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Rapidly Solidified High Temperature Aluminum Alloys

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Richland, WA USA

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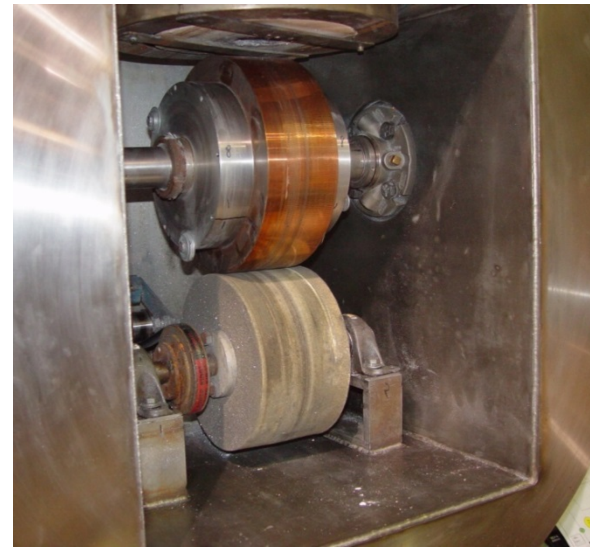
Project ID# PM044

Outline

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

Molten metal enters the Melt Spinner



Rapidly Solidified Flake Material

Hot Pressed → Extruded
Hot Pressed → Extruded + Forged

Project Overview

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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

TRANSMET CORPORATION
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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.

Objectives (3/2014 – 3/2015)	Relevance
Increase the ultimate tensile strength of aluminum alloys at elevated temperature	Higher operational temperatures and weight reductions improve engine performance and fuel efficiency.
Show production method is viable by the successful scale up of rapidly solidified and extruded material.	Direct Cost Savings Mechanical alloying (MA) is too expensive for large-scale commercial applications
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	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 → **Complete (2014)**
  - Flake Material
  - Extrusions
  - Forged Extrusions
- Perform Iterative testing to identify key process parameters → **In Progress (2014)** **on track**
  - Tensile Testing (Room & Elevated Temperature)
  - Failure Analysis & Characterization of tested specimens
- 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

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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

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2014					2015												
Apr	May	Jun	Jul	Aug	Sept	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sept

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

Indicates Go/No-Go Decision Points

***Demonstrate Cummins required B10 service life**

Complete Project Overview 2011-Current	FY11	FY12	FY13	FY14	FY15
Identify Candidate Materials and Processing Methods/Small Scale Production					
Production and Testing of Candidate Alloys & Methods					
Downselect Alloy & Processing Method					
Large Scale Production Continued Testing & Specimen Characterization					
Complete Characterization\Publication & Annual Report Submitted					

Technical Accomplishments I

Evaluation of Production Scale RS Material

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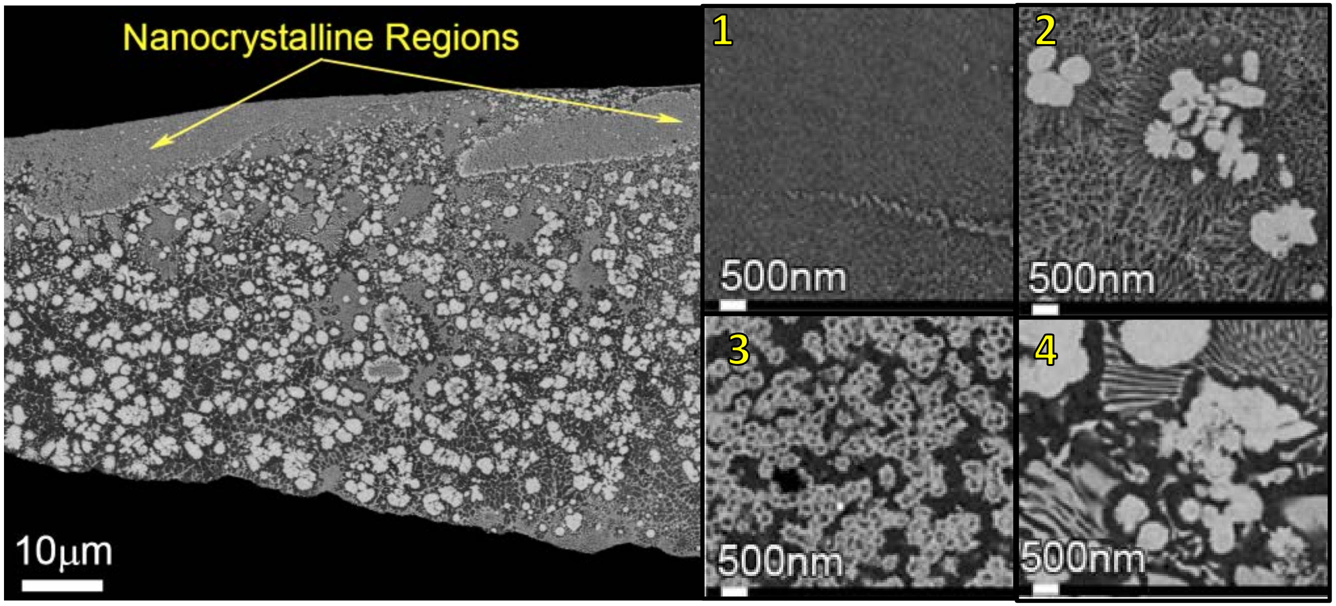


