EWI Project No. 55232GTH
Annual Review
April 19, 2016

Additive Manufacturing of Fuel Injectors

NETL – 2016 Crosscutting Research and Rare Earth Elements Portfolios Review

Prepared by:

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[1]EWI, [2]Solar Turbines Inc.

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Outline

- Project Team
- Motivation
- Objectives
- Project Status
- Project Milestones, Budget, and Schedule as Related to SOPO Tasks
- Project Risks and Risk Management Plan
- Summary

The Project Team

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Shawn Kelly, (PI)
Mahdi Jamshidinia, (Engineer-AM)
Scott Newhouse, (PM)

Solar Turbines

A Caterpillar Company

Daniel Ryan, (PI)
Preston Montague (Materials Technology)
David Teraji, (PM)

NETL

Sydni Credle (PM)

EWI Activities in Metal AM

- **Merge expertise and equipment to solve technical challenges across the AM value chain:**
  - Expertise in AM processes, lasers, materials, NDI, sensing and controls, modeling, and ultrasonics.
- **Process capability in metal AM:**
  - EOS M280, ARCAM A2X, RPM 557
  - Material development, in-process sensing, NDI
  - Design for AM, next generation processes/machines.
- **Other AM process areas:**
  - Arc- and laser- directed energy deposition, ultrasonic AM.
- **Operate the Additive Manufacturing Consortium:**
  - Next meeting at EWI, May 4-5, 2016.

Downhole Drill Bit in 420 SS

4140 Steel

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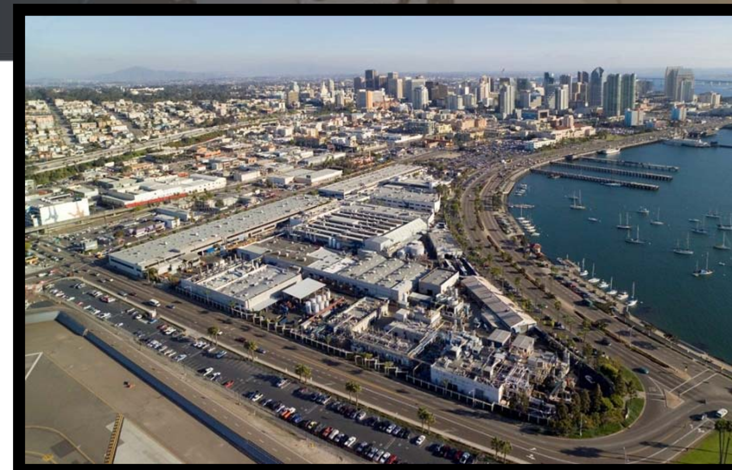
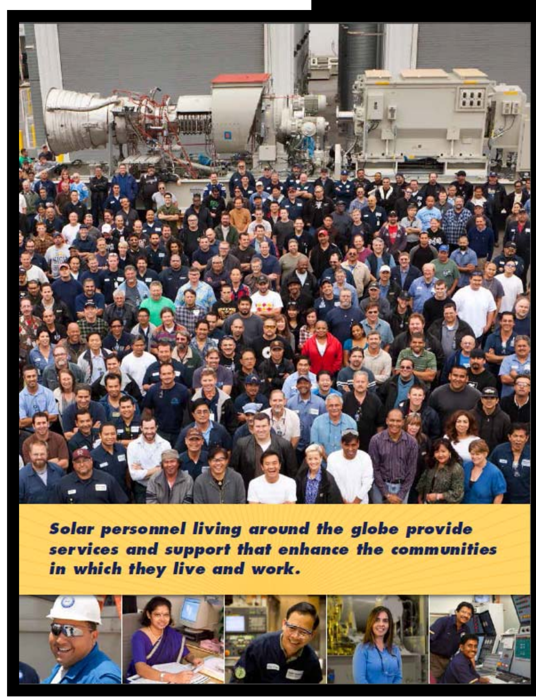
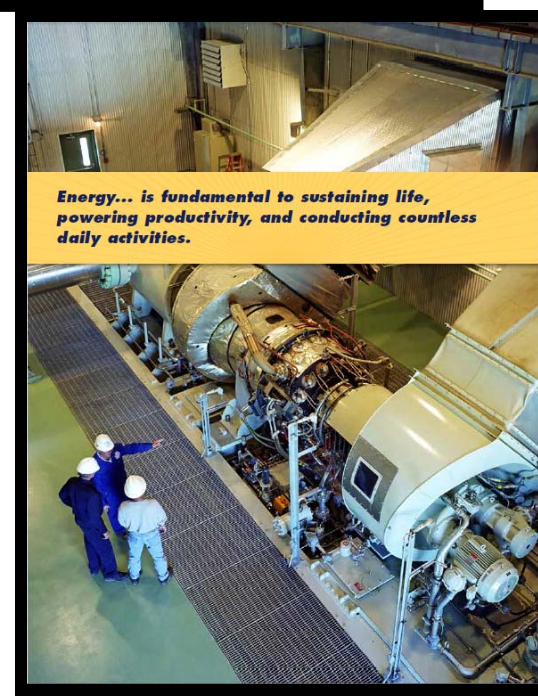
Solar Turbines Overview

- **Headquartered in San Diego, CA**
- **Industrial Gas Turbines**
  - 1.1 to 22 megawatts
  - 1600 to 30,000 horsepower
- **Company Numbers:**
  - 7,000 employees
  - 14,500 units installed
  - 100 countries
  - 2 billion operating hrs.
- **Industries served**
  - Power generation
  - Oil and gas production and transmission







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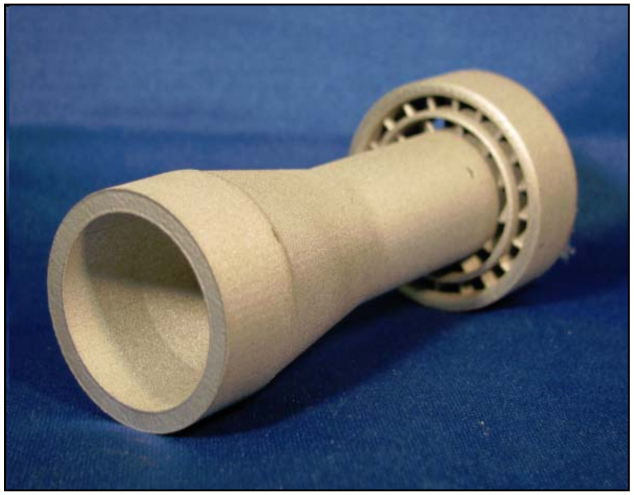
Motivation

- **Gas turbine components:**
  - Very specific design (difficult to cast)
  - Long lead time.
- **Fuel injector tip:**
  - Alloy X
    - Ni-Cr-Fe-Mo alloy
    - Solid solution strengthened.



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Objective

- **Objective:**
  - To develop a novel process to qualify the AM technique of laser powder bed fusion (L-PBF) for complex gas turbine components made of high temperature nickel-based alloys
  - To investigate the effect of input powder stock and AM process variables on resultant microstructure and mechanical properties for the alloy material
  - Post-processing, including heat treatment and the use of finishing technologies will also be employed in order to achieve required dimensional and surface finish requirements for the component.

