*) with EDAX energy dispersive X-ray analysis (*EDS*) system) VQ

SEM / EDS: Pyrolysis Zone



- **Carbon, Oxygen Silicon measureable at interface with EDS – mainly Carbon on the fiber surface, dominated by Silicon in the resin**

SEM / EDS: Virgin Zone



- **Carbon, Oxygen Silicon measureable at interface with EDX – mainly Carbon on the fiber surface, dominated by Silicon in the resin**

Flexible Ablators Char Depths

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Key results

- Thicker char depths resulted from laser testing at 1.07 microns versus 10.6 microns, indicating some level of transmission and in-depth absorption at the shorter NIR wavelength.
- This different response - even for carbon fiber materials - impacts
  - material response to shock layer spectral radiation
  - in-depth response to thermal radiation at high temperature gradients.
- Char and pyrolysis zones can be used to plunder test models to extract more information: e.g. distinguish effects of additives, solvents
- Char depths were moderate for the second pulse exposure used to simulate entry after aerocapture, (consistent with bondline temperature and mass loss measurements) that these materials have good potential for dual-pulse aerocapture and entry missions.
- Carbon-fiber reinforced materials had limited char growth under the high heat flux option – so flexible / deployable materials can substitute for rigid TPS materials in that regime. Can we go higher?

Conclusions and future work

- The EDL laser tests were one component of screen tests used to downselect materials to further develop and characterize.
- Char and pyrolysis zone depths give physical evidence of peak temperature reached in depth: The pyrolyzing material acts as a temperature indicator within its characteristic thermal decomposition range. Char depth comparisons are useful:
  - to verify material response models when high fidelity models are available – need to validate density of observed char zone.
  - especially useful to efficiently compare large arrays of novel materials, and/or process and composition modifications.
  - complements quick-turnaround screening using bondline temperatures and mass loss measurements - before characterization or in-depth instrumentation.
- **Future work:**
  - Compare char depth trends from laser tests to arcjet and other tests. (Note that test conditions are not identical. (Planned)
  - Characterize char and pyrolysis zones with SEM, EDS, FTIR (In progress)
  - Confirm measurements of char density vs. char zones from images for use in high-fidelity thermal model verification. (In progress)
  - Test high-performing flexible ablators to find upper heat flux / heat load limits.

Acknowledgements of Support and Teamwork

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Thanks to:

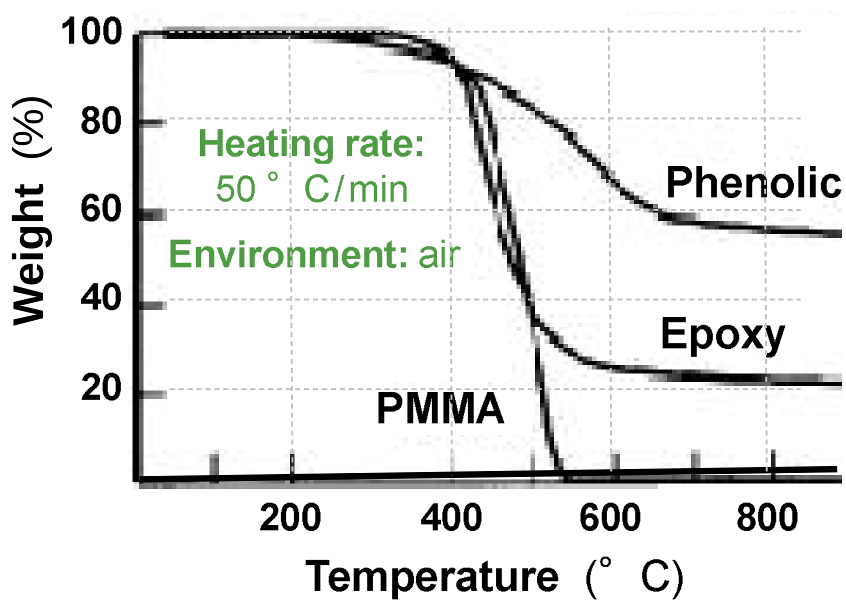
- The NASA Ames EDL/TDP Flexible TPS element lead, Robin Beck
- EDL/TDP materials development and evaluation team (Parul Agrawal, Jim Arnold, Al Covington, Matt Gasch, Howard Goldstein, Bernie Laub, Joe Mach, Frank Milos).
- Dave Kinney and Kathy McGuire for simulation results.
- Joe Conley for improving the presentation.
- John Bagford and Dan Seibert from the LHMEL facility.

