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- Milestones
- Technical Approach
- Results and Accomplishments
  - Improvements in Tensile Strength
  - Microscopy Characterization
- Reviewer Responses
- Collaborators
- Remaining Challenges
- Future Work
- Summary
Project Overview

**Project Timeline**
- Start: May 2011 (CRADA)
- Finish: Sept. 2015 (delayed due to subcontractor equipment problem)
- 85% Complete

**Budget**
- Total project funding
  - DOE – $1,115K
    - FY11 Funding - $300K
    - FY12 Funding - $395K
    - FY13 Funding - $300K
    - FY14 Funding - $120K
    - FY15 Funding – carryover
- Cummins and commercial participants providing $1115K cost share as in-kind materials and effort

**Barriers**
- Lack of suitable aluminum alloys meeting elevated temperature strength and durability requirements for heavy duty diesel propulsion applications
- High temperature and high strength aluminum alloys in existence require expensive processing methods.
- Material processing requires scale-up and development of supplier base
- Non-disclosed (business sensitive) processing parameters of scaled up material

**Partners**
- Cummins
- UC Riverside
- Transmet Corporation
- Rapid Solidified Metals Flakes, Fibers, Spheres, Other
  "Shape Makes A Difference"
Project Relevance

Overall Objective: To develop aluminum alloys with enhanced high temperature strength for improved efficiency in heavy duty diesel engines while maintaining low cost, high volume production capability.


- Increase the ultimate tensile strength of aluminum alloys at elevated temperature
- Show production method is viable by the successful scale up of rapidly solidified and extruded material.
- Test and evaluate the produced RS Al material, extruded and forged to show material performance at or above the 300MPa/300°C and Cummins B10 service life requirements

Relevance

- Higher operational temperatures and weight reductions improve engine performance and fuel efficiency.
- Direct Cost Savings Mechanical alloying (MA) is too expensive for large-scale commercial applications
- Enhanced Material Performance in competition with more expensive titanium and nickel-based alloys in selected applications
Milestones & Go/No-Go’s (FY14-15)

► Receive and characterize Transmet production scale rapidly solidified
  • Flake Material
  • Extrusions
  • Forged Extrusions
Complete (2014)

► Perform Iterative testing to identify key process parameters
  • Tensile Testing (Room & Elevated Temperature)
  • Failure Analysis & Characterization of tested specimens
In Progress (2014) on track

► Meet 300MPa/300°C design criteria
In Progress (2014) on track
(298MPa /300°C)

► Perform fatigue testing on best product material
Tasked (2015)

► Establish cost and performance benefits of fabricated Al alloys over competing materials (high temperature steels, Ni alloys, titanium)
Tasked (2015)

► Summarize, publish & report results
In Progress (2015) on track
**Technical Approach/Strategy**

**Unique Aspects of RS Approach**
- Identification of Al alloys has focused on research and development of new alloy compositions which have previously been untested.
- Rapid solidification is an ideal low cost method for producing large quantities of material that have significant potential for high temperature strength.
- Transmet Corp. was identified and partnered with to produce large scale RS material of the alloy composition developed at PNNL.
- Detailed microstructural investigations on the RS material and heat treated flake are providing insight on the thermal processing history of the samples.

**Key Barriers Addressed**
- Lack of suitable aluminum alloys meeting high temperature strength and durability requirements for heavy duty diesel engine applications.
- High temperature and high strength aluminum alloys in existence require expensive processing methods (Mechanical Alloying).
- Material processing requires scale-up and development of supplier base.
- Unknown (proprietary) processing parameters of Transmet material make it difficult to define key process controls for future fabrication.

