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High Temperature Carbonization (HTC)

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Overview

Timeline

- Project Start: 11/17/20*
- Project End: 9/30/22
- Progress: 20%

* Due to internal delays

Budget

Overall budget:

- FY21 – FY11: $3.5M

Detail:

- FY21: $2.0M
- FY22: $1.5M

Barriers

- Barriers addressed
  - Cost: A goal of this project is to reduce energy consumption in the carbon fiber conversion process and therefore total carbon fiber cost.
  - Inadequate supply base: Another goal of this project is to reduce the required processing time for carbonization and therefore increase overall throughput.

2017 U.S. DRIVE MTT Roadmap Report, section 4

Partners

- Project lead: ORNL
- Partner: 4X Technologies

(formerly RMX Technologies)

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Relevance

- <u>Project title:</u>
  High Temperature Carbonization (HTC):
  - Relies on dielectric heating
  - Faster and more efficient that conventional
  - At atmospheric pressure
- <u>Project Goals:</u>
  - Reduce unit energy consumption of HTC stage (kWh/kg) by ca. 20% (ca. 5% of the cost reduction on the carbon fiber (CF) overall manufacturing process)
  - Produce equal or better quality carbon fiber
  - Scale the technology to a nameplate capacity up to one annual metric ton by project end date

Milestones

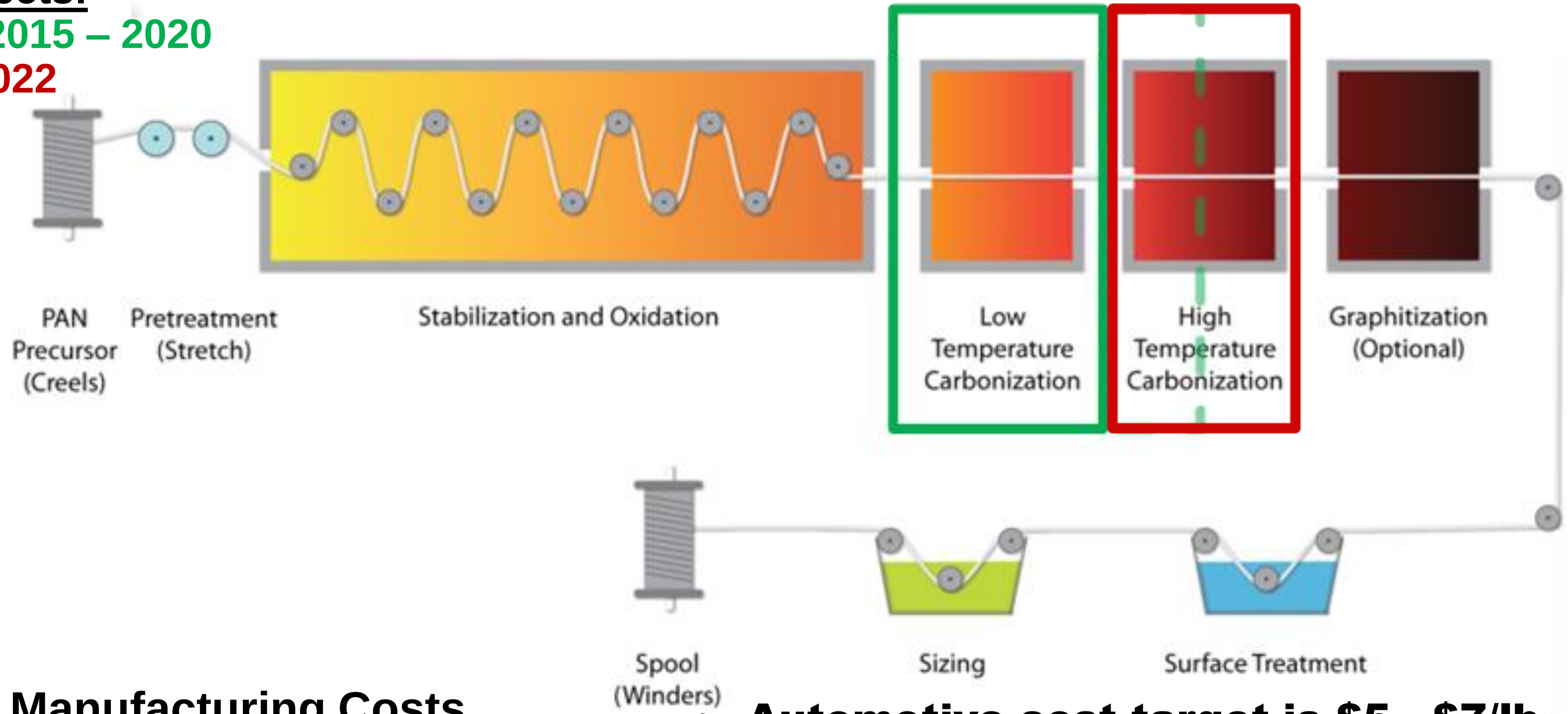
Date	Milestone FY2021	Status
Dec. 30, 2020	**M1:** Data and design Evaluation between two applicator configurations. Down selection of one of them to go to HTC.	**Completed: Feb. 26, 2021**
March 30, 2021	**M2:** design with CEM modeling completed. Beginning of procurement.	**Not completed**
June 30, 2021	**M3:** Commissioning of HTC. Stable operation (15min).	**N/A**
Sept. 30, 2021	**M4 (Go/No-Go):** CF production (1.6g/cc, 300 ksi tensile strength, and 25 Msi modulus).	**N/A**