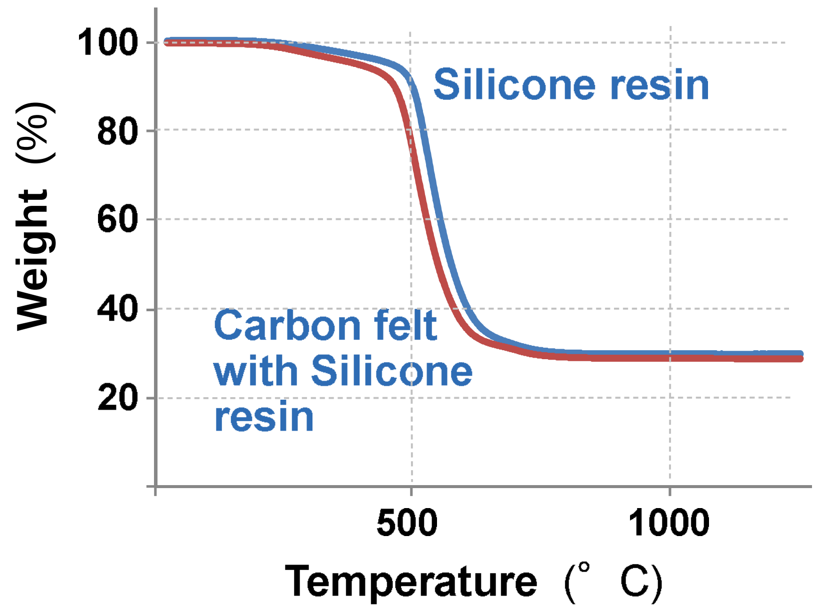
Flexible Ablators Char Depths: *Background*

Ablator Modeling

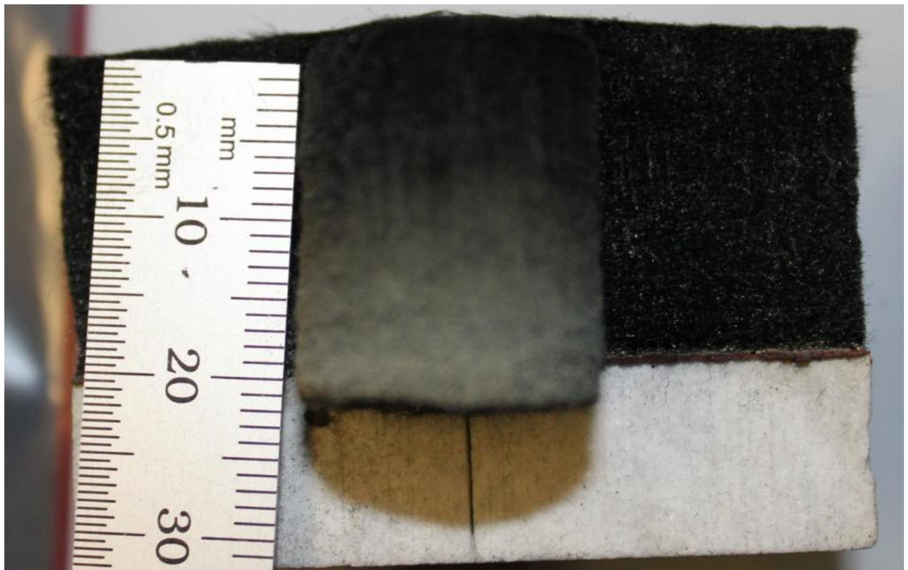
Thermogravimetric analysis (TGA) **shows that**