**Research Integration**
- CRADA Project with Cummins, Inc.
- Transmet Corp. produced 500 lb. of RS flake and converted the material to 75 mm diameter extruded product.
- Commercial scale cost analysis has been initiated by Cummins and PNNL, and will use input from Transmet and aluminum producers.
## Strategy Status & Go/No-Go’s for FY14-15

<table>
<thead>
<tr>
<th>2014</th>
<th>2015</th>
</tr>
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<tbody>
<tr>
<td>Apr</td>
<td>May</td>
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- Received 500lbs (Production Scale) of Extruded and Forged Aluminum Flake for Testing
- Fabrication & Evaluation of Extruded & Forged Tensile Specimens
- High Temperature Evaluation of RS material (300MPa Target at 300°C)
- Characterization & Evaluation of High Temperature Effects
- Iterative Improvements, Fatigue Testing
- ID Performance Benefits
- Reporting

*Demonstrate Cummins required B10 service life*

### Complete Project Overview 2011-Current

<table>
<thead>
<tr>
<th>Complete Project Overview 2011-Current</th>
<th>FY11</th>
<th>FY12</th>
<th>FY13</th>
<th>FY14</th>
<th>FY15</th>
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<tr>
<td>Identify Candidate Materials and Processing Methods/Small Scale Production</td>
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<td>Production and Testing of Candidate Alloys &amp; Methods</td>
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<td>Large Scale Production Continued Testing &amp; Specimen Characterization</td>
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<tr>
<td>Complete Characterization/Publication &amp; Annual Report Submitted</td>
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Technical Accomplishments I
Evaluation of Production Scale RS Material

FLAKE MATERIAL
Strong cooling gradients were seen in the flake material producing four microstructural zones.
1-Nanocrystalline
2-Dendritic/Cellular
3-Rings
4-Lamella/Eutectic Phase

Consolidation of flake (hot pressed and extruded) shows initial morphologies are maintained.

Refined flake microstructure is likely to achieve high strength & meet design criteria.
## Technical Accomplishments 2

### Tensile Results

#### Small Scale Tensile Testing (Status at Previous Review)

<table>
<thead>
<tr>
<th>Alloy Designation</th>
<th>Condition</th>
<th>Test Temperature (°C)</th>
<th>Yield Strength (MPa)</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>Failure Strain (%)</th>
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<tbody>
<tr>
<td>AFM-11 shot</td>
<td>Extruded</td>
<td>Room</td>
<td>287.0</td>
<td>411.2</td>
<td>1.8</td>
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<tr>
<td>AFM-11 shot</td>
<td>Extruded</td>
<td>300°C</td>
<td>185.6</td>
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<td>AFM-11 flake</td>
<td>Extruded</td>
<td>Room</td>
<td>427.8</td>
<td>493.6</td>
<td>7.2</td>
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<tr>
<td>AFM-11 flake</td>
<td>Extruded</td>
<td>300°C</td>
<td>256.8</td>
<td>256.8</td>
<td>17.0</td>
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</table>

Small scale testing led to material down selection
FY14/15 has focused on evaluating RS flake material

<table>
<thead>
<tr>
<th>Alloy Designation</th>
<th>Condition</th>
<th>Test Temperature (°C)</th>
<th>Yield Strength (MPa)</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>Failure Strain (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AFM-11 flake</td>
<td>Extruded</td>
<td>Room</td>
<td>544.2</td>
<td>544.2</td>
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<tr>
<td>AFM-11 flake</td>
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<td>Forged Ext.</td>
<td>Room</td>
<td>351.3</td>
<td>360.7</td>
<td>2.7</td>
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<td>AFM-11 flake</td>
<td>Forged Ext.</td>
<td>300°C</td>
<td>200.0</td>
<td>202.7</td>
<td>2.1</td>
</tr>
</tbody>
</table>

Extruded flake shows higher strength than forged extrusions.

- Room and high temperature tensile testing have been successfully demonstrated
- Large scale extruded AFM-11 flake alloy nearly meets 300MPa/300°C milestone target

Project Barrier Encountered: Proprietary processing conditions of Transmet RS material blur understanding of strength reductions seen in forged extrusions.