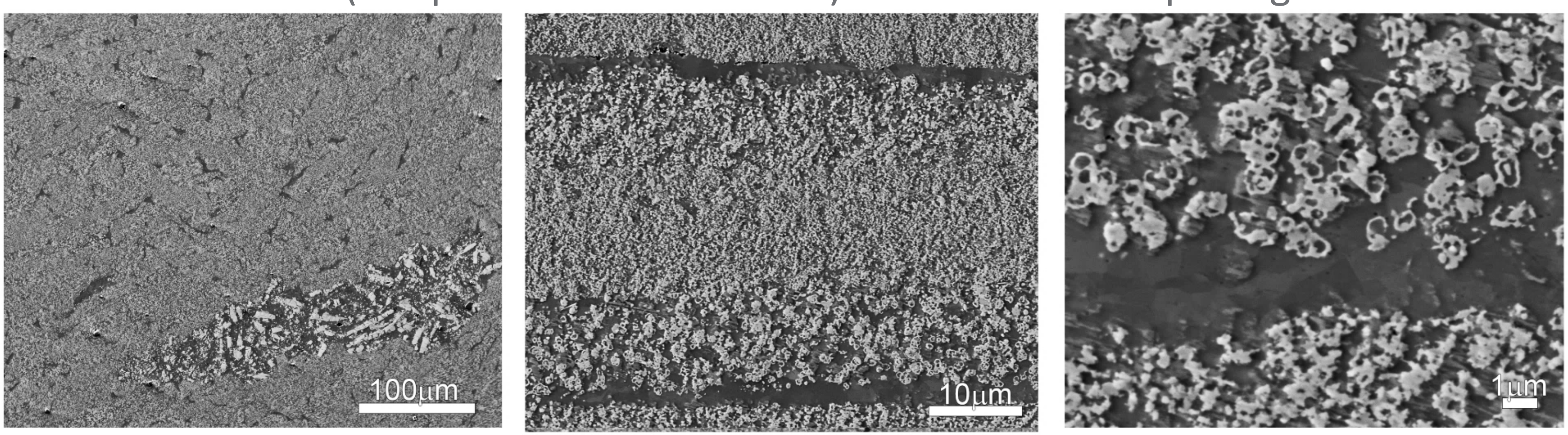
FLAKE MATERIAL

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.

Extruded Flake



Refined flake microstructure is likely to achieve high strength & meet design criteria

Technical Accomplishments 2
Tensile Results

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Small Scale Tensile Testing (Status at Previous Review)

Alloy Designation	Condition	Test Temperature (°C)	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Failure Strain (%)
AFM-11shot	Extruded	Room	287.0	411.2	1.8
AFM-11 shot	Extruded	300°C	185.6	185.6	5.8
AFM-11 flake	Extruded	Room	427.8	493.6	7.2
AFM-11 flake	Extruded	300°C	256.8	256.8	17.0

Small Scale testing led to material down selection

FY14/15 has focused on evaluating RS flake material

FY14-15

Alloy Designation	Condition	Test Temperature (°C)	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Failure Strain (%)
AFM-11 flake	Extruded	Room	544.2	544.2	3.6
AFM-11 flake	Extruded	300°C	278.4	298.0	6.1
AFM-11 flake	Forged Ext.	Room	351.3	360.7	2.7
AFM-11 flake	Forged Ext.	300°C	200.0	202.7	2.1