- pyrolyzing resin reaction kinetics limit the temp range of decomposition
- carbon felt has minimal effect on the decomposition process



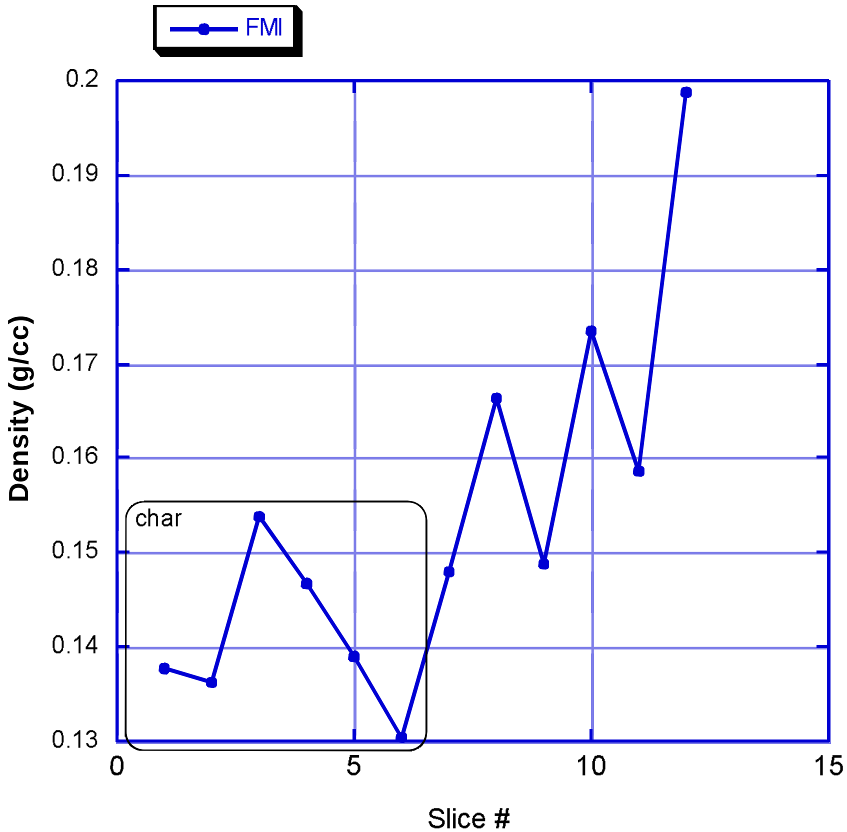


Char density profile:Backup

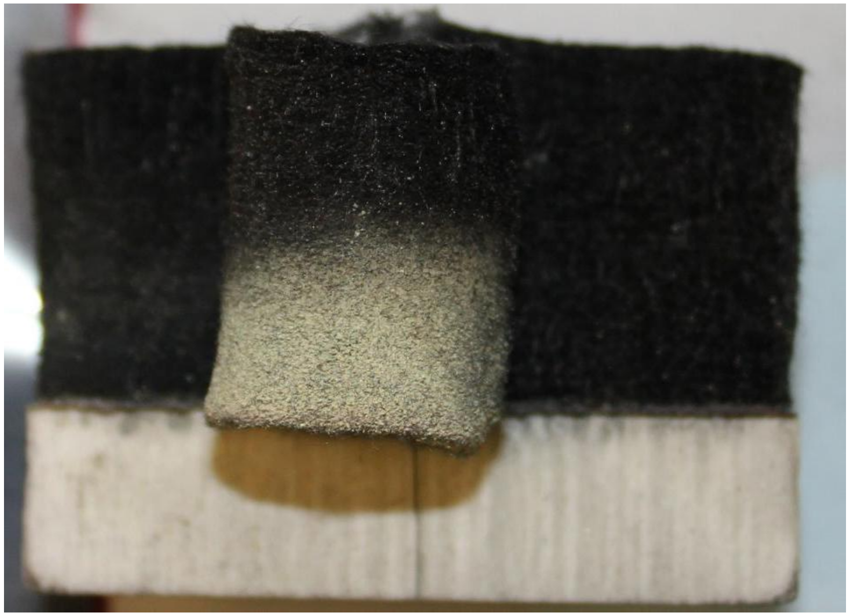


Carbon Phenolic 1 with a silicone seal coat

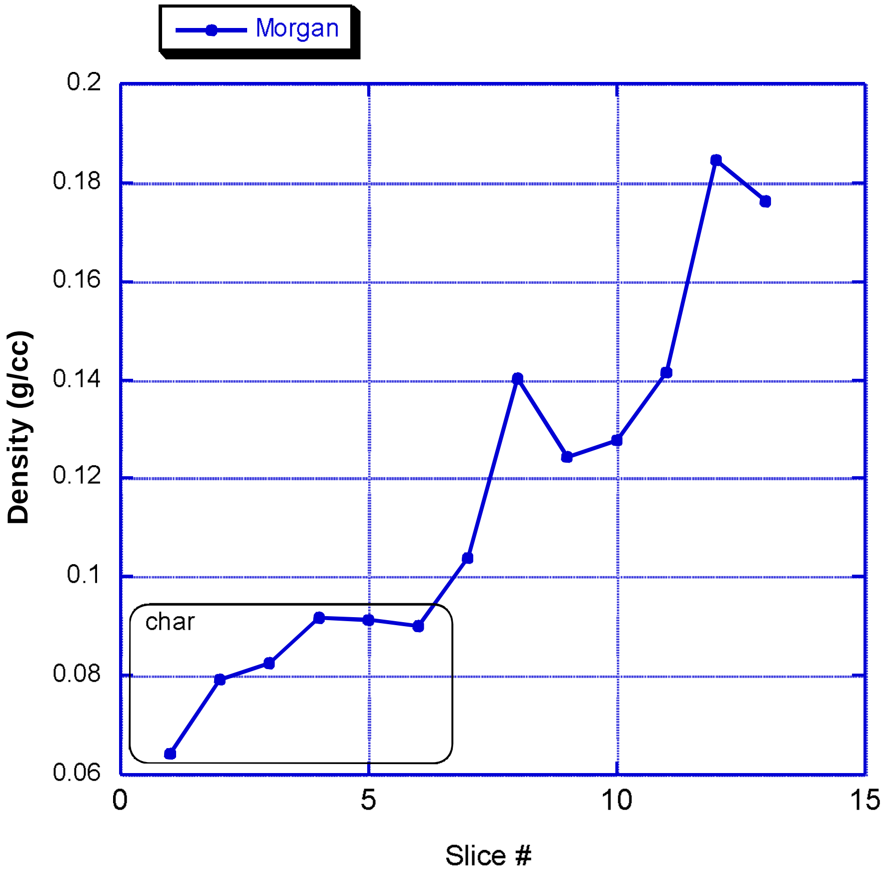
- Each slice corresponds to ~ 1mm thickness
- Felt variability contributes to scatter in density profile
- Virgin density is ~ 0.23g/cc
- **Char density profile: Must correlate visual char depths with density for thermal models**



Char density profile data



Carbon Phenolic 2 with a silicone seal coat



- **Each slice corresponds to ~ 1mm thickness**
- **Virgin density is ~ 0.19g/cc**