Actions: 1) Evaluate microstructure of tensile specimens and 2) Identify temperature vs. microstructure relationships to understand heat treatment effects on RS flake
Technical Accomplishments 3

Evaluation of Tensile Specimen Microstructures

- Extruded samples (top) have a more refined microstructure than the forged extrusions (bottom)
- Significant structure changes were not seen between the RT and 300°C target operational temperatures
  - Dark contrast regions are representative of pure aluminum indicating phase segregation
- Phase decomposition (longer time at elevated processing temperature) is likely responsible for strength reduction of the forged material.

Tensile microstructures indicate a need to evaluate phase stability of AMF-11 at elevated temperatures (similar those expected during consolidation & forming) to define ideal processing temperatures.
Rapid Solidification is a non-equilibrium process, knowledge of high temperature phase transformations can be used to create fine dispersions of second phase particles that have the potential to increase strength. Growth of large second phase particles can lead to volumetric changes, void formation and decrease strength.

- Heat treatments were performed on flake in the expected processing temperature regime
- Microscopy has indicated new phases have potential to form in the 450-500°C temperature range
- Precipitation of new phases can impact strength positively or negatively depending on coherency and phase properties.

Material examination indicates coarsening and phase segregation is likely in the processing temperature regime encountered for both the extruded and extruded and forged material.

Illustrates a need for detailed characterization of second phase particles
Technical Accomplishments 5
Microstructure near Fe-rich Precipitates

- TEM EDS analysis of the needles identified in the 450°C (10hr) + 500°C(3hr) specimen indicated a composition resembling that of the equilibrium phase Al3Fe with trace impurities of Si, V and Mn.

<table>
<thead>
<tr>
<th>Element</th>
<th>Mass%</th>
<th>Atom%</th>
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<tbody>
<tr>
<td>Al</td>
<td>52.11</td>
<td>68.94</td>
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<tr>
<td>Si</td>
<td>0.61</td>
<td>0.78</td>
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<tr>
<td>V</td>
<td>0.84</td>
<td>0.59</td>
</tr>
<tr>
<td>Mn</td>
<td>1.04</td>
<td>0.68</td>
</tr>
<tr>
<td>Fe</td>
<td>45.4</td>
<td>29.02</td>
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</table>

- Atomic resolution of the Fe-rich precipitate interface (below) shows the region is single phase supporting Al3Fe phase identification.

TEM results have helped to increase understanding of phases that precipitate on heating in this alloy system. Mechanical evaluation of the precipitate material is still needed.
Response to Previous Years Comments

**Reviewer Comment:** “A critical initial performance measure might have been set higher than was actually needed. The industry partner, Cummins, determined that tensile strength of 250 mega Pascals (MPa) was sufficient for the project team’s needs, which was different than the project’s initial objective of 300 MPa.”

**PNNL Response:** The 300 MPa goal was applied in an effort to broaden potential applications of the material beyond air handling, to ultimately be determined by the industry partner.

**Reviewer Comment** “In a follow-up project addition of another research institute or university could be considered to build up the project theory.”

**PNNL Response:** An additional collaboration with UC Riverside was added in FY14-FY15.

**Reviewer Comment** “The project was nearing completion, and that material supply issues should be assessed.”

**PNNL Response:** All Aluminum flake extrusions and forged material needed for project completion have been received by PNNL.
Collaboration & Coordination

► Cummins, Inc. - Principal industry partner, CRADA partner
  Fatigue testing, Cost Analysis, Engine Rig Testing

► Transmet Corporation - Commercial melt spinning and
  processing of rapidly solidified flake

► University of California, Riverside – Technical expert advisor
FY15 Remaining Challenges & Barriers

► Evaluate mechanical properties of Fe-rich phase precipitated during high temperature heat treatment to understand its effect on strength.

► Test low temperature Transmet forged extrusions of Al flake.

► Successfully meet 300MPa/300°C project milestone.

► Perform fatigue testing on best product.

► Complete cost and performance benefit analysis of RS Al flake material.

