Close-Proximity Electromagnetic Carbonization (CPEC)

(*Low Temperature Carbonization, LTC*)

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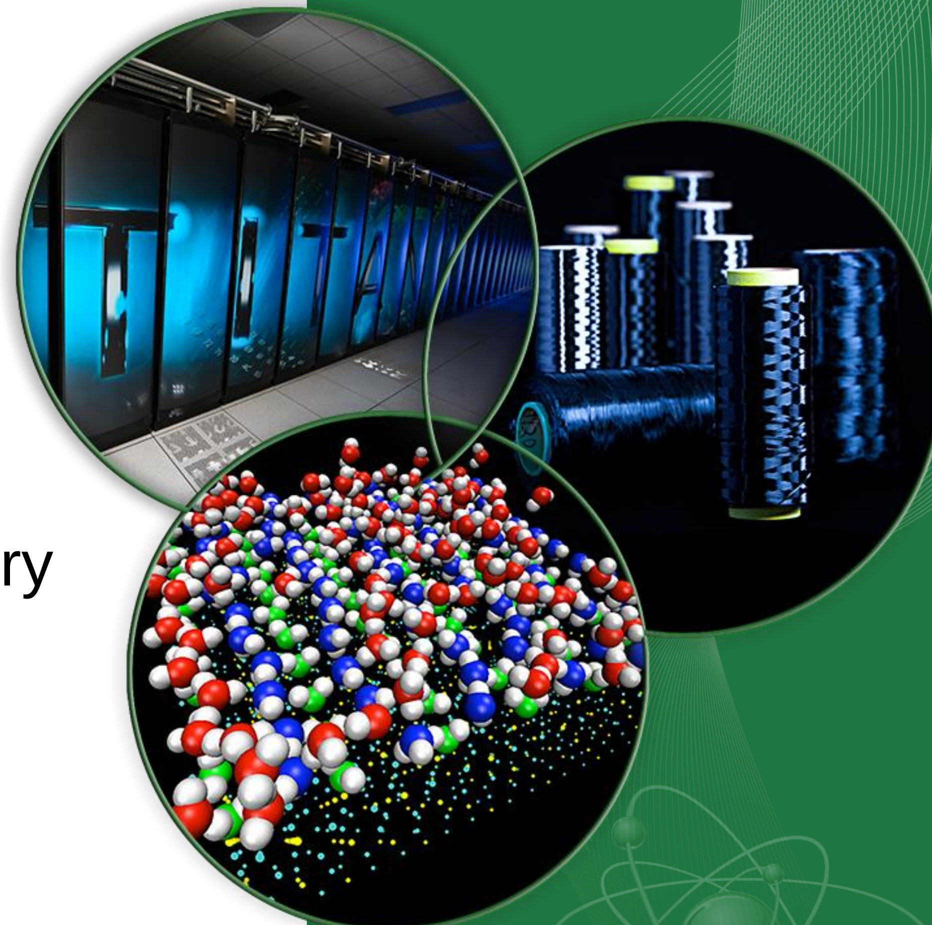

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Overview

Timeline

- Project Start: 10/1/15
- Project End: 9/30/19*
- Progress: ca. 65%

* Extension (at no additional cost) was requested and approved

Budget

Initial budget planning

- FY16 – FY19: $4.5M

Effective budget:

- Funding received in FY16: $1.5M
- Funding for FY17: $1.35M (10% cut)
- Funding for FY18: $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

- Project title:
  Close Proximity Electromagnetic Carbonization (CPEC):
  - Low temperature carbonization process (LTC)
  - Relies on dielectric heating (no convection)
  - Faster and more efficient that conventional
  - At atmospheric pressure.
- Project Goals:
  - Reduce unit energy consumption of LTC stage (kWh/kg) by ca. 50% (ca. 5% of the cost reduction on the CF overall manufacturing process).
  - Produce equal or better quality carbon fiber.
  - Scale the technology to a nameplate capacity of 1 annual metric ton and demonstrate by project end date (in progress).