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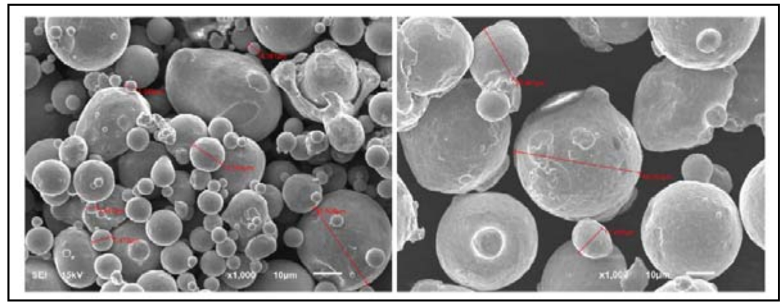
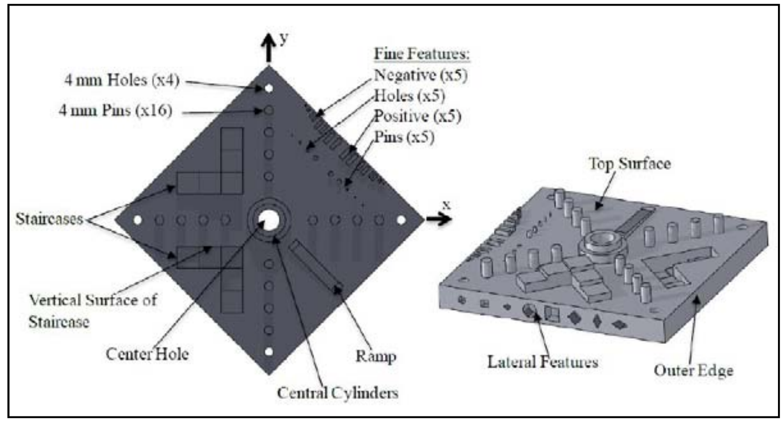
Powder Evaluation

- **Powder sensitivity:**
  - Powder characterization
  - Produce and characterize initial metallography and mechanical property.
- **Test artifact:**
  - Geometric accuracy
  - Surface finish
  - Geometry impact on metallurgy.





Define Powder Specification

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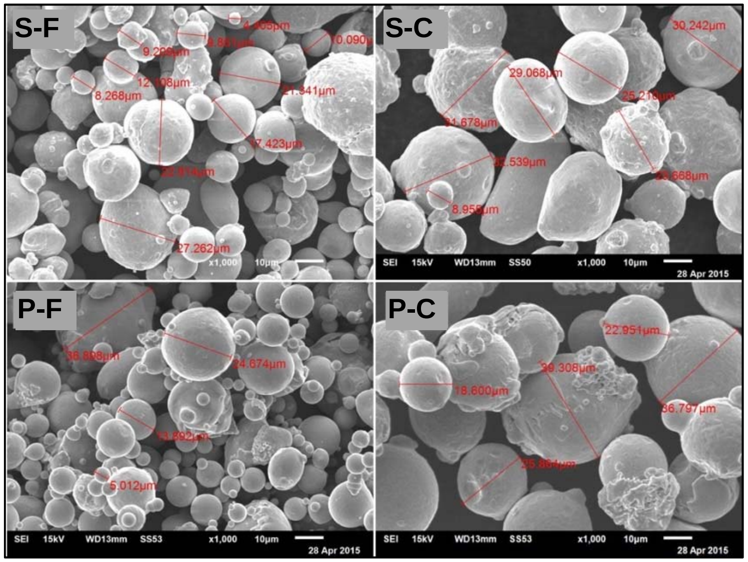
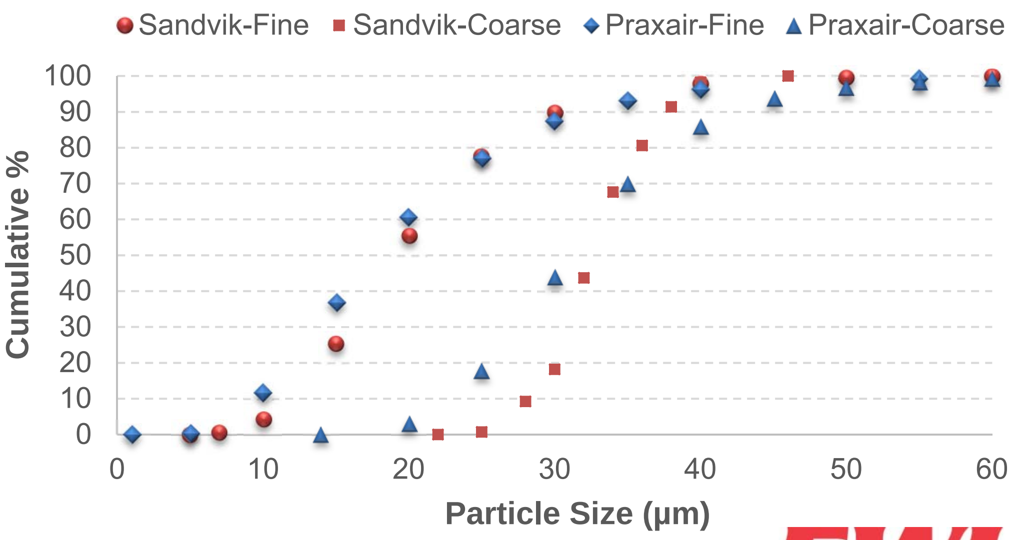
Task 1

- **Powder evaluation:**

Vendor	Type	Min. Desired Size (μm)	Max. Desired Size (μm)	Fine (%)	Coarse (%)	Cost Comparison per lb. (350 lb order)
Sandvik	Fine	5	38	0.1% < 5 um	0.8% > 38 um	100%
Sandvik	Coarse	20	45	4.2% < 20 um	0.5% > 45 um	132%
Praxair	Fine	5	38	2% < 5.5 um	1 > 38 um	190%
Praxair	Coarse	16	45	1% < 16 um	1% > 45 um	195%





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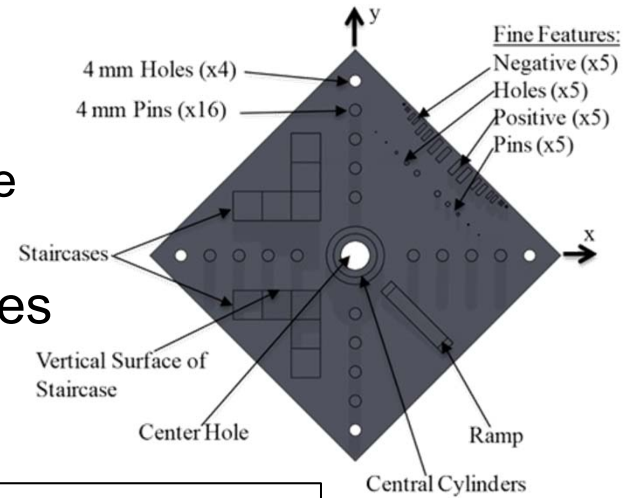
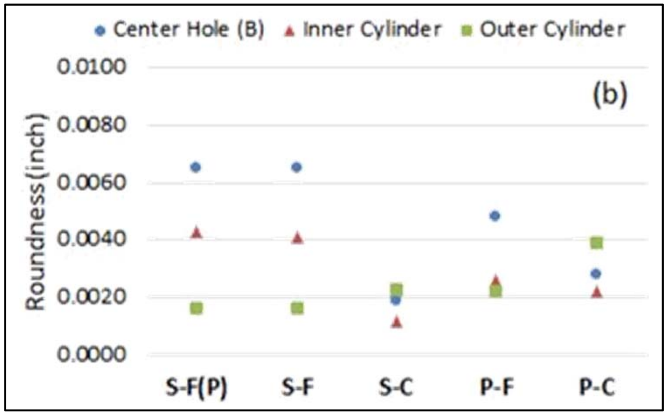
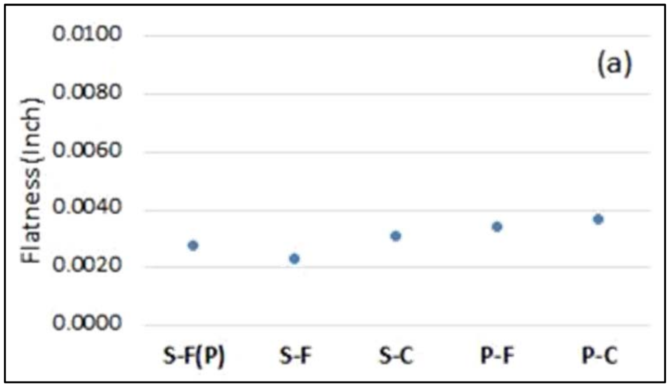
Task 2

- **Dimensional inspection (stereoscopy and CMM):**
  - NIST test artifact, made with the four powders
    - S-F (P): before removal from the build plate
  - The L-PBF process was capable of producing fine features, and met capabilities of investment casting
    - Further improvement in Task 3.