Date	Milestone FY2022	Status
Dec. 30, 2021	**M5:** Process 4 tows of 24k filaments ea. CF production (Density = 1.7 g/cc, tensile strength = 400ksi, modulus = 25Msi, residence time = 200s)	**N/A**
March 30, 2022	**M6:** Process 4 tows of 24k filaments ea. CF production (Density = 1.7 g/cc, tensile strength = 500ksi, modulus = 27Msi, residence time = 200s)	**N/A**
June 30, 2022	**M7:** Process 4 tows of 24k filaments ea. CF production (Density = 1.7 g/cc, tensile strength = 550ksi, modulus = 29Msi, residence time = 160s)	**N/A**
Aug. 31, 2022	**M8:** Process optimization. CF production (tensile strength = 550ksi, modulus = 29Msi). Energy balance acquisition.	**N/A**
Aug. 31, 2022	**M9:** Demonstrate HTC at least 5% cost savings versus conventional carbonization.	**N/A**
Sept. 30, 2022	**M10:** Final report with discussion about further technology implementation/scale up.	**N/A**

<u>NOTE:</u> The HTC project began with almost two months of delay (administrative reasons). This calendar does not take this consideration into account.

Approach (conventional PAN processing)

Two VTO projects:
LTC (CPEC): 2015 – 2020
HTC: 2021 - 2022



Major Manufacturing Costs	
Precursor	ca. 43%
Oxidative stabilization	18%
Carbonization	13%
Graphitization	15%
Other	11%

- **Automotive cost target is \$5 - \$7/lb**
- **Tensile property requirements are 250 ksi, 25 Msi, 1% ultimate strain**
- **ORNL is developing major technological breakthroughs for major cost elements**

LTC: low temperature carbonization
CPEC: close proximity electromagnetic carbonization (name of the design)

Approach (CPEC)

- Conventional furnaces consume significant energy heating large volumes of inert gas surrounding the fiber.
- If thermal energy could be directly coupled from an energy source to the fiber, tremendous energy savings could be realized.
- This project uses electromagnetic coupling to directly heat the fiber
- Dielectric/Maxwell-Wagner heating mechanisms are utilized

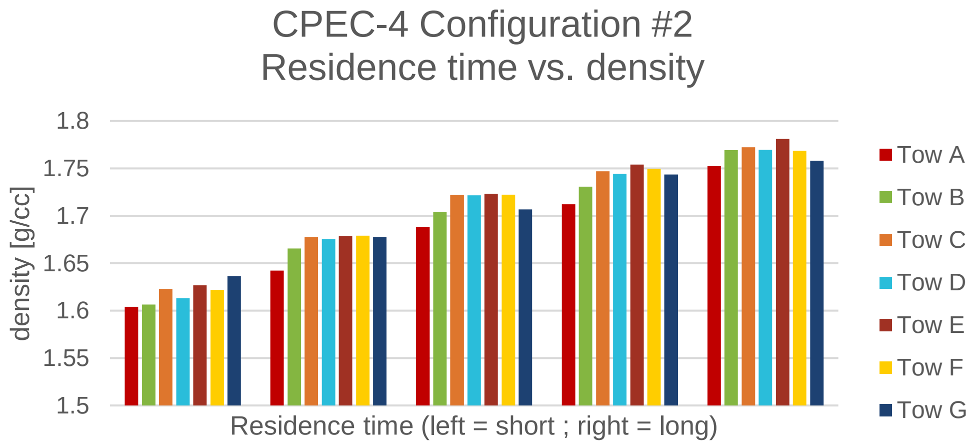
$$P_v = 2\pi f|E|^2\varepsilon_0\varepsilon' tan\delta$$

