Recycling and Sustainability of Additive Manufacturing Feedstocks

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Outline

• Oak Ridge National Laboratory and Manufacturing Demonstration Facility Overview
• Metal Powder Utilization for Additive Manufacturing
  – Range of Powder Feedstocks & Synthesis
  – Recycling Powders (Inconel 718 and Ti-6Al-4V)
  – Hydride-Dehydride Powder Use in Electron Beam Powder Bed Depositions
• Polymer Additive Manufacturing
  – Thermoplastics
  – Carbon Fiber Recyclability
  – Future Use of Bio-Derived Polymers
The Manufacturing Demonstration Facility at Oak Ridge National Laboratory

Core Research and Development

- R&D in materials, systems, and computational applications to develop broad capability of additive manufacturing

Industry Collaborations

- Cooperative research to develop and demonstrate advanced manufacturing to industry in energy related fields

Education and Training

- Internships, academic collaborations, workshops, training programs, and course curriculum for universities and community colleges.

Supported by DOE’s Advanced Manufacturing Office

Neutron scattering: SNS and HFIR

- World’s most intense pulsed neutron beams
- World’s highest flux reactor-based neutron source

Advanced Materials

- DOE lead lab for basic to applied materials R&D
- Technology transfer: Billion dollar impacts

Leadership-class computing: Titan

- Nation’s most powerful open science supercomputer

Advanced Manufacturing

- Novel materials
- Advanced processing
# MDF Strategic Plan 2016-2021

<table>
<thead>
<tr>
<th>MDF Mission</th>
<th>MDF Vision</th>
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</thead>
<tbody>
<tr>
<td>Develop and mature additive manufacturing and composite technologies for clean energy applications.</td>
<td>A competitive America using additive and composite processes in mainstream manufacturing industries to achieve carbon neutrality and energy independence.</td>
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## Goals

1) Improved Performance Characteristics of AM Components

2) Qualification and Certification of AM Components for Intended End Use

3) AM Systems Optimized to Achieve Mainstream Manufacturing Application

4) Comprehensive Understanding of AM Process Capabilities and Limits
Metal Powder Feedstock Project Overview

- **Project Objective:** Perform R&D for feedstock material to
  - Improve the supply chain integration
  - Better understand the process science and nomenclature needed for AM.

- **The project will**
  - Determine required powder qualities for enabling successful builds
  - Investigate starting materials to decrease the overall cost

- **Summarized Problem:** New supply chains and feedstock providers with ample production rates are required for commercialization.

### Titanium Powder Available and Relative Costs

<table>
<thead>
<tr>
<th>Type of Feedstock</th>
<th>$/kg</th>
<th>SEM Image of Typical Morphologies</th>
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</thead>
<tbody>
<tr>
<td>PREP and PA</td>
<td>407 - 1,210</td>
<td></td>
</tr>
<tr>
<td>Gas Atomized</td>
<td>165 - 330</td>
<td></td>
</tr>
<tr>
<td>HDH</td>
<td>66 - 176</td>
<td></td>
</tr>
<tr>
<td>Sponge Fines</td>
<td>11 - 33</td>
<td></td>
</tr>
<tr>
<td>Alternative Powders</td>
<td>33 - 88</td>
<td></td>
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</table>

DOE- Advanced Manufacturing Office, Feedstock Materials Study

- Project focused on the development of powder, and particulate feedstocks for AM.

1. Powder physical characteristics to successfully build powder bed AM parts

2. Powder compositions to meet mechanical criteria

Inconel 718 & Ti-6Al-4V

Common PM Characteristics
- Apparent & Tap Density
- Spheroidicity
- Flowability
- Chemistry
- Particle Size Distribution
- Porosity

Unique Characteristics
- Spreadability
- Recyclability
- Powder Bed Density
Over Nine Different Inconel 718 Powder Sources Evaluated and Characterized

- 5 Different Atomization Techniques
- All Chemistries Fell Within Specification
- Spheroidicity and Flowability Characterization
- Laser Scattering and Sieve Data Taken for Powder Size Distribution