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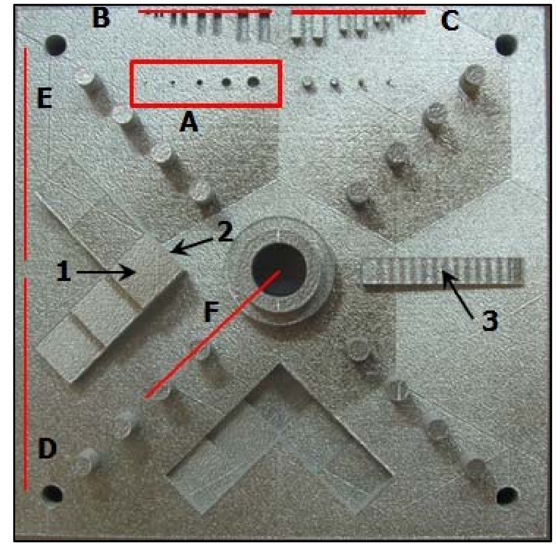
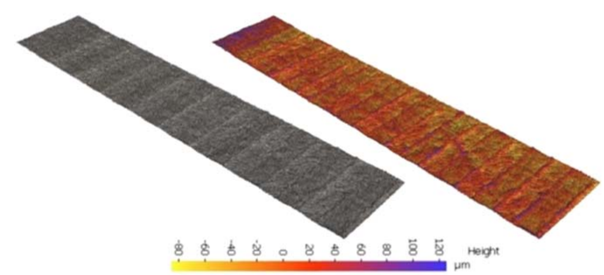
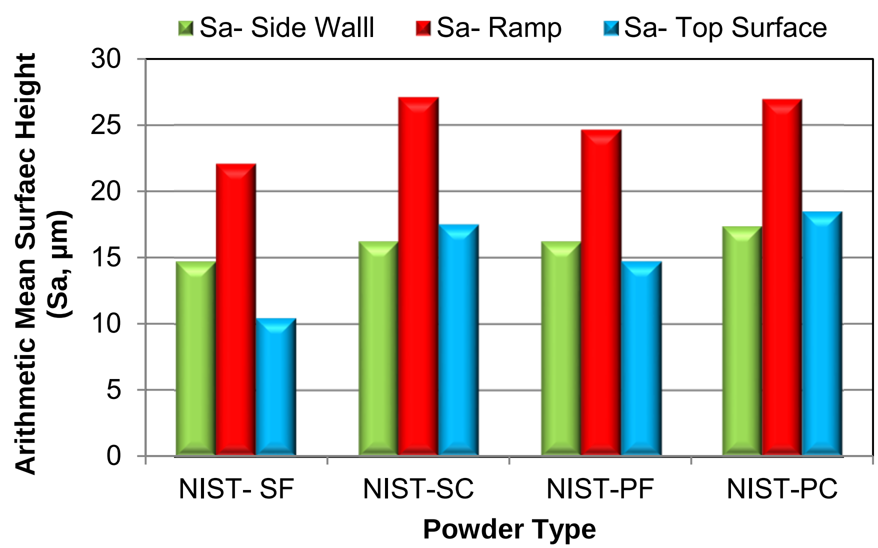
Task 2

- **Surface roughness measurement:**
  - Alicona IF Sensor R25 machine
    - Fine powders were slightly better (S_a).
    - Typical allowable limit for the surface finish of investment casting
      - (Ra) less than 125 µin. (3.17 µm)
  - Further optimization in Task 3.







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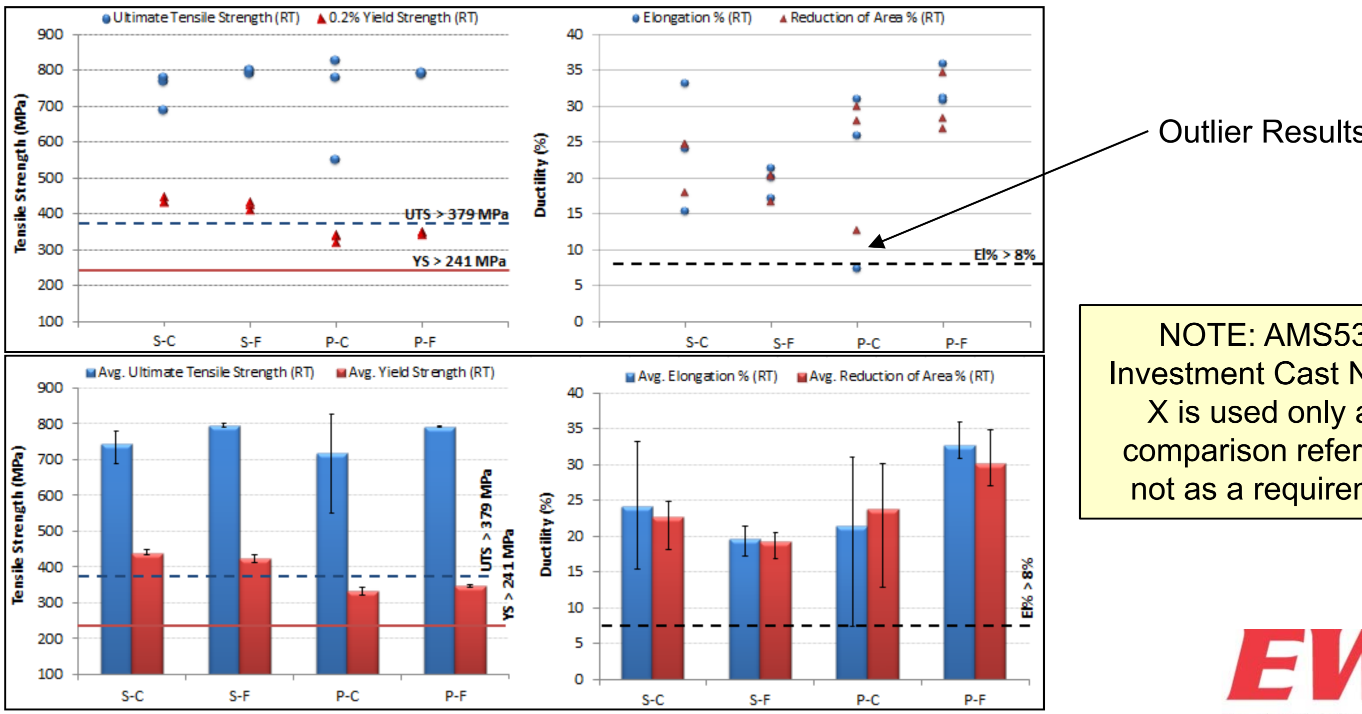
Task 2

- **Stress relief heat treatment:**
  - All of the specimens underwent the stress relief heat treatment, while still attached to the build plate
    - 2150°F
    - 1 hour
    - Rapid argon cooling.

- **Mechanical test:**
  - Tensile
    - Room temperature
    - Elevated temperature (1500°F/815.5°C)
  - Creep
    - Elevated temperature (1500°F/815.5°C)
  - Low cycle fatigue
    - Elevated temperature (1000°F/538°C).

Task 2

- **Tensile test:**
  - Room temperature
    - Three tensile specimens: ASTM E8-09, specimen 3
    - Almost all powders meet the requirements of AMS 5390.



NOTE: AMS5390 Investment Cast Ni Alloy X is used only as a comparison reference, not as a requirement.