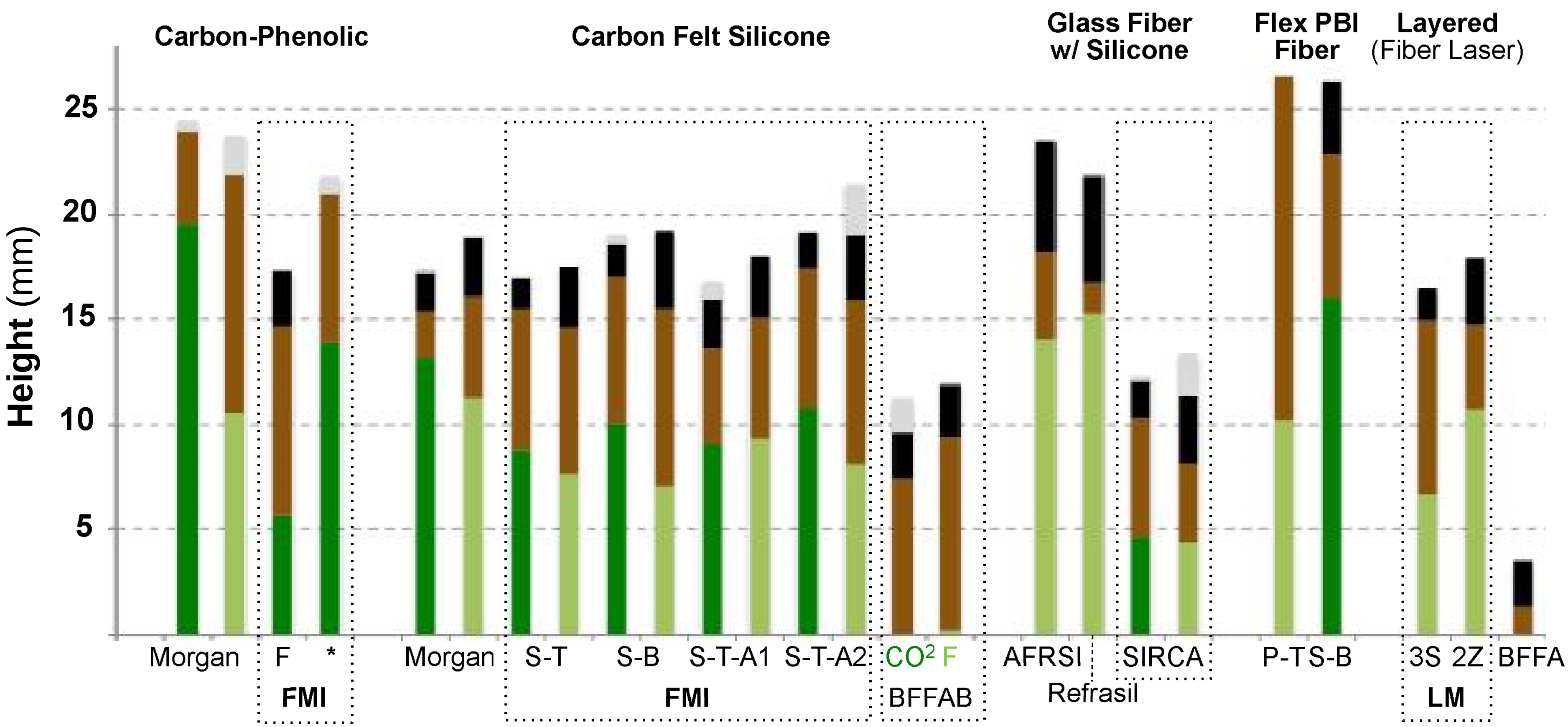
- **Char density profile:**
- **Must correlate visual char depths with density for thermal models**

First Pulse: Nominal Aerocapture Heating Level

Nominal Heating (CO^2 and Fiber lasers)