<table>
<thead>
<tr>
<th>Table 2a: Comparison of External Microscopy Images (Top) with Cross Sections (Bottom) of Gas Atomized Ni Alloy 718 Powders</th>
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<tbody>
<tr>
<td><img src="image1.png" alt="Image" /></td>
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<tr>
<td><img src="image7.png" alt="Image" /></td>
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</table>

<table>
<thead>
<tr>
<th>Table 2b: Comparison of External Microscopy Images (Top) with Cross Sections (Bottom) of Other Atomization Ni Alloy 718 Powders</th>
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</thead>
<tbody>
<tr>
<td>Rotary Atomized</td>
</tr>
<tr>
<td>Powder G</td>
</tr>
<tr>
<td><img src="image13.png" alt="Image" /></td>
</tr>
<tr>
<td><img src="image16.png" alt="Image" /></td>
</tr>
</tbody>
</table>
Developing the Powder Characteristics Required for Powder Bed Technologies

- **Gas Atomized**
  - Powder Induced Porosity: 0.873%
  - Process Induced Defects

- **Rotary Atomized**
  - Powder Induced Porosity: 0.491%
  - Process Induced Defects

- **Plasma Rotated Electrode**
  - Powder Induced Porosity: 0.000%
  - Process Induced Defects

<table>
<thead>
<tr>
<th>Process Type</th>
<th>Powder Porosity</th>
<th>Deposit Porosity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas Atomized</td>
<td>0.873%</td>
<td>0.117%</td>
</tr>
<tr>
<td>Rotary Atomized</td>
<td>0.491%</td>
<td>0.037%</td>
</tr>
<tr>
<td>Plasma Rotated Electrode</td>
<td>0.000%</td>
<td>0.000%</td>
</tr>
</tbody>
</table>

Porosity in Powder and Deposit percentages are given for each process type.
Morphology of Ti-6Al-4V Powders

Plasma Atomized (PA)

Similar Results for Ti-6Al-4V
- EIGA contains limited porosity
- PREP and PA powders largely free of pores at low resolution

Flowability, Tap Density, and Other Powder Characteristics Available for Inconel 718 and Ti-6Al-4V

Electrode Induction Gas Atomized (EIGA)

Plasma Rotated Electrode Powder (PREP)
Recyclability Study of E-Beam Powder Bed

Studies were conducted on both Inconel 718 and Ti-6Al-4V feedstocks in Arcam Electron Beam Powder Bed:

- Inconel 718 was performed using Plasma Atomized powders
- Ti-6Al-4V was performed using EIGA powders
- Fabricate components using a standard build geometry in x-y with variation in Z height
- Worst case scenario where all powder was used each time
## Chemical Analysis of Powder and Builds (Inconel 718)

<table>
<thead>
<tr>
<th>Element</th>
<th>As Received Powder</th>
<th>Powder After Build 6</th>
<th>Build 1</th>
<th>Build 6</th>
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<td></td>
<td>Wt%</td>
<td>Wt%</td>
<td>Wt%</td>
<td>Wt%</td>
</tr>
<tr>
<td>Carbon</td>
<td>0.044</td>
<td>0.042</td>
<td>0.044</td>
<td>0.040</td>
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<td>Sulfur</td>
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<td>--</td>
<td>0.002</td>
<td>0.002</td>
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<tr>
<td>Oxygen</td>
<td>0.014</td>
<td>0.022</td>
<td>0.006</td>
<td>0.009</td>
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<tr>
<td>Nitrogen</td>
<td>0.020</td>
<td>--</td>
<td>0.016</td>
<td>0.015</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>0.0002</td>
<td>0.0002</td>
<td>0.0005</td>
<td>0.0003</td>
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<tr>
<td>Nickel</td>
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<tr>
<td>Chromium</td>
<td>18.40</td>
<td>18.09</td>
<td>17.84</td>
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<tr>
<td>Molybdenum</td>
<td>3.10</td>
<td>2.96</td>
<td>3.13</td>
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<tr>
<td>Niobium</td>
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<td>4.84</td>
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<tr>
<td>Manganese</td>
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<td>0.088</td>
<td>0.080</td>
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<tr>
<td>Copper</td>
<td>0.15</td>
<td>0.14</td>
<td>0.13</td>
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<td>Aluminum</td>
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<tr>
<td>Titanium</td>
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<td>0.86</td>
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<td>0.91</td>
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<tr>
<td>Silicon</td>
<td>0.19</td>
<td>--</td>
<td>0.22</td>
<td>0.23</td>
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<tr>
<td>Phosphorous</td>
<td>0.006</td>
<td>--</td>
<td>0.008</td>
<td>0.008</td>
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<tr>
<td>Boron</td>
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<td>--</td>
<td>&lt;0.0005</td>
<td>&lt;0.0005</td>
</tr>
<tr>
<td>Cobalt</td>
<td>0.14</td>
<td>--</td>
<td>0.15</td>
<td>0.15</td>
</tr>
<tr>
<td>Iron</td>
<td>18.59</td>
<td>18.57</td>
<td>18.70</td>
<td>18.42</td>
</tr>
</tbody>
</table>