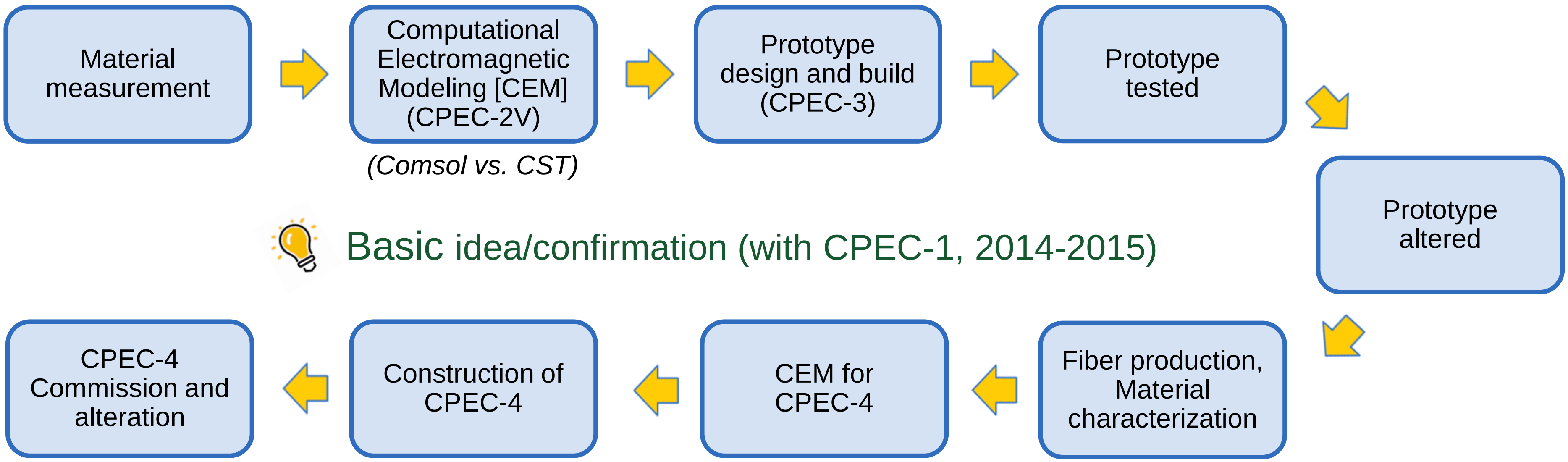
FY19 Milestones

Date	Milestone	Status
February 28, 2018	**M10:** Complete assembly of CPEC-4 and demonstrate stable/proper operation of all subcomponents for 20 minutes.	**Expected to Sep 15, 2018 (1st ext.)* Completed: 3/2019**
June 30, 2018	**M11:** Successfully carbonize 4x24k tows with final mechanical properties of greater or equal to 250 ksi tensile strength and 25 Msi Modulus in under 60 seconds.	**Expected to Feb 28, 2019 (1st ext.)* July 15, 2019 (2nd ext.)**
September 30, 2018	**Go/No Go M12:** Demonstrate at least 5% cost savings of the overall CF manufacturing process using CPEC technology versus conventional carbonization.	**Expected to Mar 31, 2019 (1st ext.)* Sept 30, 2019 (2nd ext.)**

** A first project extension was approved until March 31, 2019.*
A second extension was approved until September 30, 2019.