  - P_v volumetric power transferred to the material.
  - ε' is the relative dielectric constant.
  - ε_0 is permittivity of free space, 8.85418782 x 10^{-12} F/m.
  - $|E|$ is the magnitude of the local electric field intensity (V/m).
  - $tan\delta$ is the loss tangent of the material.
  - f is the operational frequency.

Progress — Concept validation

- Evaluation of LTC system pushed to the maximum of its potential (extreme condition with limited modification)

Material density

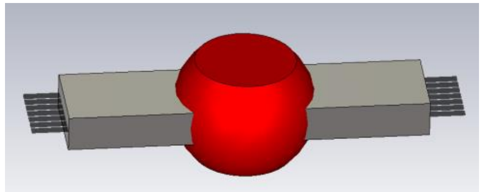


- Existing setup (designed for LTC) used for performance evaluation
- 7 tows of oxidized PAN (OPF, 50k filaments) are processed simultaneously
- 5 different residence times
- Full carbonization attempt in one stage (perf. vs. residence time)
- Power is set to its maximum (constant)

Based on density:

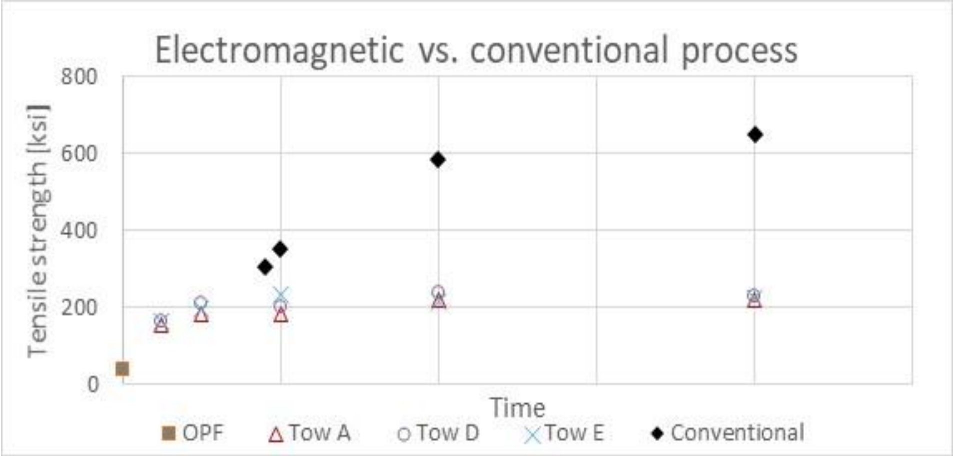
→ Very short process: with ~1.62 g/cc, LTC level only is achieved

→ Very long process: with ~1.76 g/cc, HTC level is achieved



Schematic representation of LTC system (CPEC) with 7 tows

Mechanical properties



- 3 tows, A, D, E (among the 7) are tested at each residence time
- Results are compared with a modified conventional process set to mimic a one stage process
- Temperature distribution between CPEC-4 and the modified conventional setup is the main difference (potentially higher temperature in the conventional process)
- Short residence time could not be achieved using the conventional line (technical reasons)

Based on Mechanical:

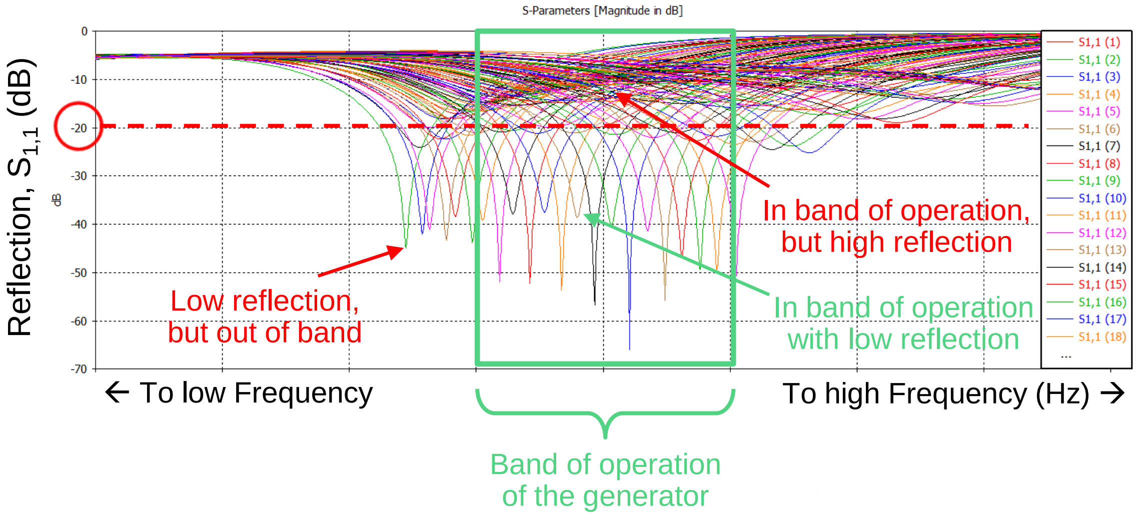
→ Very short process: with ~150 – 200 ksi, LTC level is achieved

→ Very long process: with ~200 ksi, HTC level is NOT achieved (500 – 600 ksi is expected)

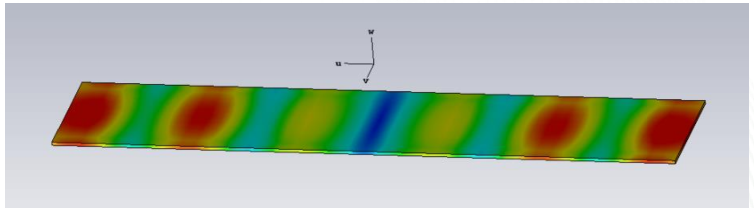
→ TWO-STAGE PROCESS REQUIRED

Progress – CEM

- <u>CEM is used to design the future setup:</u>
  - Design methodology:
    - Energy transfer is frequency dependent: low reflection bands must be in the band of operation of the generator (green window)
    - Reflection coefficient $S_{1,1}$ is minimized = good energy absorption and temperature elevation (ideally if $S_{1,1} < -20\text{dB}$ = good tuning)
    - Pattern of radiation must be appropriate
    - Design of applicator must be realistic
      - Materials are selected for:
        - » EM properties
        - » Thermal properties
        - » Mechanical properties
      - Parts must be manufacturable and assemblable
      - System must be safe
  - Examples:
    - Reflection study for a given design (parameter sweep)



    - Pattern of radiation on a 200k filament tow



A band of 200k filaments (4 x 50k filaments) propagating along the "u" axes. The gradient of color shows a typical pattern of radiation.

As of early May 2021, important progress is made, but all the criteria are still not satisfied simultaneously

Progress - Ancillary activities

- Procurement:
  - Procurement of some parts with long lead time already begun
    - Transmission line (partial)
    - Power measurement system
    - Fiber handling system in process of upgrade (design completed, quotation in progress)
- Conventional conversion equipment (for baseline and comparative study)
  - 2 furnaces refurbished and installed
  - Fiber handling system designed/fabricated/received
  - Some tasks remains to be done:
    - Nitrogen line
    - Exhaust system

- Lab space management
  - Former LTC system modified, upgraded (monitoring), etc.
  - Space for the future setup established (with all utilities and support systems)
- Feedstock material supply
  - 2 types feedstock material with different packaging were acquired (design of the fiber presentation accomplished for both forms of material)
- Administrative tasks
  - Delay in setting the contract (ORNL changed its contract procedure): beginning: November 17, 2021

Response to Previous Year Reviewer's Comments

- Previous year scoring (AMR 2020):
  - Relevant to current DOE objectives: **N/A**
  - Resource allocation: **N/A**

→ This is the first year for this project: no previous review

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Collaboration and coordination

ORNL is performing this project in collaboration with:

4XTechnologies — Joint development. Equipment construction and experimental work performed at this site.

4XTechnologies is a dynamic startup located in Knoxville, TN, with a core focus on plasma science and engineering and experience in fiber treatment/conversion and environmental applications.