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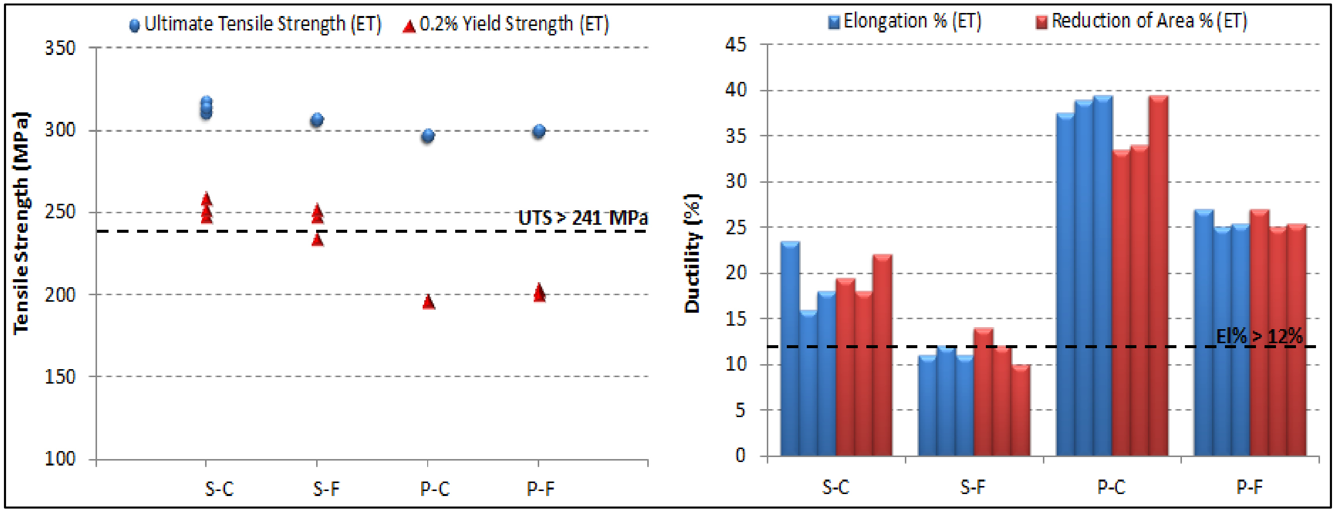
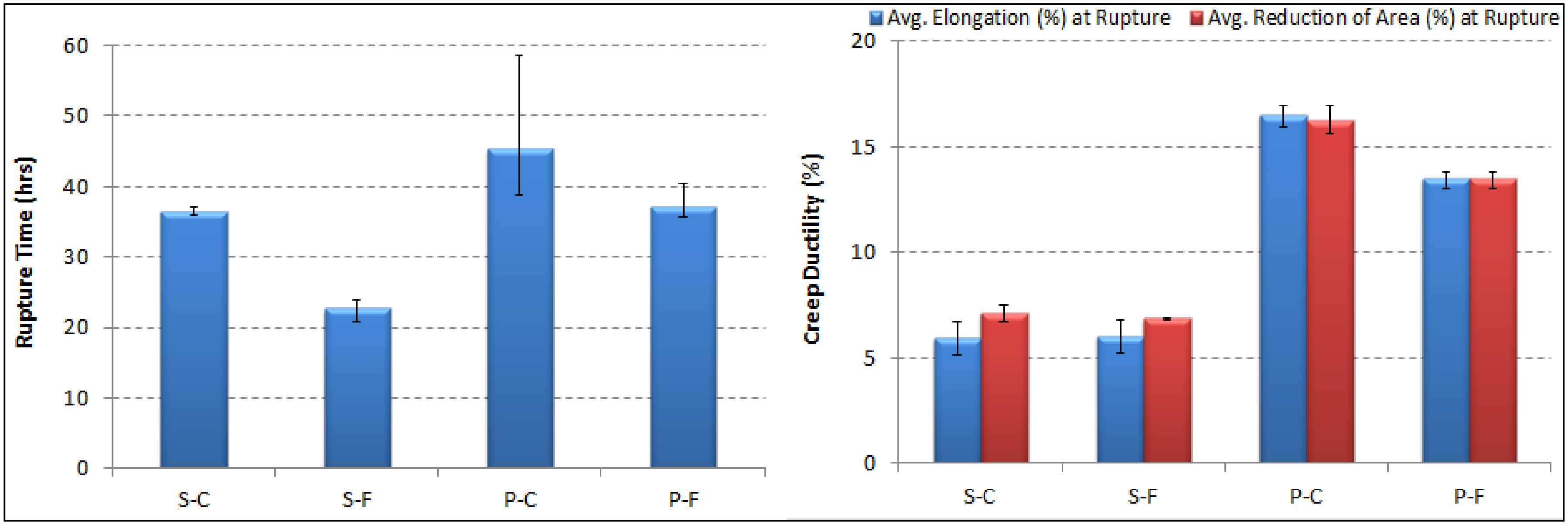
Task 2

- **Tensile test:**
  - Elevated temperature
    - Sample preparation
      - Three tensile specimens: ASTM E8-09, specimen 3
      - 20 minutes of soak time
    - S-F had a slightly lower average ductility (11.33%).



Task 2

- **Creep test:**
  - 1500°F (815.5°C), under a 15-ksi stress
    - Praxair powders had longer rupture times, with a higher ductility
    - S-F had the shortest rupture time.



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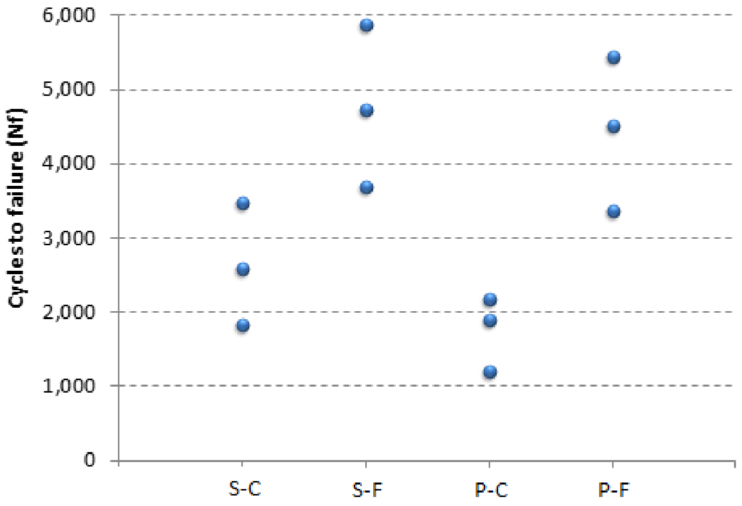
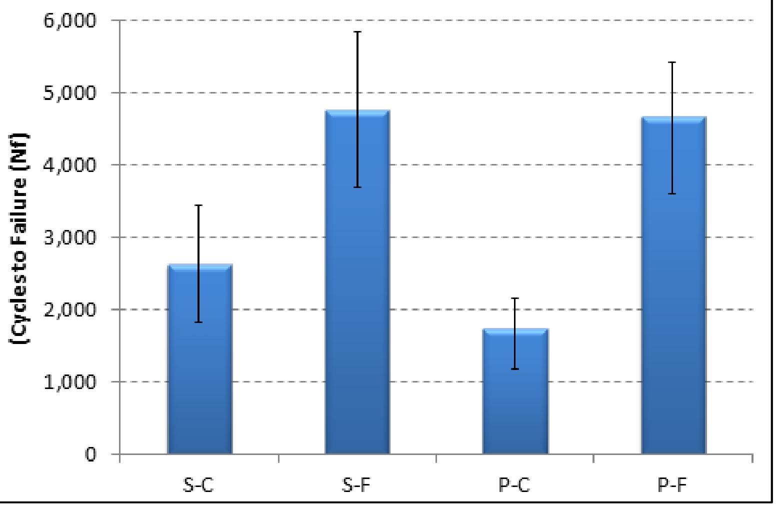
Task 2

- **Fatigue test:**
  - 1000°F (538°C) with a sinusoidal waveform
  - Total strain range and stress ratio of 0.6% and -1, respectively
  - S-F showed the longest average fatigue life
    - Fine grains
    - Precipitates (intergranular and transgranular).