Historical Development

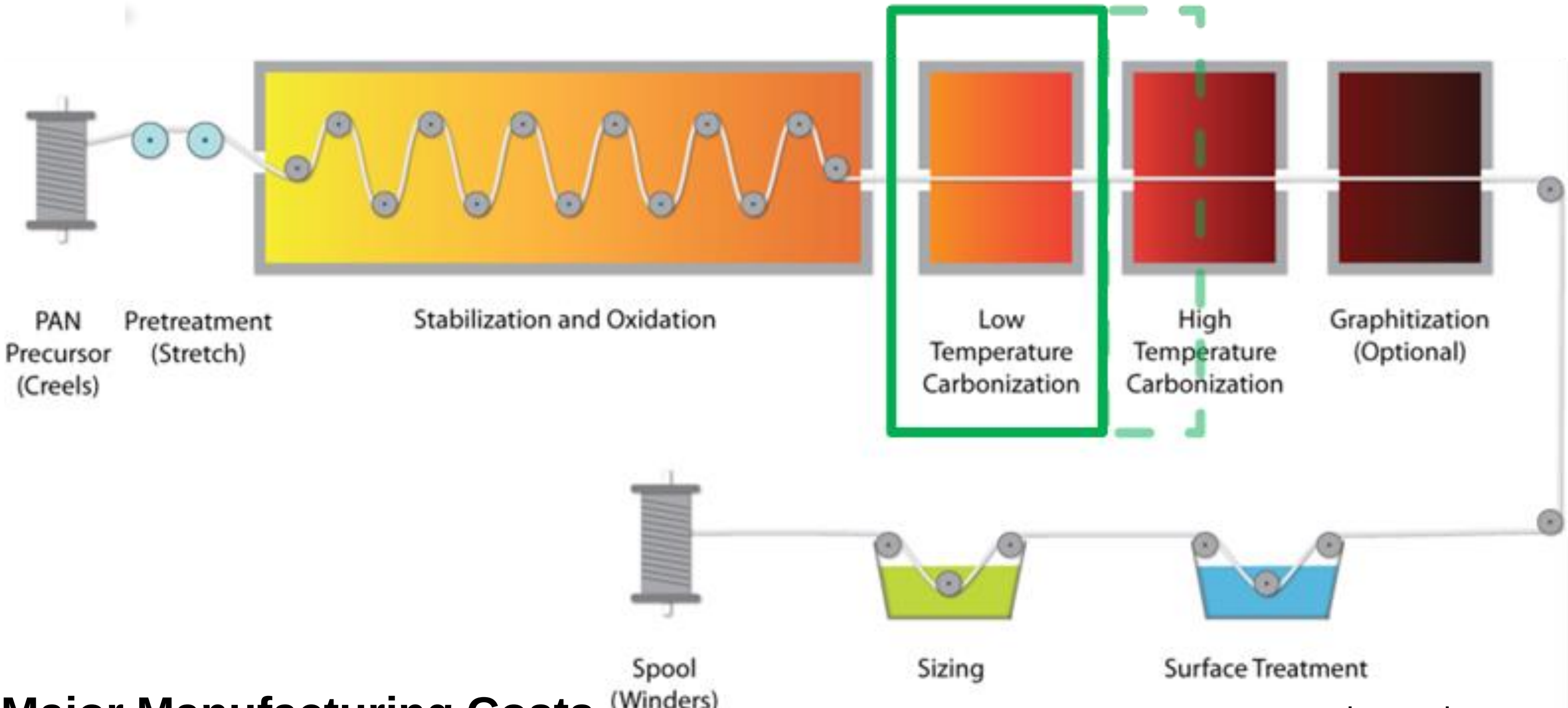
- Project flow:



- Material measurement/data acquisition (FY16):
  - wide span of temperature (10 carbonization levels) and frequency (broad band)
- CPEC-3 (FY17): demonstration of feasibility on batch or continuous process (one tow of 24k)
- CPEC-4:
  - Design completed (FY17-18); Ordering of the parts and components (FY18)
  - Construction (FY-18 until 04/19)
  - Commissioning and performance evaluation begun on 04/19 (partial delivery of the generator system)

Approach

Conventional PAN Processing



Major Manufacturing Costs

Precursor	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**

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Approach

- 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 – not the surrounding (hardware, gas, etc.).
- The 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.

Technical Accomplishments and progress (development as May 10,2019)

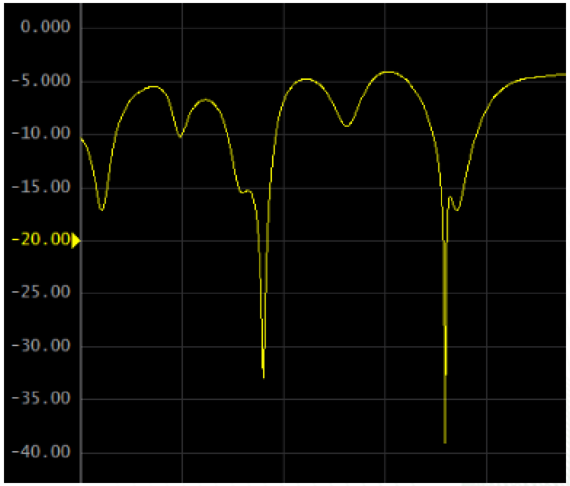
- Construction of CPEC-4:
  NOTE: CPEC-3 produced fiber achieving tensile strength of 340ksi and modulus of 25Msi in spite of no close loop control (data available in technical backup).
  - Modification of the lab space and the facility due to:
    - The vertical position of the setup → lab re-organization with modification of existing structure.
    - Power limitation in the facility (subcontracted) (power required by the generator was not available) → new power install inside and outside of the facility.