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Remaining Challenges and Barriers

- Based on past experience and the knowledge acquired with the LTC project, anticipated barriers are:
  - Need for a better applicator thermal insulation
  - Need for a better selection of dielectric materials
  - Possible insufficient power supply capacity. Evaluation in progress. Backup solution already available.
  - Further development of CEM model that encompasses thermal property changes
  - Hardware specification rated for higher rate of energy transfer

CEM modeling: computational electromagnetic modeling

Proposed Research

- FY21-22: High temperature carbonization using EM (HTC)

  – FY21: (design, construction, and testing)
    - **Complete the CEM** modeling study and identify an acceptable design
    - **Create a CAD model** (mechanical drawing)
    - **Machine and assemble** the applicator parts
    - Commission, **operate**, modify/upgrade (based on need) the system and reach the subsequent milestones related to the production of CF material

  – FY22: (fiber production)
    - Complete the **overall cycle** of carbonization using electromagnetic energy
    - Evaluate the **energy benefits**
    - This complete cycle will require **two discreet processing units in series** (LTC + HTC). This approach needs to be demonstrated at the end of this project.

** Any proposed future work is subject to change based on funding levels*

Summary

- HTC project initiated on Nov 17, 2020
- Evaluation of the potential of the overall limits and thresholds of the LTC technology:
  - Required fiber density can be achieved, but the corresponding mechanical requirements are not satisfied
  - This clarifies the technological path to follow
- CEM is used to evaluated multiple potential designs
  - Selection of a promising design
  - Fine tuning parameters via CEM iterations
  - Once acceptable results are achieved, construction of the physical system
- Progress on ancillary work and hardware acquisitions


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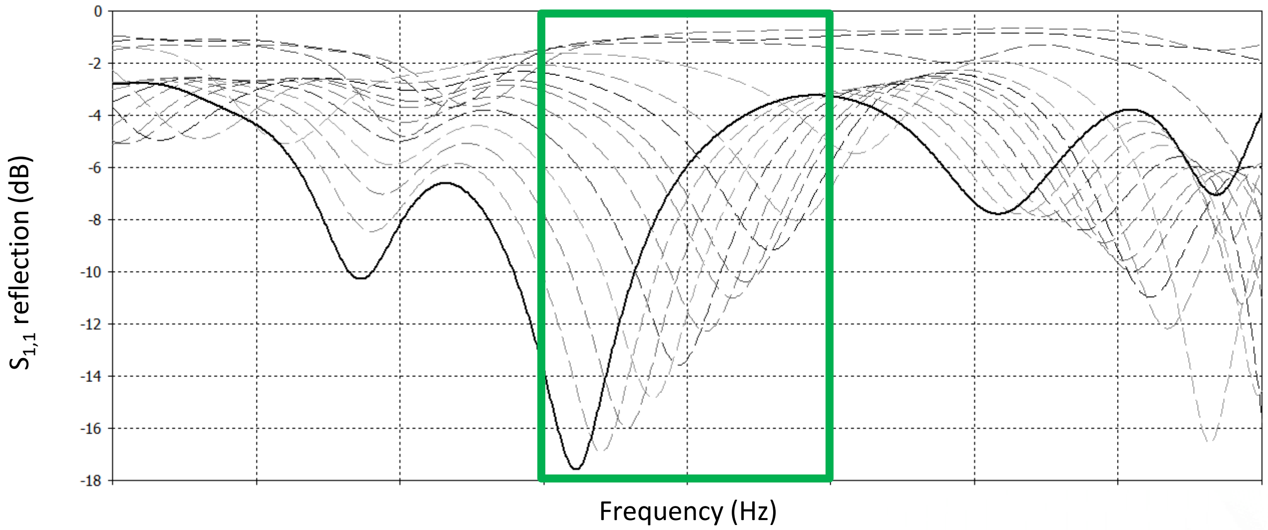
Questions?
Thank you for your attention

Technical Backup

Technical Accomplishments (FY2020)

Energy reflection: example of parameter sweep with CEM modeling



Plot: EM energy vs. frequency for a given design. Each dotted line represents a calculation for a given parameter value.

This example shows how a low reflection band shifts with a single parameter change. In this case, as the parameter is changed, the reflection is decreasing. However, it remains above the target of -20dB until the frequency leaves the band of operation (the green frame). This invalidates this parameter change.