Aero Capture: 115 W/cm^2 for 30 sec

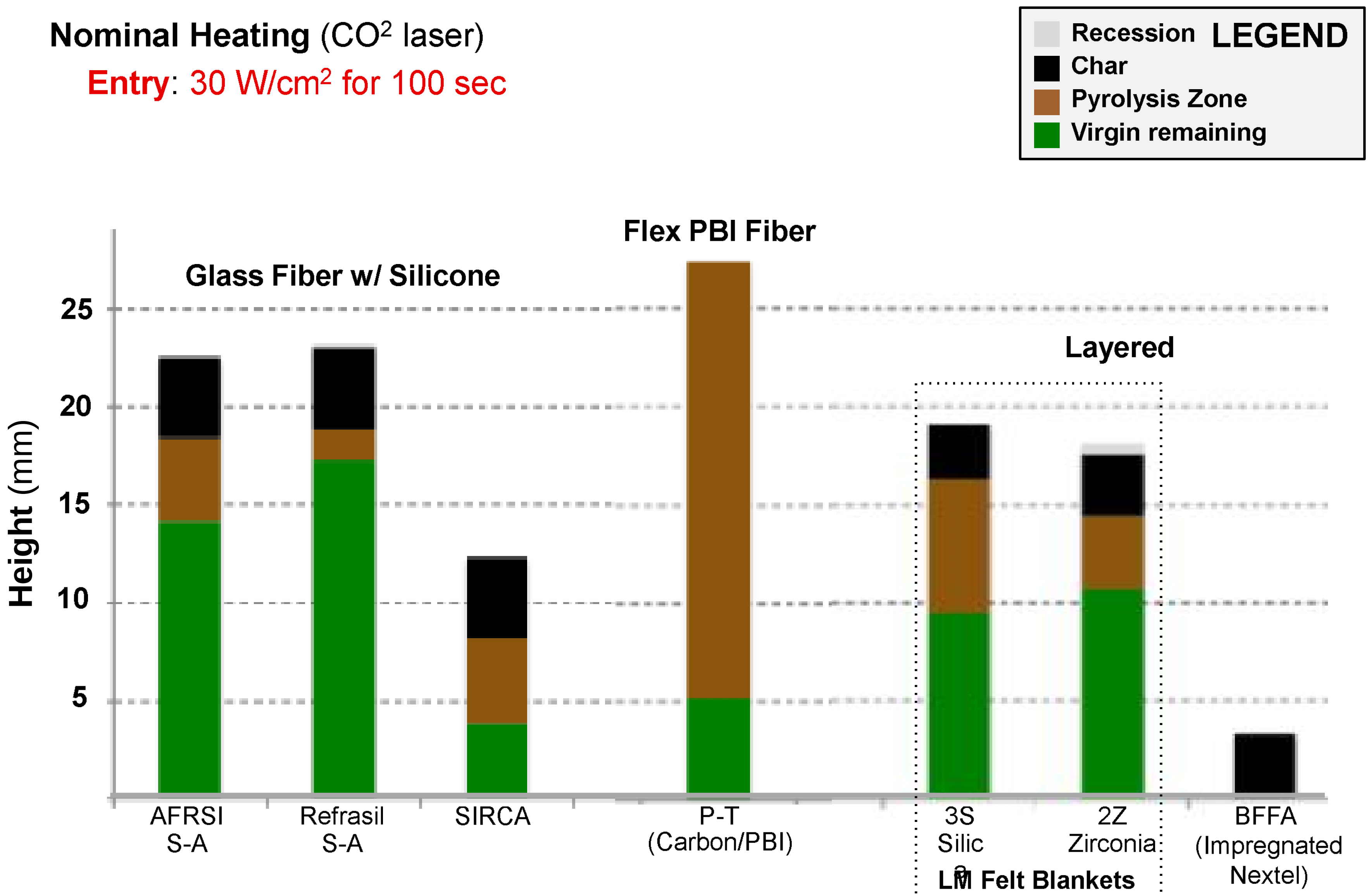
LEGEND
- **Recession**
- **Char**
- **Pyrolysis Zone**
- **Virgin remaining** (Fiber laser)
- **Virgin remaining** (CO^2 laser)



Second Pulse: Nominal Entry Heating

Nominal Heating (CO2 laser)