  - Installation of a vertical frame and all subcomponents:
    - Design, installation, and commissioning of a new fiber handling system.
    - Design, installation, and commissioning of the fiber pre-treatment section.
    - Design, construction, and installation of the process chamber and its applicators.
    - Safety analyze: design and construction of units for radiation containment.
  - Every single device/component was tested for performance prior to integration

Technical Accomplishments and progress (continuation)

- Creation a control system (LabVIEW development):
  - Development of the main human machine interface (HMI).
  - Monitoring of process and environmental data.
  - Data acquisition.
  - Safety system (automated interlocks base on sensors).

- Commissioning of the vessel
  - Performance consideration
    - Excellent energy transfer:
      low return loss on peaks in the band of interest (S_{11} < -30dB)
    - Limited crosstalk: max S_{21} ~ 10% (at full power)
  - Safety consideration
    - All measurements < ICNIRP* population standard at the band of interest



*ICNIRP: International Commission on Non-Ionizing Radiation Protection

Technical Accomplishments and progress (Recent development as May 10,2019)

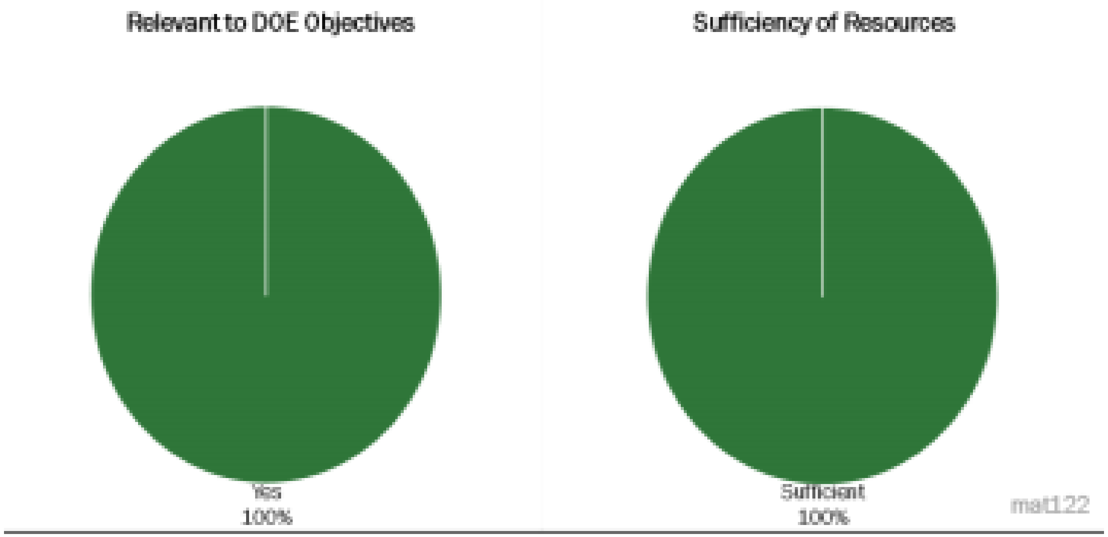
- Generator system was ordered on Feb 21, 2018
  - Delivery promised: July 2, 2018 (18 weeks lead time)
  - Effective delivery: March 12, 2019 (8 months delay, details in backup section)
  - No possible back-up solution with conventional generator*
    - state of the art in the RF area
    - Close loop controlling during processing
- No processed material with CEPC-4 yet (May 10, 2019).
- Actions since receipt of the first generator:

Action	Beginning	End
Installation (Generator)	3/13/2019	3/19/2019
Performance check (Generator)	3/28/2019	4/3/2019
Safety assessment	3/28/2019	4/23/2019
Commissioning (entire setup)	4/23/2019	N/A
First attempt of material processing	5/2/2019	5/2/2019
Second attempt	5/3/2019	5/3/2019
Corrective action	5/6/2019	Pending

* This type of generator was selected for its uncommon performance compare to other products on the market. Replacement solution was not possible.

Response to Previous Year Reviewer's Comments

- Previous year scoring:



- Question 1:

Approach to performing the work—the degree to which technical barriers are addressed, the project is well-designed, and well planned.