► Complete project FY15 final project report & finalize and submit publications currently in preparation.
Proposed Future Work

**FY15**
- Perform nanoindentation of needle-like phase to evaluate strength properties compared to that of the surrounding matrix.
- Evaluate low temperature Transmet forgings
  - Tensile (Room Temp & 300°C)
  - Microscopic characterization of low temperature forgings
- Perform elevated temperature fatigue test on best product material
- Finalize and submit publications currently in preparation
- Complete project FY15 final project report

**FY16**
- No work is proposed as 2015 is the final year of funding for this project.
Summary Slide FY14-15

Relevance

• Develop aluminum alloys with enhanced high temperature strength that can be processed using low cost, high volume methods
• Successful completion will result in both weight reductions and improved efficiency of heavy duty diesel engines – in direct alignment with the VT Program

Approach & Accomplishments

► Received large scale production Al flake, extruded and forged material
► Evaluated elevated and room temperature mechanical response
► Showed large scale processing is viable
► Identified reduced strength of forged material
► Performed heat treatment tests on flake to identify possible root causes of strength reduction in the forged material
► Advanced microscopic characterization to evaluate phase transformations

Future Work for FY15

► Evaluate strength properties of Fe-rich phase compared to that of the surrounding matrix.
► Evaluate mechanical response of low temperature Transmet forgings & perform microscopic characterization
► Fatigue Test best production material
► Finalize and submit publications currently in preparation
► Complete FY15 final project report
TECHNICAL BACK-UP SLIDES
Laboratory scale melt spinning flake machine with controlled atmosphere chamber closed (left) and open (right)
Prior evaluation of high temperature aluminum alloys

<table>
<thead>
<tr>
<th>Alloy Designation</th>
<th>Fe</th>
<th>Si</th>
<th>V</th>
<th>Cr</th>
<th>Ti</th>
<th>Mn</th>
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<tr>
<td>Al-12Fe</td>
<td>12.4</td>
<td>2.3</td>
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<tr>
<td>Al-8.5Fe</td>
<td>8.5</td>
<td>1.7</td>
<td>1.3</td>
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<tr>
<td>AFCT</td>
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<td>3.4</td>
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<tr>
<td>AFM-11</td>
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<td>0.9</td>
<td></td>
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<td>AFM-13</td>
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<table>
<thead>
<tr>
<th>Alloy Designation</th>
<th>Extrusion Temperature(°C)</th>
<th>Elastic Modulus (GPa)</th>
<th>Elastic Modulus (GPa) 300°C</th>
<th>Tensile Yield Strength(MPa)</th>
<th>Tensile Yield Strength(MPa) 300°C</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>Failure Strain (%)</th>
<th>Failure Strain (%) 300°C</th>
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<tbody>
<tr>
<td>Al-8.5Fe - EB</td>
<td>450</td>
<td>83.5</td>
<td>74.5</td>
<td>345.0</td>
<td>210.9</td>
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<tr>
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<td>256.8</td>
<td>493.6</td>
<td>7.2</td>
<td>17.0</td>
</tr>
</tbody>
</table>

EB= vacuum electron beam welded can without hot press  HP= vacuum hot pressed billet. Failure strains measured using an extensometer. Notes: yield strength and ultimate strength are the same at the 300 C test temperature. Extension at failure calculated from gage section measurements.
Microstructural features in the room temperature condition

a) Dendrites
b) Angular precipitates in a cellular structure
c) Rosettes.

Production scale flake & lab scale shot material microstructures are compared

**SHOT MATERIAL**

Microstructural features in the room temperature condition

- a) Dendrites
- b) Angular precipitates in a cellular structure
- c) Rosettes.

**FLAKE MATERIAL**

Strong cooling gradients were seen in the flake material producing 4 microstructural zones.

- 1-Nanocrystalline
- 2-Dendritic/Cellular
- 3-Rings
- 4-Lamella/Eutectic Phase

Refined flake microstructure is more likely to achieve higher strength & meet design criteria
• Dark contrast regions are pure aluminum indicating phase segregation
• Forged and forged extrusion microstructures (NOT TENSILE TESTED SPECIMENS) appear similar with the exception of what resembles a needle like phase seen in the forged extrusions