- Minimal preferential elemental losses during fabrication of builds.
- Chemistries of builds are consistent with that of the powder.
### Chemical Analysis of Powder and Builds (Ti-6Al-4V)

<table>
<thead>
<tr>
<th>Element</th>
<th>As Received Powder</th>
<th>Powder after Build 5</th>
<th>Build 1</th>
<th>Build 5</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Wt%</td>
<td>Wt%</td>
<td>Wt%</td>
<td>Wt%</td>
</tr>
<tr>
<td>Aluminum</td>
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<td>6.36</td>
<td>5.82</td>
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<tr>
<td>Vanadium</td>
<td>4.14</td>
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<td>0.141</td>
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<tr>
<td>Nitrogen</td>
<td>0.025</td>
<td>0.025</td>
<td>0.019</td>
<td>0.025</td>
</tr>
<tr>
<td>Carbon</td>
<td>0.015</td>
<td>0.018</td>
<td>0.020</td>
<td>0.028</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>0.0016</td>
<td>0.0024</td>
<td>0.0025</td>
<td>0.0015</td>
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<tr>
<td>Iron</td>
<td>0.19</td>
<td>0.20</td>
<td>0.20</td>
<td>0.22</td>
</tr>
<tr>
<td>Niobium</td>
<td>--</td>
<td>&lt;0.002</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Tungsten</td>
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<td>&lt;0.002</td>
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<td>--</td>
</tr>
<tr>
<td>Copper</td>
<td>--</td>
<td>--</td>
<td>0.015</td>
<td>0.029</td>
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<tr>
<td>Silicon</td>
<td>0.022</td>
<td>0.023</td>
<td>0.024</td>
<td>0.020</td>
</tr>
</tbody>
</table>

- **Ti-6Al-4V Chemistry Changes**
  - Increased oxygen content after 5 builds.
  - Aluminum content fluctuates.
  - Overall oxygen and aluminum content still within specification (175 hours, 5 build cycles).
Recyclability Study

Studies were conducted on both Inconel 718 and Ti-6Al-4V powders to determine the worst case scenarios – Initial Powder Chemistries determine the powder usage over multiple cycles.

Inconel 718

- No change in chemistry over 270hrs of use.
- Minimal change in flowability.
- Can be used for a large number of cycles.

Ti-6Al-4V

- Oxygen concentration in builds increased to 0.17wt% over 150hrs of usage.
- Chemistry limits the powder use.
Additive Manufacturing of HDH Process Powder

Hydride-Dehydride Process

Hydride
\[ \text{Ti} + \text{H}_2 \rightarrow \text{TiH}_2 \]

Crush/size down

Dehydride
\[ \text{TiH}_2 \rightarrow \text{Ti} + \text{H}_2 \]
HDH Powder Successfully E-Beam Deposited Using ARCAM

- HDH CP Ti and Ti-6Al-4V
  - CP Ti HDH Powder Successfully Deposited
  - 6-4 Deposited, But Not Fully Dense Due to Powder Particle Size Distribution