– Very encouraging comments from the reviewers:
  - "Excellent Project. The approach is sound. This should reduce the energy requirements".
  - "Project has made good headway in its goal to reduce energy consumption in the LTC-process".
  - "Potential to replace conventional thermal methods with directed energy coupling".
  - "The reviewer is simply left wondering if the technology can be extended to other elements of CF production for greater impact".

Answer:

➢ Thank you for your favorable comments

Response to Previous Year Reviewer's Comments

- <u>Question 2:</u>

Technical Accomplishments and Progress toward overall project goals—the degree to which progress has been made and plan is on schedule.

  - "The accomplishments from the Close Proximity Electromagnetic Carbonization (CPEC)- 3 furnace are excellent. The reviewer wished the standard deviations were less. The reviewer said that the mix of modeling and experimental accomplishments are very strong".
  - A significant amount of work accomplished. This work has demonstrated the feasibility of the technology and should also be recognized. Scatter in the strength results of event the most promising trail was too high. No data presented regarding energy requirements for the LTC. The project goals and relevance will be significantly enhanced by incorporating a comprehensive cost model to reflect increase in line capacity from shorter residence time as well as the impact on energy reduction".
  - "More work is needed to improve mechanical properties and reduce scatter".

<u>Answer:</u>

➢ CPEC-3 was a device built for proof of concept, capable of processing one single tow in batch or in continuous mode. This device offered low capability of adjustment to the morphological changes that the load experiences while processing. These morphological changes occur extremely rapidly, whereas the setting or the cavity adjustments were performed only manually. The tuning of the setup could not follow the morphological changes in real time. CPEC-4 will be more advanced in this area. As a consequence, the process will be more homogeneous and controllable.

Response to Previous Year Reviewer's Comments

- Question 3:

Collaboration and Coordination Across Project Team.

- "The collaboration with 4X Technologies (formerly RMX Technologies), 4M Carbon Fiber Corp., and Litzler is very good".
- "The team is focused on this targeted effort for carbonization. The plasma experts, the oven manufacturer, and a CF supplier are the proper team for this project.
- "Industrial partners appear adequate to support this project".

Answer:

➢ *This is a perfect match for the capabilities of the involved institutions. The partners offer highly specialized capabilities that complement each others.*

Response to Previous Year Reviewer's Comments

- Question 4:

Proposed Future Research—the degree to which the project has effectively planned its future work in a logical manner by incorporating appropriate decision points, considering barriers to the realization of the proposed technology and, when sensible, mitigating risk by providing alternate development pathways.

  - "Proposed research hinges on the CPEC-4 is a reasonable goal".
  - "Proposal is clear and concise. Would also be helpful for the author to present or outline the steps of economic evaluation".
  - "Addresses all the challenges now foreseen in the project".

Answer:

- *An economical evaluation requires measurement on CPEC-4 while processing under normal conditions. This evaluation will be the last step of this project.*

- *As of early Apr, the CPEC-4 is operating according to the design specifications. At this moment, no additional problems are expected.*

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Response to Previous Year Reviewer's Comments

- Question 5:

Relevance—Does this project support the overall DOE objectives?

- "The reviewer said that CF cost reduction is very important".
- "This project addresses the cost of CF by focusing efforts on reducing the energy for the Carbonization".
- "The current project supports the overall goal to expand the use of light-weighting materials".
- "The project deals with alternate ways of producing CF and may have an impact on the U.S. manufacturing".

Answer:

- More information about this issue can be provided based on experimental data. As indicated before, as a conclusion to this project, an economical evaluation will be carryout.

Response to Previous Year Reviewer’s Comments

- Question 6:

Resources—How sufficient are the resources for the project to achieve the stated milestones in a timely fashion?

- “The funding is sufficient”.
- “The team has adequate resources and expertise”.
- “Resources are sufficient for the project”.
- “Appears to be sufficient funding”.

Answer:

- *At this moment, it seams that the funding will be acceptable. Toward the end of the project, a more accurate statement about the budget/resources can be established.*

Collaboration and coordination

ORNL performed this project in collaboration with:

4XTECHNOLOGIES

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.