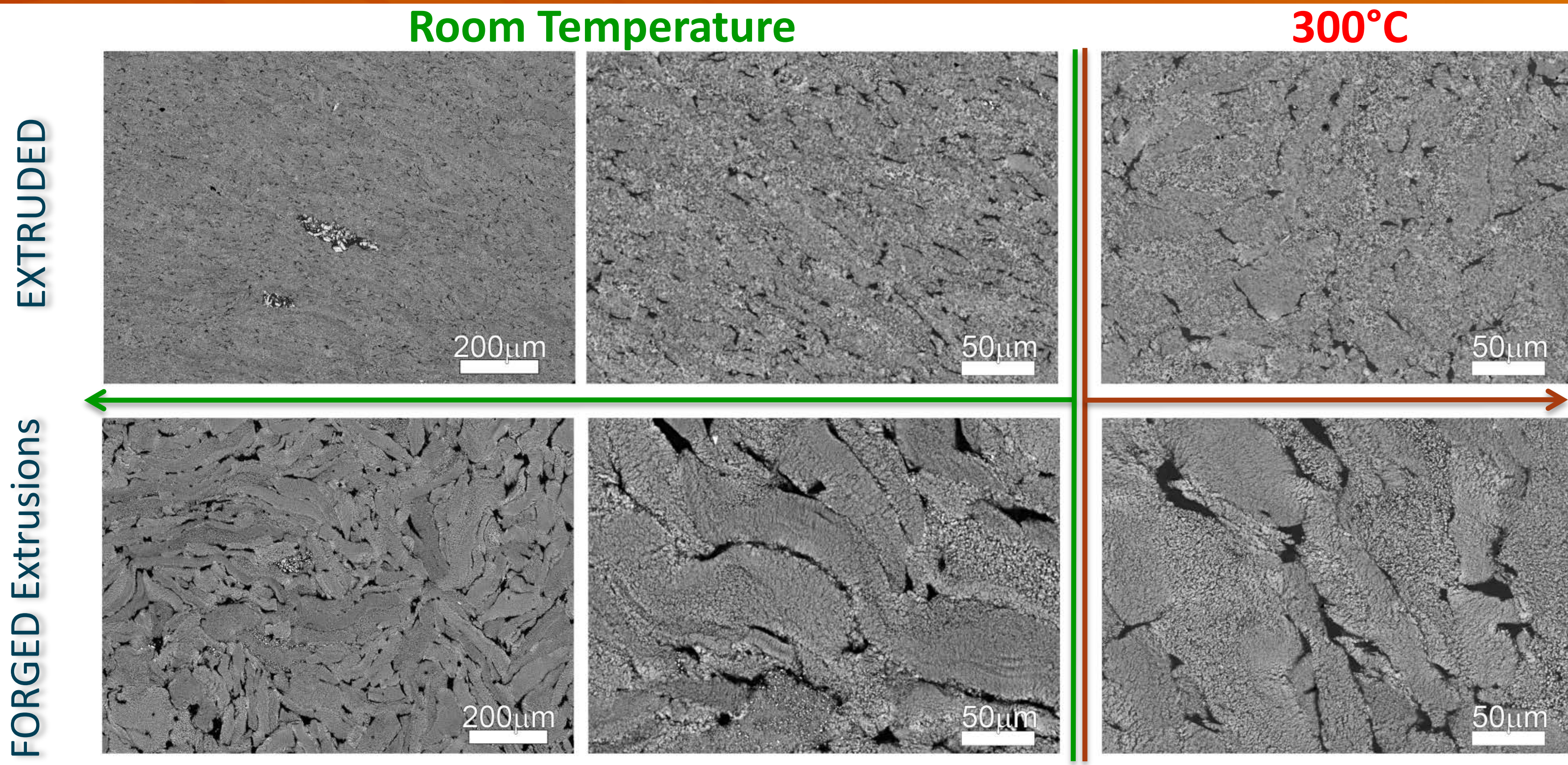
Extruded flake shows higher strength than forged extrusions.

- **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.