- HDH CP Ti Meets ASTM Specifications

- HDH 6-4 with Smaller Particle Size Distribution To Be Tested

*First Time HDH Successfully Deposited!*
**Microstructural and Chemical Analysis**

### Chemical

<table>
<thead>
<tr>
<th>Sample Identification</th>
<th>Oxygen</th>
<th>Nitrogen</th>
<th>Carbon</th>
<th>Iron</th>
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</thead>
<tbody>
<tr>
<td>Powder</td>
<td>0.167</td>
<td>0.163</td>
<td>0.056</td>
<td>0.050</td>
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<tr>
<td>CP Ti Run 1</td>
<td>0.166</td>
<td>0.154</td>
<td>0.024</td>
<td>0.021</td>
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<tr>
<td>CP Ti Run 2</td>
<td>0.165</td>
<td>0.153</td>
<td>0.023</td>
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<tr>
<td>Sample Run 1</td>
<td>0.163</td>
<td>0.154</td>
<td>0.024</td>
<td>0.021</td>
</tr>
<tr>
<td>CP Ti</td>
<td>0.163</td>
<td>0.154</td>
<td>0.024</td>
<td>0.021</td>
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**ASTM Standard**

<table>
<thead>
<tr>
<th></th>
<th>F-1</th>
<th>F-2</th>
<th>F-3</th>
<th>F-4</th>
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<tbody>
<tr>
<td>Oxygen</td>
<td>0.18</td>
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<td>Nitrogen</td>
<td>0.03</td>
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<tr>
<td>Carbon</td>
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<tr>
<td>Iron</td>
<td>0.02</td>
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No Porosity Found

**Meets Specification for Grade 1**
# Mechanical Data

<table>
<thead>
<tr>
<th>Specimen Number</th>
<th>Yield (PSI)</th>
<th>Ultimate (PSI)</th>
<th>Elongation (%)</th>
<th>.2% Elongation (%)</th>
<th>Total Plastic Strain (%)</th>
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<td>ASTM F-1</td>
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<td>35000</td>
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<td>24 (min)</td>
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<tr>
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<td>63760</td>
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<td>83870</td>
<td>25.67</td>
<td>3.277</td>
<td>22.393</td>
</tr>
</tbody>
</table>

Meets Mechanical Specifications with Strength Comparable to Grade 4 and Ductility of Grade 1
Research focus:

- **In-situ characterization control**
  - Thermography
  - Vision systems

- **Materials development**
  - Specific strength equivalent to heat treated Al alloys
  - High strength and fiber reinforced polymers

- **Isotropic mechanical properties**
  - Process modifications to improve build-direction strength

- **BAAM**
Polymers material development

- Low-cost desktop systems used for material development
- CF reinforced polymers have demonstrated 2x increase in strength and 4x increase in stiffness
- Reinforced polymers allow high resolution, low distortion printing
- Performance of low-cost printers equivalent to higher-cost industrial systems when using reinforced materials
Residual Issues in FDM AM

- Additive Manufacturing is:
  - Slow: Deposition Rates ~ 1-2 ci/hr
  - Expensive: Material Cost ~ $2.25/ci or ~$58/lb (ABS)
  - Small Parts: Largest Workspace 3’ x 2’ x 3’ (31,104 ci)
    - 3.5 Years to build a part that takes up the full volume at 1ci/hr
Big Area Additive Manufacturing (BAAM)

• **Obstacle:** Most additive processes are slow (1-4 in³/hr), use higher cost feedstocks, and have small build chambers.

• **Solution:** ORNL has worked with equipment manufacturers and the supply chain to develop large scale additive processes that are bigger, faster, cheaper, and increase the materials used.