Remaining Challenges and Barriers

- Process material and achieve DoE's programmatic required mechanical properties.
- Enact a control system that monitors and reacts to:
  - Local area electromagnetic radiation levels. *(permanent monitoring)*
  - Vessel temperature monitoring and control. *(data required for energy and economical evaluation)*
  - Near real-time radiation response to material morphology. *(CPEC-4 will operate in a close-loop feedback mode)*
- Ensure proper full scale operation of CPEC-4 as predicted with acceptable uniformity width-wise. *(As May 10, CPEC-4 is operational but needs some refinements)*

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Proposed Future Research

- FY19
  - Upcoming work:
    - Fulfillment of MS10-MS12: Normal operation of CPEC-4 with 4 tows 24k with 60s achieving 250 ksi/25 Msi.
    - An economical evaluation of this technology will be performed as a final stage on this project. (MS12)
  - This project is scheduled to come to conclusion at the end of FY2019.

  - Propose research for a comprehensive solution for full carbonization process based on CPEC technology.

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

Summary

- CPEC-4 furnace was successfully built based upon:
  - material characterization
  - computer modeling
  - previous experimental work with CPEC-3.
- Some components of CPEC-3 were dismantled and have been repurposed into CPEC-4.
- The generator-based project delay: still an issue (first generator delivered mid March, 2019). Tested for performance. (Apr 3, 2019)
- CPEC-4 fully modeled with tunable and accessible design.

Thank you for your attention

Questions?

Technical Backup

Timeline/delay justification, Power generator's issue

Delivery of the power supply system: timeline of the 8 months delay:

Date of Action	Expected delivery time at 4X technologies	Comments
2/21/2018	7/15/2018	Original commitment (purchase order issued by ONRL)
6/15/2018	8/15/2018	Manufacturer requests more time for testing: Reengineering of the back panel needed
8/7/2018	9/24/2018	Procurement issue
9/6/2018	10/15/2018	Request for additional time for testing
9/26/2018	12/15/2018	Manufacturing issue with a smaller version
1/8/2019	2/15/2019	Teleconference between ORNL, 4X, and the manufacturer: commitment to ship the system to a partner/contractor to complete the construction
1/28/2019	N/A	Reception of the system by the partner: Beginning of evaluation of the work
1/30/2019	3/8/2019	Teleconference between ORNL, 4X, and the partner: Remaining work load estimated: ~10%
3/12/2019		First part of the system delivered (2nd part expected by end of May).

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Technical Accomplishments (FY2018)

Continuous Processing of Fiber with CPEC-3 Furnace

Mechanical properties of fully carbonized fiber (as of 11/2017)

Oxidation (conventional), LTC (CPEC-3), HTC (Conventional)*

Test#	Density (g/cc)	Diameter (Avg) µm	Std. Deviation	Tensile Strength (Avg) ksi	Std. Deviation	Modulus (Avg) Msi	Std. Deviation	Strain (Avg) %	Std. Deviation	Residence Time
1	1.8032	8.05	0.35	348.70	77.50	23.42	1.84	1.49	0.28	Long
2	N/A	8.20	0.41	303.00	87.50	22.73	2.76	1.40	0.32	Short
2	1.7924	8.44	0.74	356.60	135.30	24.88	3.83	1.42	0.47	Long
2	N/A	8.00	0.80	254.20	88.90	21.42	2.59	1.22	0.43	Long
3	N/A	8.40	0.53	333.00	149.80	25.44	3.45	1.29	0.51	Short
3	N/A	8.22	0.63	292.00	91.70	22.79	3.31	1.27	0.27	Short
3	N/A	8.42	0.46	331.30	125.00	23.44	1.84	1.48	0.55	Long
4	N/A	8.09	0.62	354.60	97.60	23.64	2.42	1.48	0.32	Short
4	N/A	8.06	0.72	263.60	132.80	22.31	3.61	1.13	0.44	Short
4	1.8138	8.91	0.63	340.20	101.70	25.14	1.73	1.39	0.43	Long
4	1.8135	8.73	0.56	285.50	98.50	23.07	2.03	1.23	0.37	Long

Table 1: Mechanical properties of fully carbonized samples at HTC. All residence times in CPEC-3 are shorter than 90 seconds. The values highlighted in green surpassed the dual programmatic requirements of 250ksi tensile and 25Msi modulus simultaneously.*

* HTC: High Temperature Carbonization