Evaluation of Potential Phases Transformations

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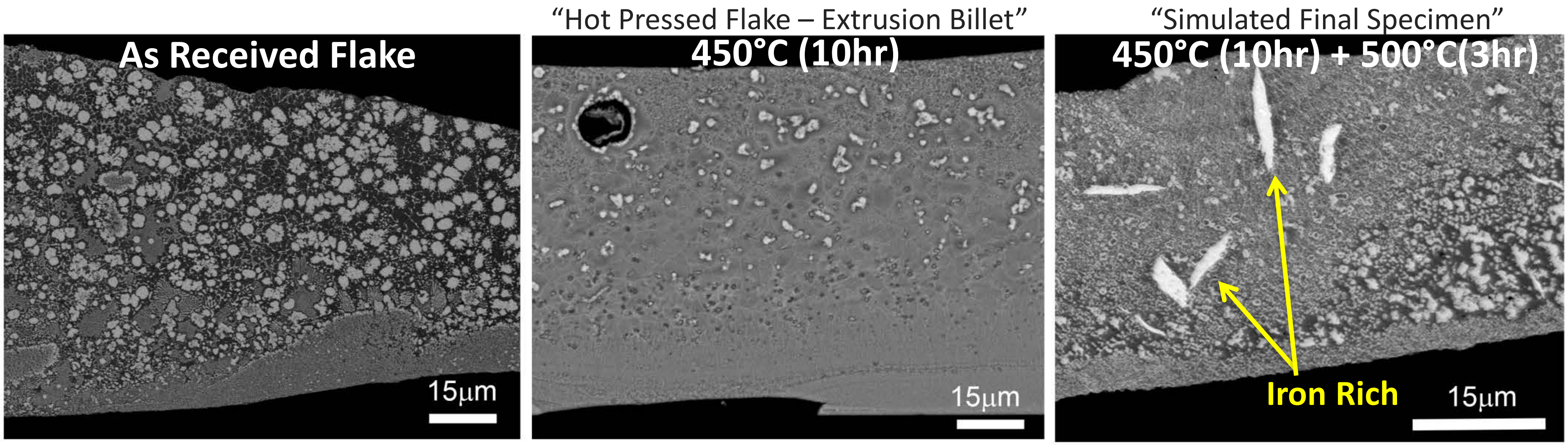
Rapid Solidification is a non-equilibrium process, knowledge of high temperature phase transformations can be used to create fine dispersions of second phase particles that have the potential to increase strength. Growth of large second phase particles can lead to volumetric changes, void formation and decrease strength.

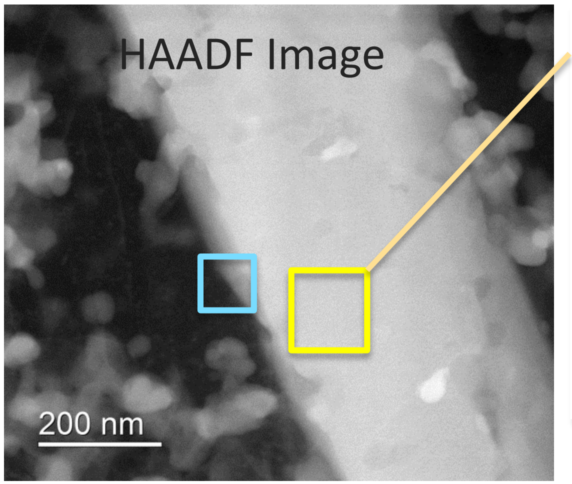


- Heat treatments were performed on flake in the expected processing temperature regime
- Microscopy has indicated new phases have potential to form in the 450-500°C temperature range
- Precipitation of new phases can impact strength positively or negatively depending on coherency and phase properties.

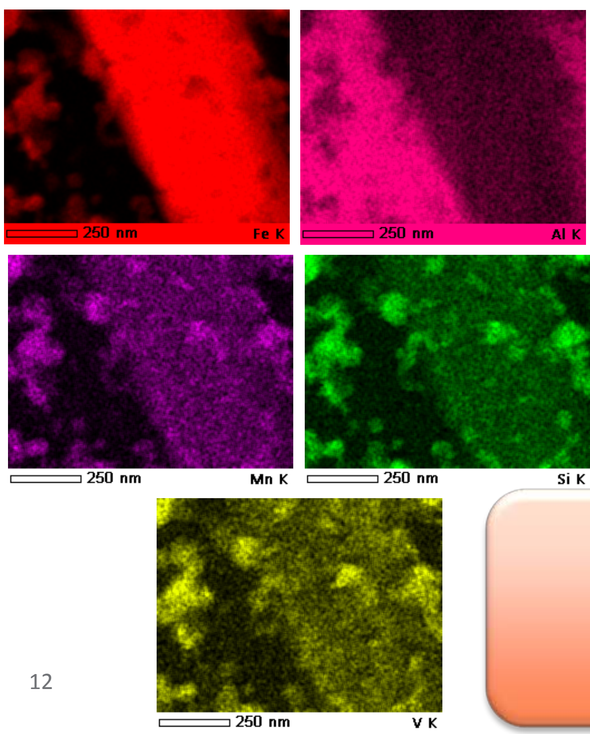
Material examination indicates coarsening and phase segregation is likely in the processing temperature regime encountered for both the extruded and extruded and forged material.