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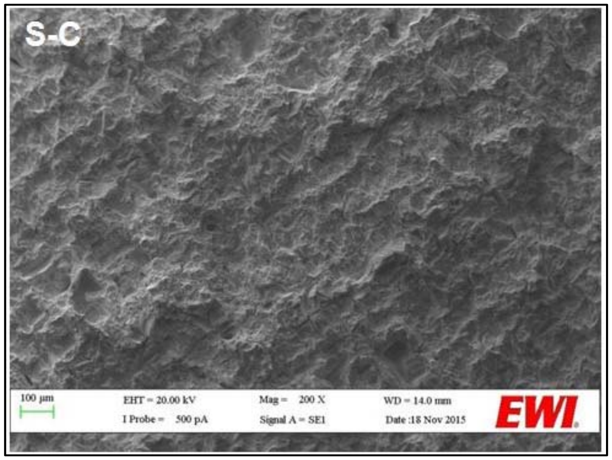
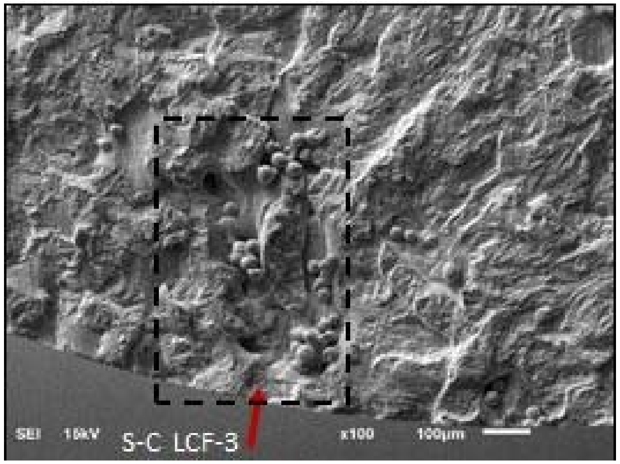
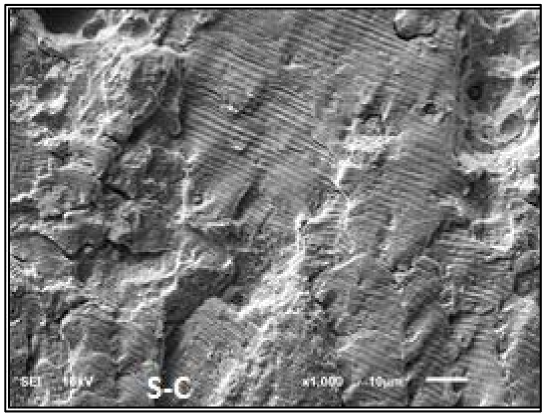
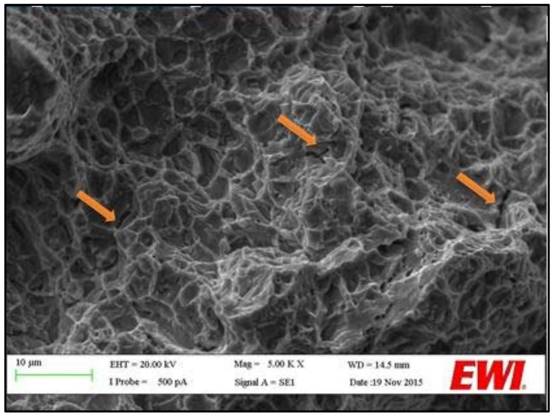
Task 2

- **Fractography:**
  - Room/Elevated temperature tensile test
    - Intergranular fracture morphology
    - Dimples on the fracture surfaces
  - Fatigue
    - Crack initiation at LOF
    - Striation (crack propagation).









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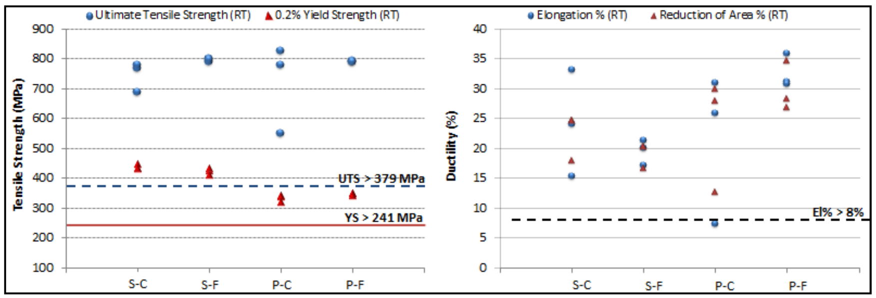
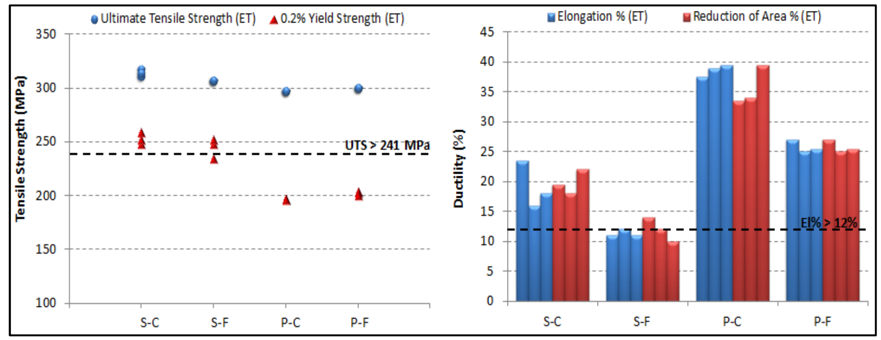
Task 2

- **Mechanical test.**

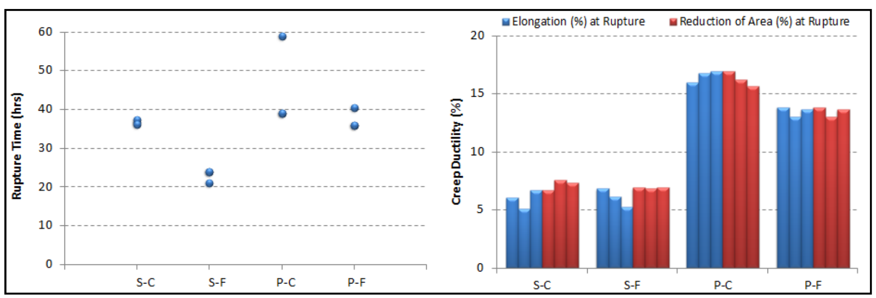
RT-Tensile



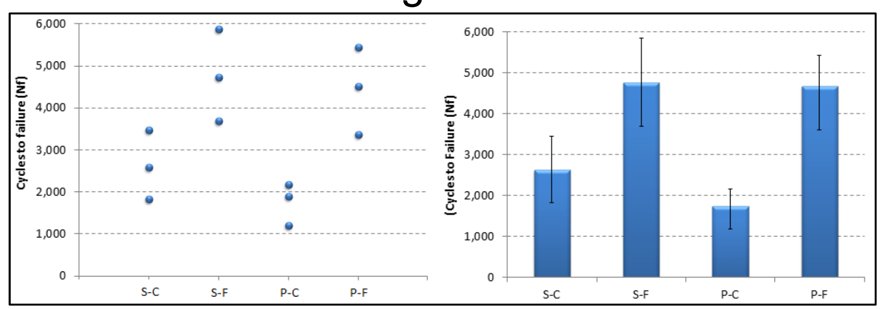
ET-Tensile



Creep



Fatigue



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Task 2

- **Powder down select:**

Powder / Properties	Cost Comparison	RT- Tensile Test			ET- Tensile Test		Creep		Fatigue
		UTS	YS	El%	UTS	El%	Hrs (rpt.)	El% (rpt.)	
S-C	100%								
S-F	132%					No HT			
P-C	190%								
P-F	195%								

- Sandvik powders
  - S-C:
    - Poor creep and fatigue properties
    - Powder leakage
  - S-F
    - Low ductility at high temperature as well as the short creep rupture time could be improved using a proper heat treatment
    - Highest fatigue cycles
    - Originally developed with the OEM.