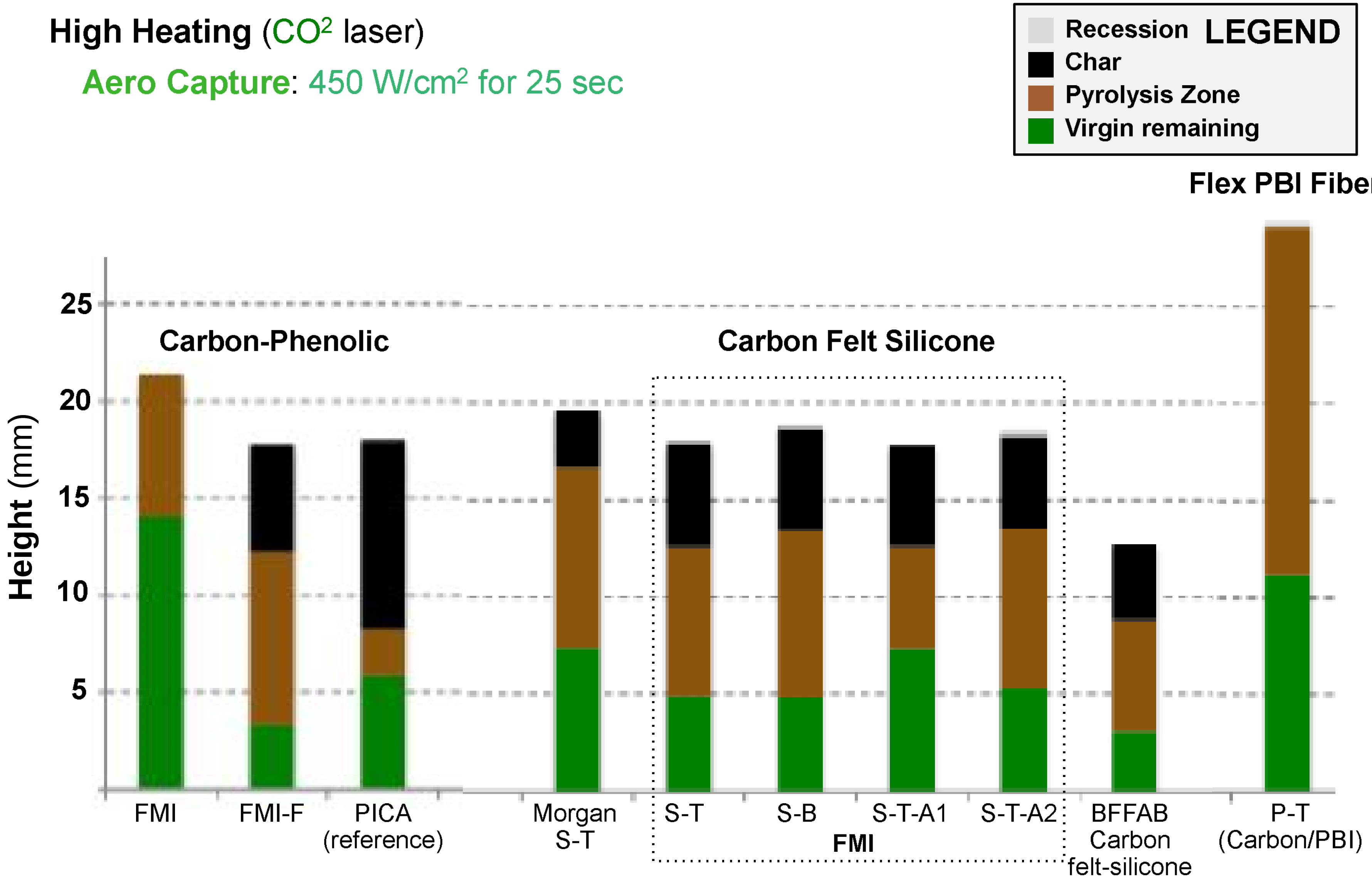
Entry: 30 W/cm^2 for 100 sec



First Pulse: High Option Aerocapture Heating Level

High Heating (CO2 laser)