- **Large Scale Printers**
  - Cincinnati System 8’x20’x6’ build volume

- **Fast Deposition Rates**
  - Up to 100 lbs/hr (or 1,000 ci/hr)

- **Cheaper Feedstocks: Pellet-to-Part**
  - Pelletized feed replaces filament with up to 50x reduction in material cost

- **Better Materials**
  - Higher temperature materials
  - Bio-derived materials
  - Composites Hybrids
BAAM Printing AMIE
Small House and Car
BAAM Uses a Screw Extruder

- Four heat zones and heated end cap
- Pressure sensor and multiple thermocouples
- Six screws with and without mixers
- Cooled feed section and screw
- Nozzle diameter 0.1” – 0.4”
BAAM Materials Processed

Thermoplastics
- PLA/Copper
- ABS
- 20% CF/ABS (large scale)
- ABS/GF
- ABS/Boron
- Nylon 12
- HIPS (High Impact Polystyrene)
- NinjaFlex
- ULTEM (high temp)
- ULTEM/CF (high temp)
- PEKK (tech collaboration- Arkema)
- PEEK/PBI (high temp)
- PPS (high temp)
- 25% & 40% CF/PPSU (high temp, large scale)
- 40% & 50% CF/PPS (high temp, large scale)
- 40%, 50% & 60% Apollo (60% low & high crystallinity) (high temp, large scale)**
- PA6/CF (large scale)
- PA6
- PA6/GF (large scale)
- PC/CF (large scale)
- Amphora

Thermosets / Reactive
- 40 & 55 vol. % copper-filled epoxy resin (base epoxy mixture) for heat exchangers
- 5-10 vol. % zirconia (ceramic powder)
- 10 vol. % CF-reinforced epoxy resin
- Bio-reinforced thermoset resins
- Epoxy/SiC
- Silicone
- Silicone/SiC
- Epoxy/Nanoclay
- Epoxy/CF
- Epoxy/MQA
- Epoxy/CNT
- Epoxy/Graphene
- Epoxy/Microspheres
- Polyurea with magnetic media
- Polyurea/CNT
- Polyurea/CF
- Environmentally-activated polyurethane
- Polyurethane
- WC, WC-Co

Break Down and Remelt

Mechanical Recycling or Thermochemical Fiber Reclamation
Recycling Strategies for Large Scale Additive Manufacturing

Big Area Additive Manufacturing

BAAM process generates high value products in energy efficient manner, however recycled feedstock is currently not accepted and printed structures are not recycled.

Printing Recycled Materials

- Using recycled plastics as a component of feedstock
- Incorporating recycled fibers in feedstock

Recycling of Printed Structure

- Re-grind of material to adjust viscosity
- Re-process plant waste
- Separate materials and re-cycle printed parts

Challenges

- Consistency of recycled feedstock
- Re-grind of large structures
- Influence of lower molecular weight on mechanical properties

Printing Recycled Materials

- ORNL printed a test sample using ABS with recycled carbon fiber from MIT
- Local Motors is investigating the influence of recycled material on mechanical properties for 0%, 20% 40%, 60%, 80% and 100% volume fraction.
Addressing Critical Challenges

Five/Ten Year Technical Goals

• 25/50% lower carbon fiber–reinforced polymer (CFRP) cost
• 50/75% reduction in CFRP embodied energy
• 80/95% composite recyclability into useful products

Impact Goals

• Enhanced energy productivity
• Reduced life cycle energy consumption
• Increased domestic production capacity
• Job growth and economic development
Increasing volume of materials to be recycled in aeronautics

Increasing volumes of materials to be recycled

- In the mid-term, end-of-life products will need to be recycled
- In the short term, most of the composite waste will come from production scraps
Aeronautics is not the only user of CFRP

- Conservatively, 30% of virgin carbon fiber ends up as scrap to be recycled.
- Ultimately, 100% of fiber needs to be recycled as end-of-life material.
Opportunity

• In North America, 29 million pounds (~13,200 MT) of carbon fiber waste estimated going to landfill per year. In the form of

  – Pre-preg, primarily aerospace production scrap
  – Secondary amount is cured production trim
  – Some pre-preg scrap has to be oven cured prior to landfill
  – Regulations vary based on constituents in resin system
  – Adds cost/time burden on composite manufacturers

• It is difficult to estimate amount of end-of-life composite
Recycled Carbon Fiber and Value