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Task 2

- **Powder down select:**

Powder / Properties	Cost Comparison	RT- Tensile Test			ET- Tensile Test		Creep		Fatigue
		UTS	YS	El%	UTS	El%	Hrs (rpt.)	El% (rpt.)	
S-C	100%								
S-F	132%					No HT			
P-C	190%								
P-F	195%								

  - Praxair powders
    - P-C
      - Shortest fatigue life
    - P-F
      - Similar or better tensile properties than those of P-C
      - Longer fatigue life

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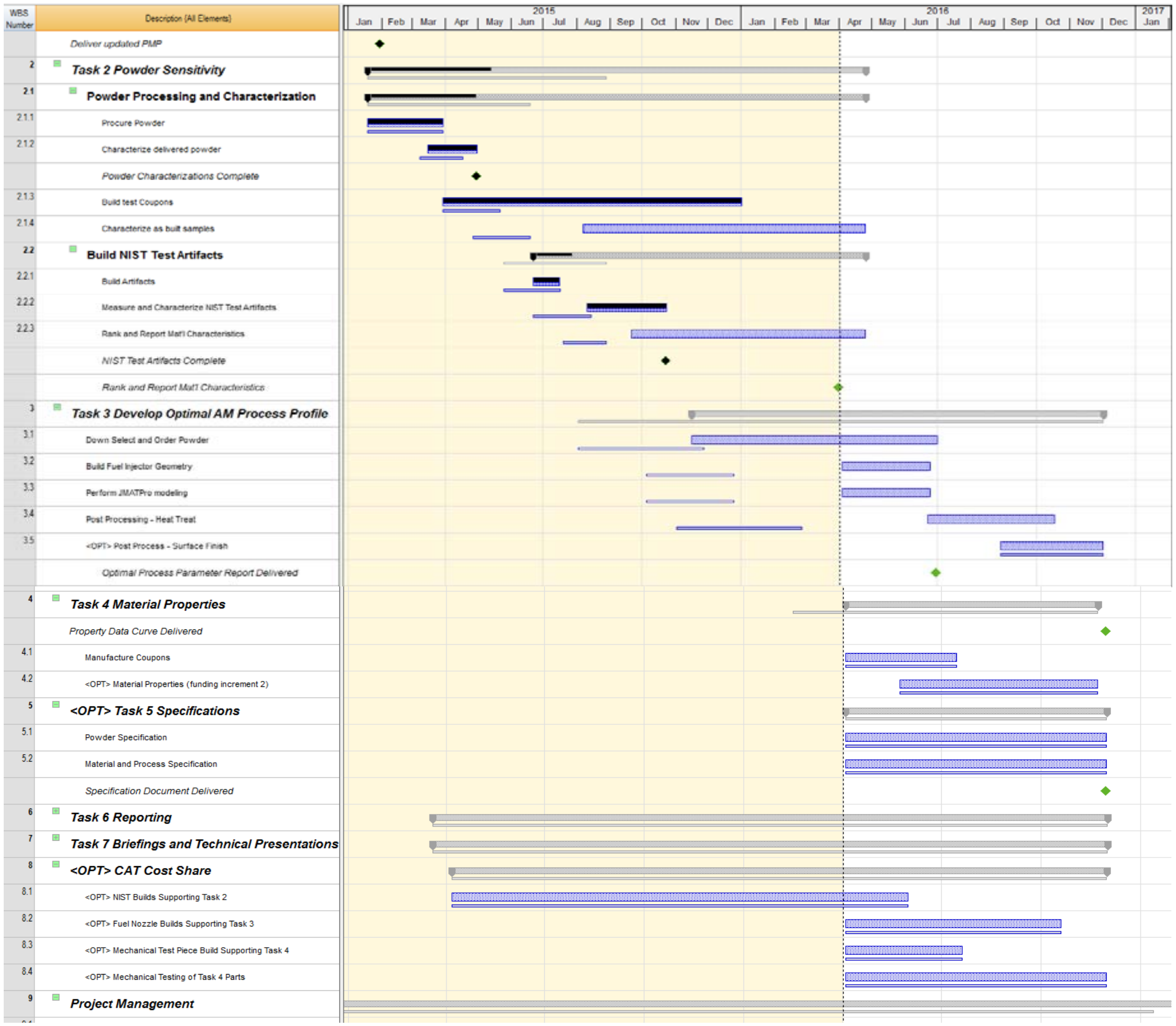
Schedule



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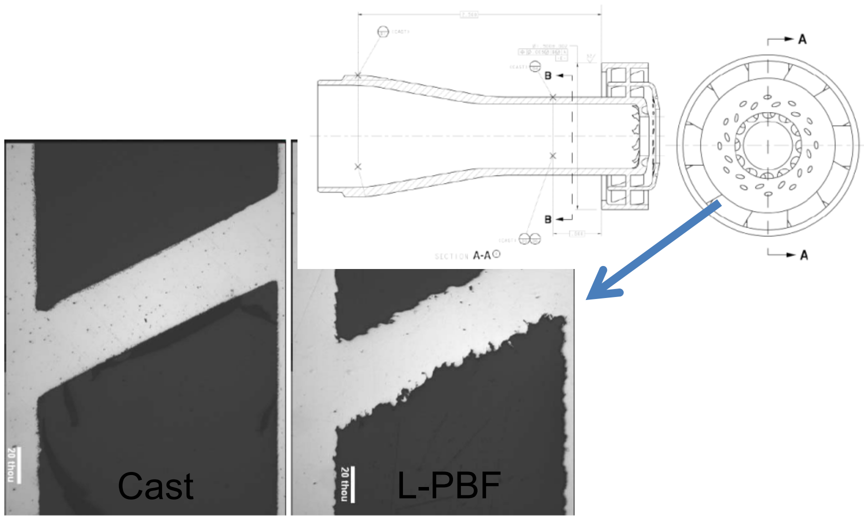
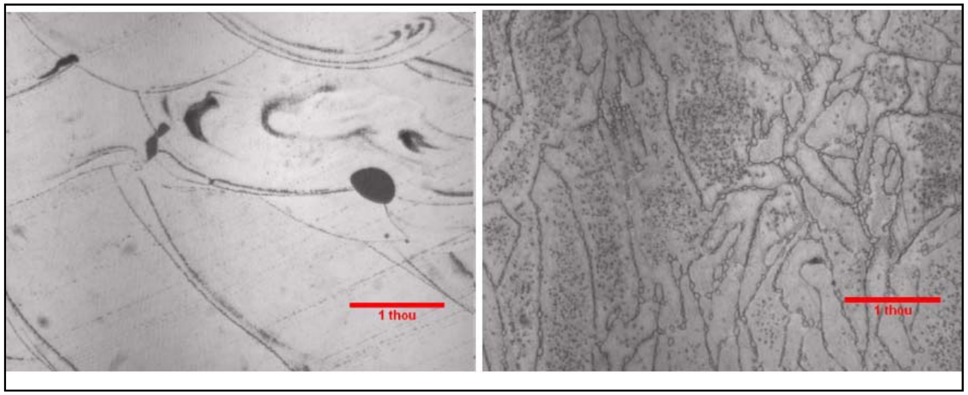
Development of Manufacturing Steps

- **Finishing:**
  - Develop test specimen
  - Determine effect of finishing process on critical features
  - Identify and test at existing providers (Mikrotek, Extrude hone).
- **Heat treatment development:**
  - Determine optimal heat treatment
  - Optimize grain size, aspect ratio, and carbides.
  - Guided by thermodynamic modeling (JMat Pro)





Define Heat Treatment and Finishing Requirements

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Developing Design Curves

- **Down select heat treatment and create additional design data curves.**

Test Type	Task 2.1: Powder Sensitivity		Task 3.1: Parameter Sensitivity		Task 3.2: HT Sensitivity		Task 4: Design Data Curves	
	Qty	Test Conditions	Qty	Test Conditions	Qty	Test Conditions	Qty	Test Conditions
Tensile - RT	3	75F	3	75F	3	75F	5	75F
Tensile - ET	3	1500 F	3	1500 F	3	1500 F	18	800F, 1200F, 1350F, 1500F, 1650F, 1800F
Creep	3	100 hr	3	100 hr	3	100 hr	30	10 hr, 100hr, 1000hr, 5000hr
LCF	3	1000F SR: 0.6	3	1000F SR: 0.6	3	1000F SR: 0.6	20	500F, 800F, 1200F SR: 0.4, 0.6, 0.8, 1.0

- **Generate process documentation:**
  - Powder spec
  - Process spec.