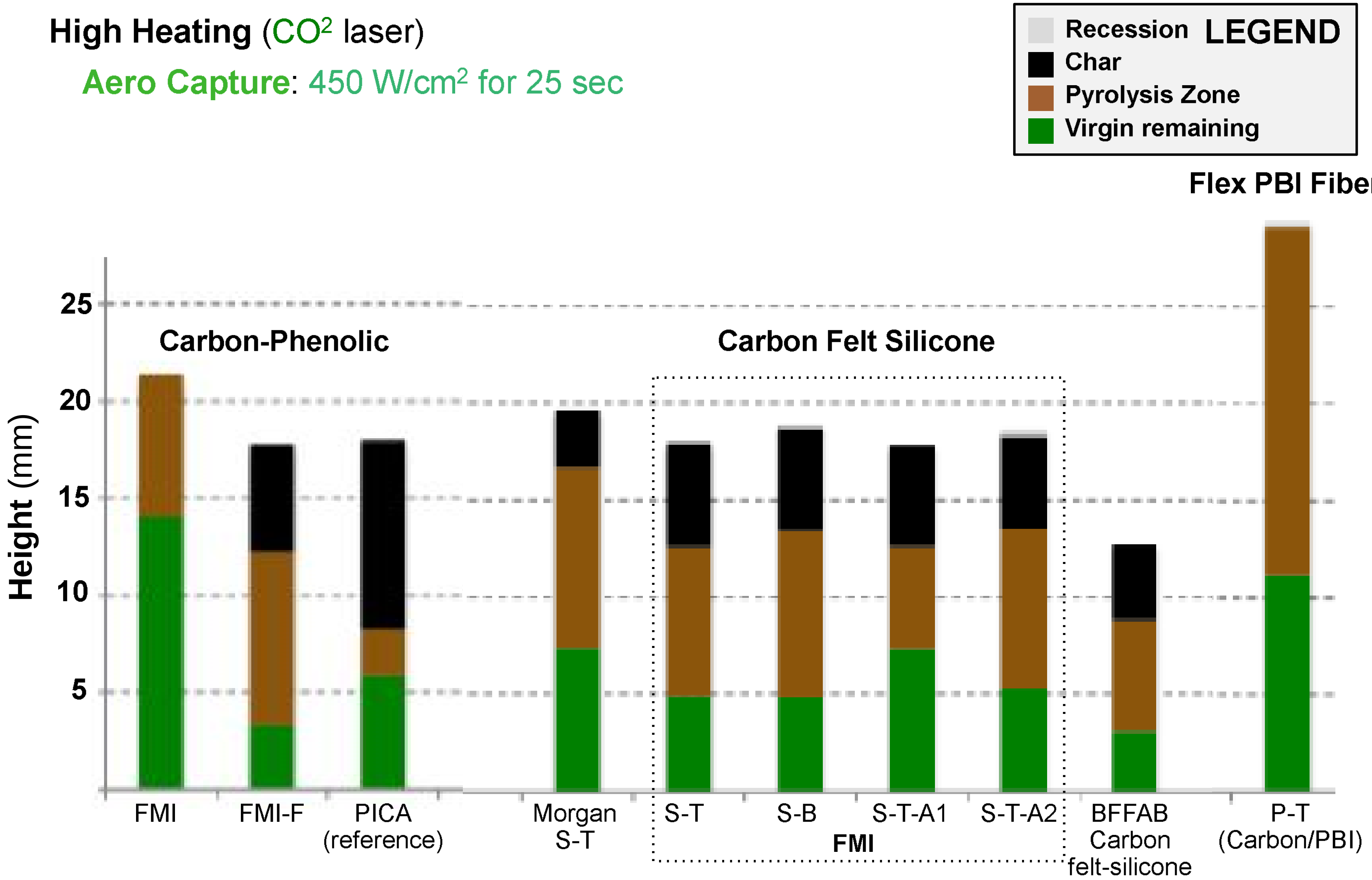
Aero Capture: 450 W/cm2 for 25 sec



Second Pulse: High Option Entry Heating

High Heating (CO^2 laser)

Aero Capture: 450 W/cm² for 25 sec



First Pulse: Nominal Aerocapture Heating Level

Nominal Heating (CO_2 and Fiber lasers)

Aero Capture: 115 W/cm^2 for 30 sec

Combined char and pyrolysis for materials, modifications, 2 lasers for the nominal test condition