Illustrates a need for detailed characterization of second phase particles

Microstructure near Fe-rich Precipitates

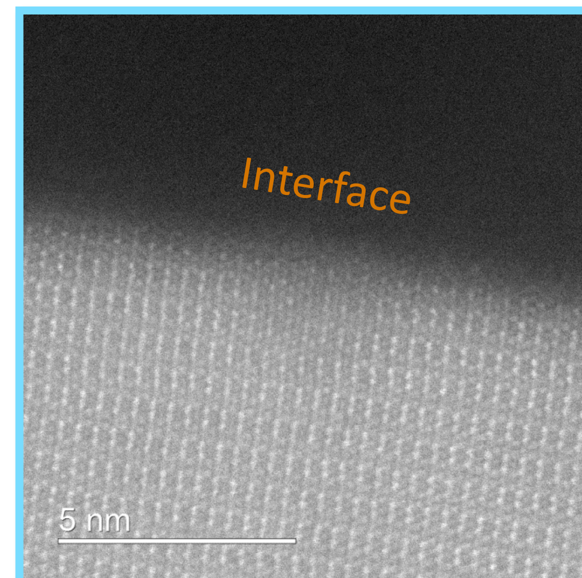


Element	Mass%	Atom%
Al	52.11	68.94
Si	0.61	0.78
V	0.84	0.59
Mn	1.04	0.68
Fe	45.4	29.02



- 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.

- 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

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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."*

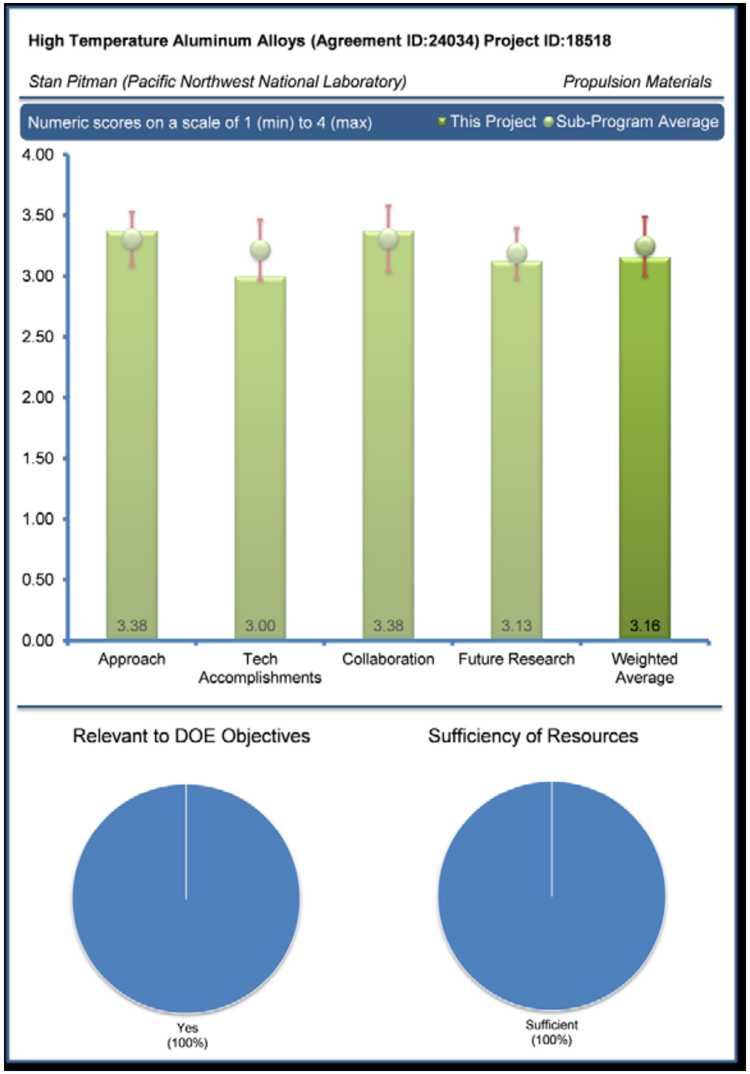
PNNL Response: The 300 MPa goal was applied in an effort to broaden potential applications of the material beyond air handling, to ultimately be determined by the industry partner.