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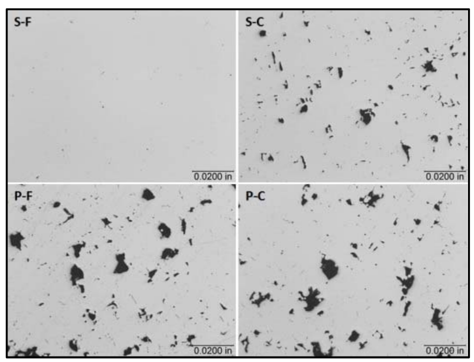
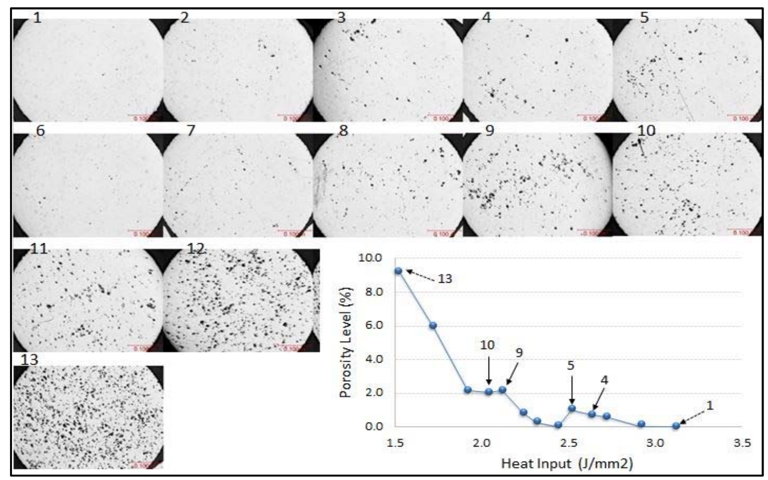
Project Technical Risk Management

- **Challenges:**
  - Incomplete spreading of powder (short-feed)
  - Un-optimized process parameters
  - Powder leaking through seal (S-C powder)
  - Excessive porosity due to the poor sealing
  - Cracking





Project Technical Risk Management

- **Challenges:**
  - Cracking
    - Potential causes
      - Geometry
      - Process parameters
      - Chemical composition
    - Possible solutions (Task 3)
      - Hot Isostatic Pressing (HIP)
      - Further process optimization
      - Chemical composition optimization (out of the scope)
      - …
  - The team will discuss the possible options with DOE/NETL.

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Summary

- **EWI and Solar Turbines met the requirements for Milestone 3.**
- **Two powders were down selected for the further development in task 3.**
  - Sandvik-Fine (5-38 µm)
  - Praxair-Fine (5-38 µm)
- **Lessons Learned:**
  - Change in powder size and supplier requires screening and process optimization prior to implementation. It can affect AM material density and also AM machine compatibility
  - PSD affects grain size and secondary particle distribution. These consequently affect LCF & creep behavior.
  - Powder chemistry apparently affects micro-cracking behavior. Additional investigation and optimization is needed.

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Questions

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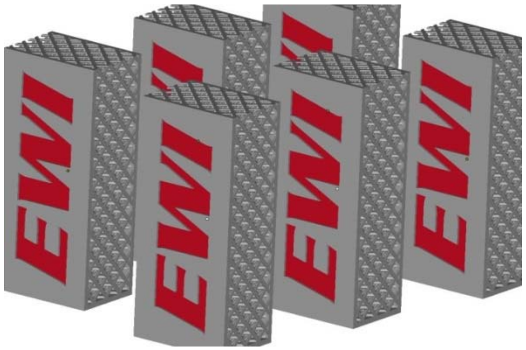

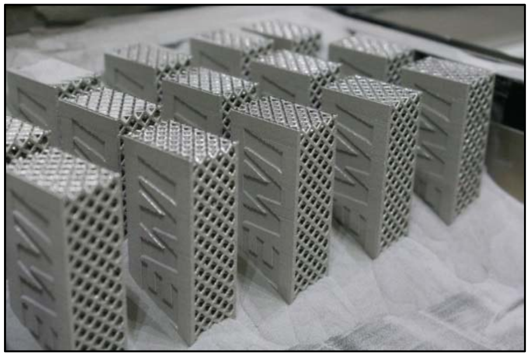

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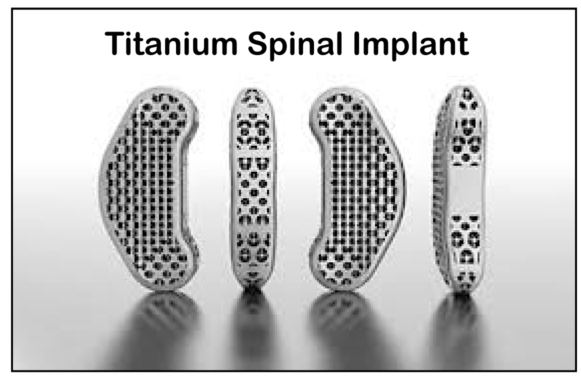
Laser Powder Bed Fusion

- **Enables complex 3D shapes:**
  - Internal passages for cooling, light-weighting.
- **Properties comparable to conventional** (depending on alloy and heat treatment, and surface condition).
- **Challenges:**
  - Building on non-planar surfaces
  - Composition grading.



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- **Potential Significance**
- Relevance to Fossil Energy
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Significance of Work

- **Process sensitivity to powder metal input stock:**
  - Material properties, geometric limitations, and surface finish.
- **Process parameter sensitivity evaluation:**
  - Heat treatment optimization, geometric effect on properties and surface finish.
- **Generate material properties design data curves for high temperature turbine applications:**
  - Necessary to support applications in FE for Alloy-X.
- **Material and process specifications that will enable standardization and quality:**
  - Powder specification
  - Critical manufacturing process characteristics that must be controlled for acceptable quality.

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Relevance to Fossil Energy

- **Alloy-X is used in many industrial gas turbine applications.**
- **AM will enable design and energy efficiencies:**
  - Faster and less costly design optimization.
  - Future applications could enable more energy efficient designs by reducing design constraints
    - Increasing fuel efficiency
    - Providing higher operating temperature

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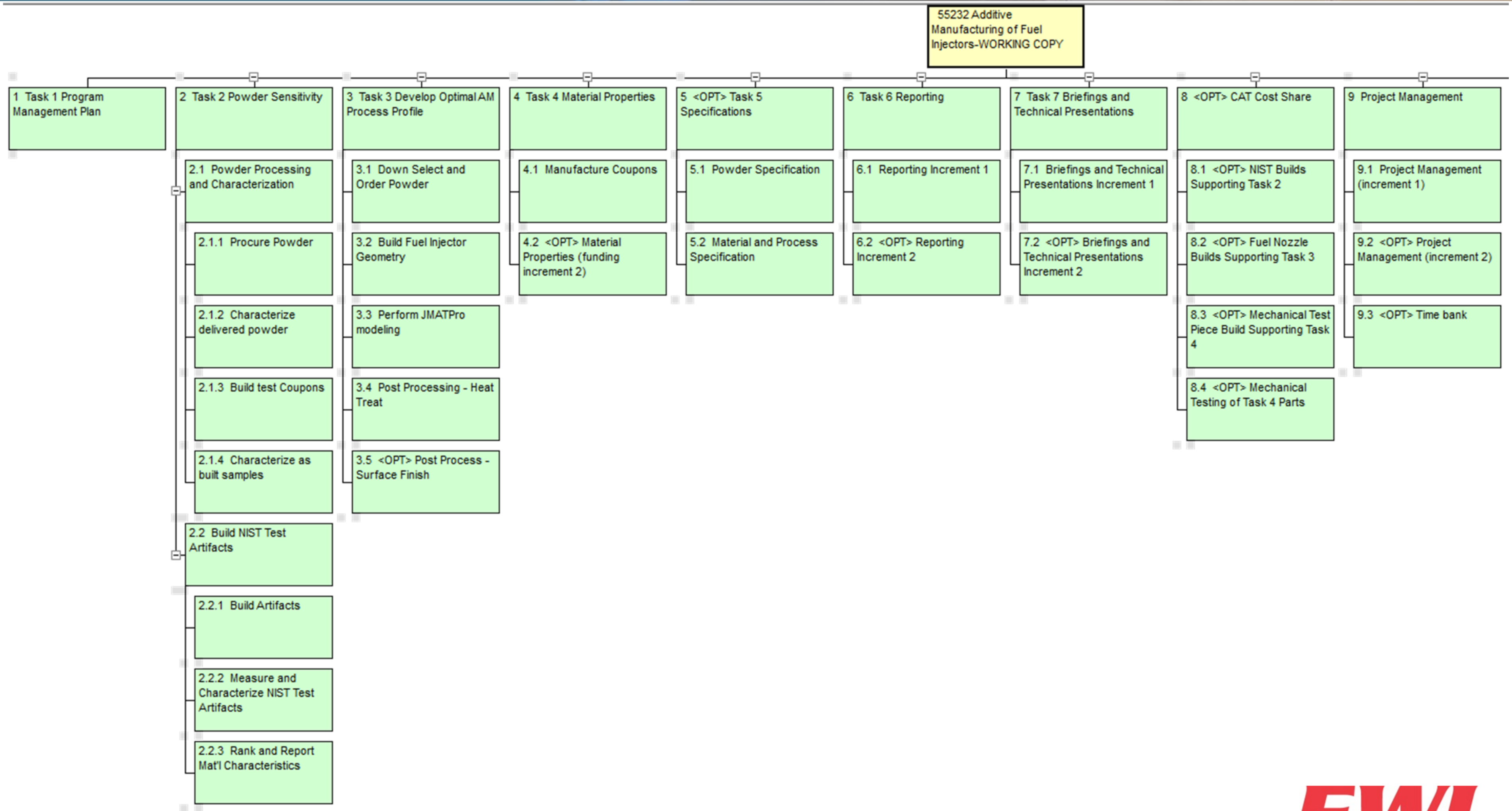