Mechanical recycling
- Shredding/Crushing/Milling
  - grinded CFRP
    - Classification
      - powdered CFRP
      - fibrous fragments
  - Waste Preparation
    - Oxidation (fluidized bed process)
    - Chemical Recycling
    - Pyrolysis
  - reclaimed CF in “fluffy” form
  - Direct molding with new resin
    - RCF-filled composite
    - RCF-reinforced composite
    - RCFRP (short-fiber, quasi-isotropic)
    - RCFRP (medium length, planar orientation)
    - RCFRP (partially aligned, high fiber content)
    - RCFRP (woven architecture)

Thermochemical fiber reclamation
- CFRP raw waste
  - clean, size-reduced CFRP waste
  - Pyrolysis
  - Reclaimed CF woven fabric

Out-of-date woven prepreg

Challenges

• Supply Chain, need to know where material comes from and what it contains (fire retardants, etc), and available in what format
  – Sorting, Classifying, labeling

• For value added products, higher fiber volume fraction preform, aligned fibers etc. which could be achieved by new methods of preforming

• Development of standard for products will give confidence to designers and end users.

• Today Landfill fee/tax is not a driver for C-fiber recycling in US. However further legislation or banning landfill will drive recycling

• Life Cycle Assessment studies for recycling processes is needed.
Recycling Proposals Received

Automotive component and 3D printed tool both use reclaimed CF

Approved

Recycling aircraft components into 3D printed automobiles

In contracting

Profitably recycling glass and carbon composites

In review cycle

Automotive component uses CF reclaimed from scrap prepreg

In review cycle
Two Main Mission in Bio-Manufacturing

- We import 49% of the petroleum used.
- We send close to $1 billion per day overseas to buy oil.

Redefining biorefinery: Develop value added byproducts from Biorefineries

Wood, Energy crops, Grasses, Waste

PRODUCTS
- Energy (fuels, heat, electricity), Chemicals, Other Materials, Food and Feed
Next Generation Agribusiness ("NGA") is about growing perennial crops and turning those crops into finished products in the same community.

It combines the two powerful economic drivers of agriculture and manufacturing.

Commercial Farming  Research & Development  Manufacturing

Farm in rural America, just as important, manufacture in rural America
Bio-Materials are high in Value chain and pull can come from Manufacturing

- Forestry and harvesting
- Wood products, Pulp & paper, packaging
- Bioenergy, heat and power
- Biofuels
- Biochemicals
- Biomaterials
- Medical cosmetics

New materials, products, services
- Present products

Biopolymers, composites...
Materials solutions are most of the time in composites

High Mechanical
High Tg Polymer
Process temp is lower than 200C
Printable
High Volume
Low Cost
Biodegradable compostable

Biodegradable
Printable
Bio-derived
• 110lbs of 20% and 30% flax fiber-PLA pellets will be received and tested at BAAM.
Cellulose Fibrils are in many forms and functionality

Advantages:
- Abundant, renewable resource with price stability
- Compostable
- Biocompatible
- High strength and modulus
- Lightweight
- Shear thinning thickener (stable against temperature and salt addition)

2014: Thomas Reuters names nanocellulose as one of the top 10 technologies that will change the world by 2025.
40%CNF-PLA
- ~93% of specific strength of Aluminum 6061-T4 alloy.
- Stronger and lower cost than CF-ABS composite

- 100% BIO

- **40%CNF-PLA:** $0.4\times2/\text{lb} + 0.6\times3/\text{lb} = 2.6/\text{lb}$
- **30%CF-ABS:** $0.3\times12/\text{lb} + 0.7\times3/\text{lb} = 5.7/\text{lb}$
SEM Images of Fracture Surfaces: Compression-molded samples

Low Resolution:
• Increase in surface roughness with increasing nanocellulose content: Indication of bulk dispersion

High Resolution:
• Cellulose nanofibrils are in bundles/network rather than individually dispersed.
• Polymer resin penetrated among the fibrils possibly creating mechanical interlocking.
BAAM-printing of Bamboo with PLA
BAAM-printing of 20% Flax fiber-PLA system