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.

Low temperature forged Al flake material fabricated by Transmet & received

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

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Relevance

- Develop aluminum alloys with enhanced high temperature strength that can be processed using of 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

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Approach & Accomplishments

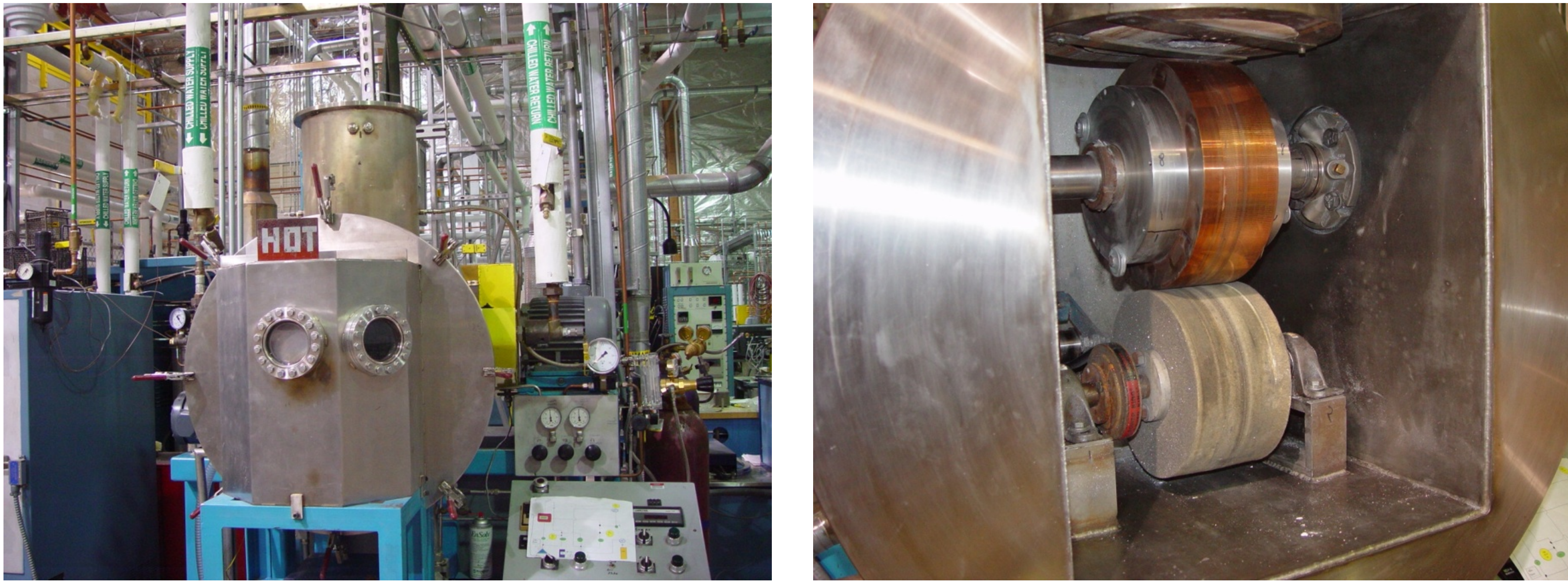
- Received large scale production Al flake, extruded and forged material
- Evaluated elevated and room temperature mechanical response
- Showed large scale processing is viable
- Identified reduced strength of forged material
- Performed heat treatment tests on flake to identify possible root causes 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

Technical Backup Slide I



Laboratory scale melt spinning flake machine with controlled atmosphere chamber closed (left) and open (right)

Technical Backup Slide II

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Prior evaluation of high temperature aluminum alloys

Alloy Designation	Fe	Si	V	Cr	Ti	Mn
Al-12Fe	12.4	2.3	1.2			
Al-8.5Fe	8.5	1.7	1.3			
AFCT	6.0			3.4	3.2	
AFM-11	11.4	1.8	1.6			0.9
AFM-13	13.2	2.6	.50			0.9

Alloy Designation	Extrusion Temperature(°C)	Elastic Modulus (GPa)	Elastic Modulus (GPa) 300°C	Tensile Yield Strength(MPa)	Tensile Yield Strength(MPa) 300°C	Ultimate Tensile Strength (MPa)	Failure Strain (%)	Failure Strain (%) 300°C
Al-8.5Fe - EB	450	83.5	74.5	345.0	210.9	390.4	19.1	25.1
Al-8.5Fe - EB	500	84.2	76.6	331.2	208.2	389.4	18.0	21.7
Al-8.5Fe - HP	500	86.9	71.8	338.1	204.2	384.9	18.3	18.4
AFCT - HP	500	95.9	80.7	400.2	226.7	448.6	12.2	18.8
AFM-11-HP	500	96.0	91.1	427.8	256.8	493.6	7.2	17.0

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.

Technical Backup Slide III

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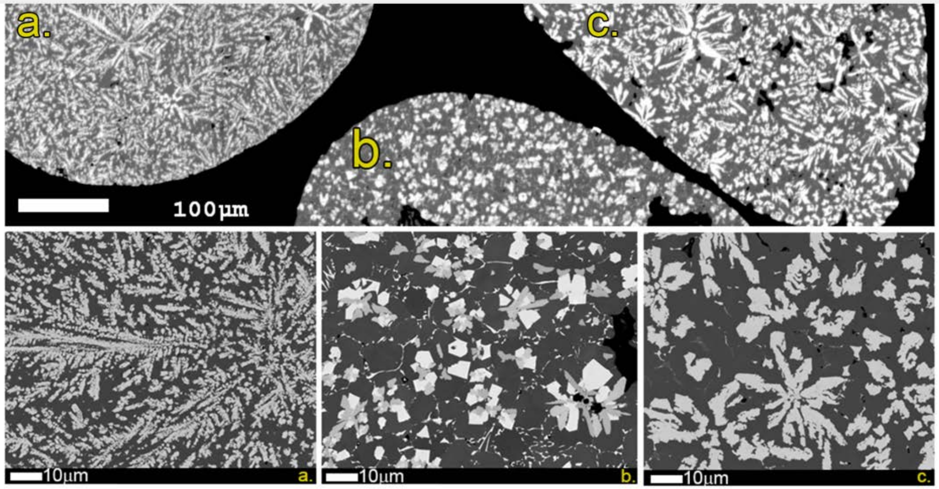
Production scale flake & lab scale shot material microstructures are compared



SHOT MATERIAL

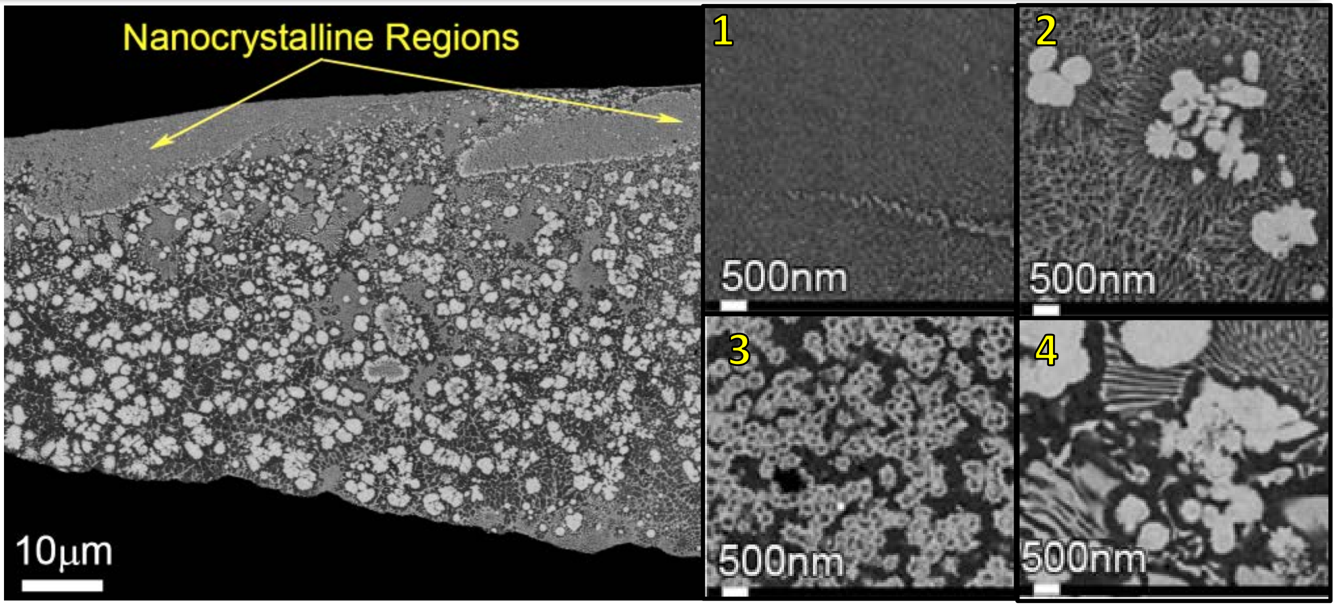
Microstructural features in the room temperature condition

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

Technical Backup Slide IV

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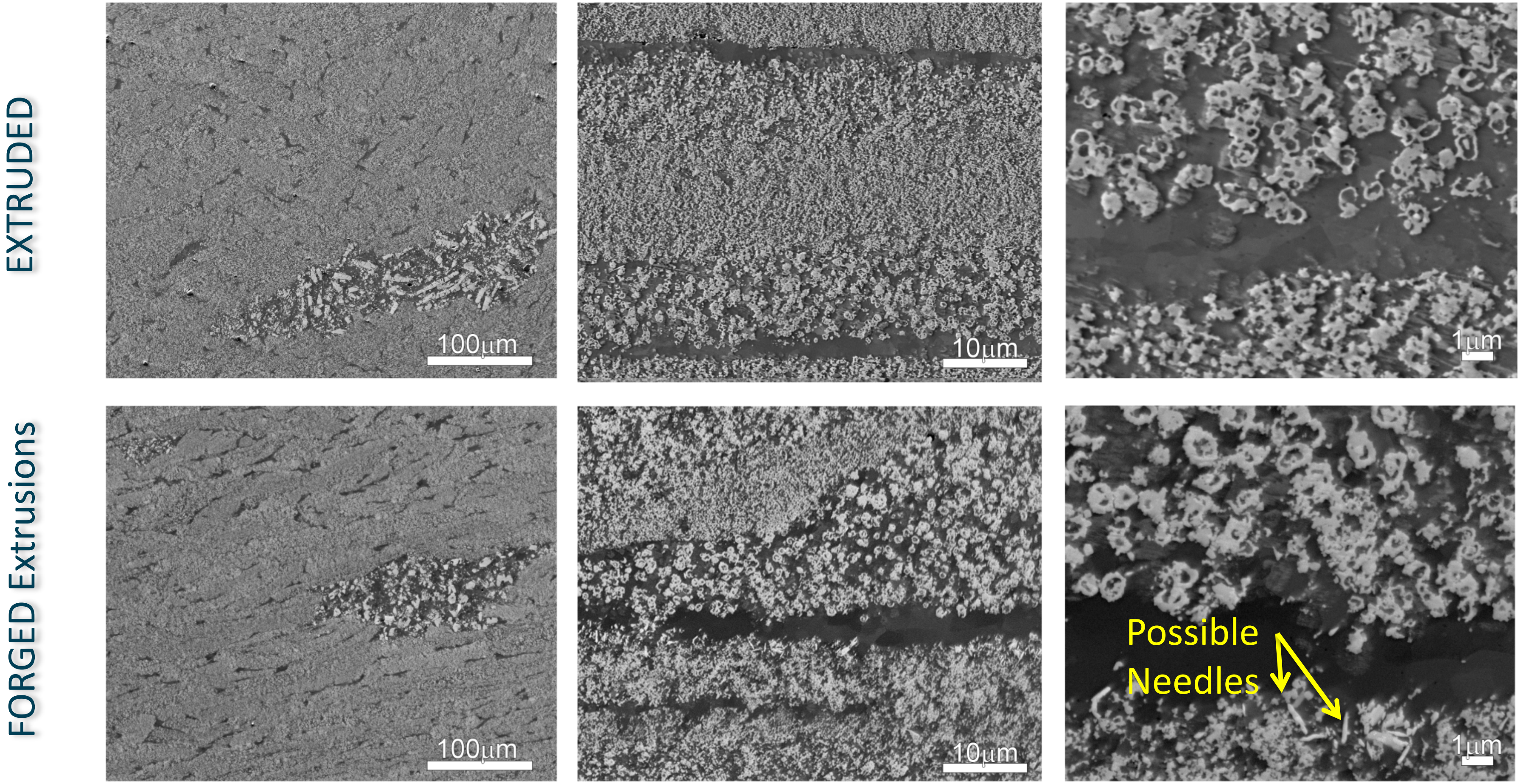

- Dark contrast regions are pure aluminum indicating phase segregation
- Forged and forged extrusion microstructures(NOT TENSILE TESTED SPECIMENS) appear similar with the exception of what resembles a needle like phase seen in the forged extrusions