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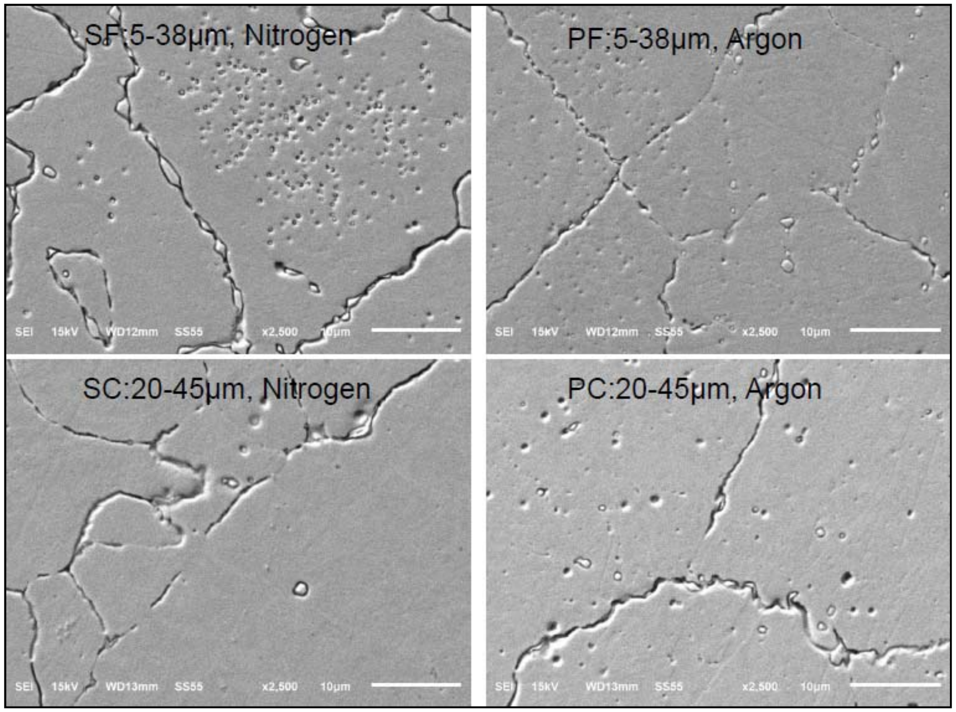
Task 2

- **Stress relief heat treatment:**
  - All of the specimens underwent the stress relief heat treatment, while still attached to the build plate
    - 2150°F
    - 1 hour
    - Rapid argon cooling.

Task 2

- **Metallography:**
  - S-F
    - Fine grains
    - Intergranular and intragranular precipitates.



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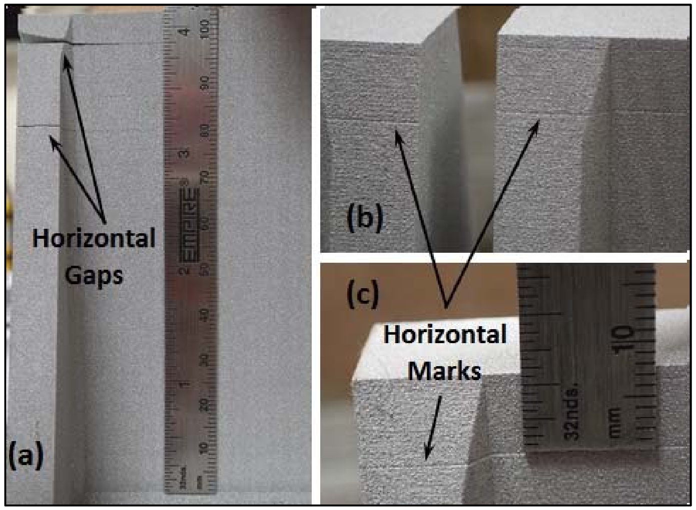
Project Technical Risk Management

- **Mechanical properties do not meet requirements:**
  - Current tensile strengths are acceptable, but low ductility
    - Address with powder and heat treatment definition.
- **Surface finish does not meet as-cast requirements:**
  - Current as-built surface finish does not meet as cast
    - Address with powder selection and post-process finishing as required.
- **Equipment availability:**
  - 3DS ProX 300 installed and operational
  - Regional supplier as backup.

Project Technical Risk Management

- **Challenges:**
  - Incomplete spreading of powder (short-feed)
    - Failure of a pin-pad locator for the powder spreader mechanism
    - The ASTM E8-09 Specimen 3 with a reduced length of 1.25 in. was used
  - A new pin-pad assembly was made from a wear-resistant alloy.



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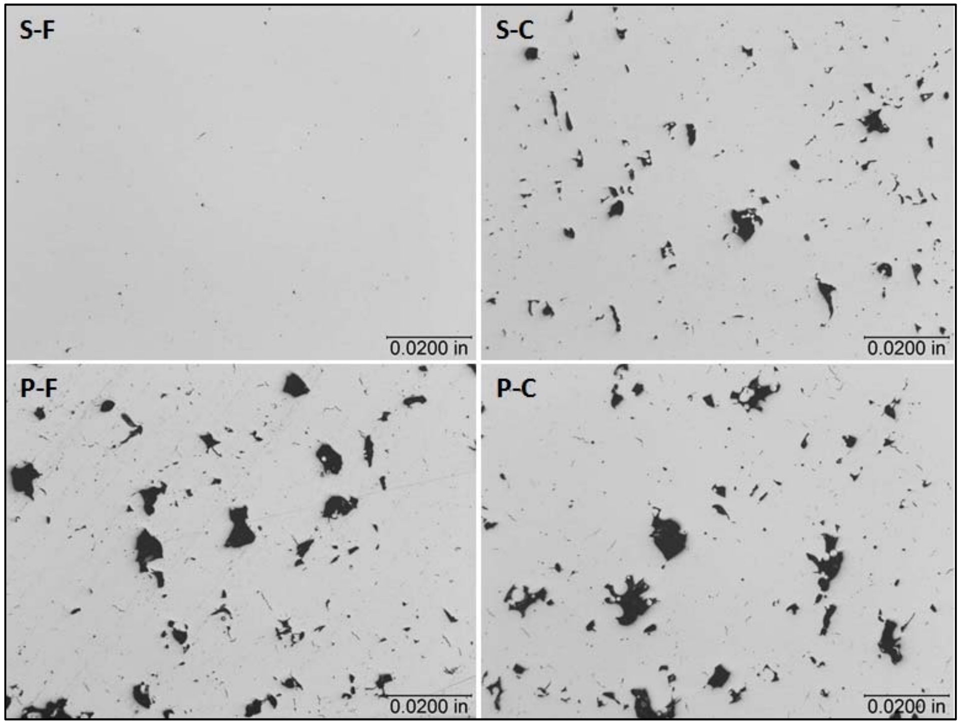
- **Challenges:**
  - Un-optimized process parameters
    - Material did not meet the density requirement for the fuel injector nozzle.
  - Process parameter development studies were conducted for S-C, P-F, and P-F powders.

	S-F	S-C	P-F	P-C
Void (%)	0.01	2.03	4.57	4.50



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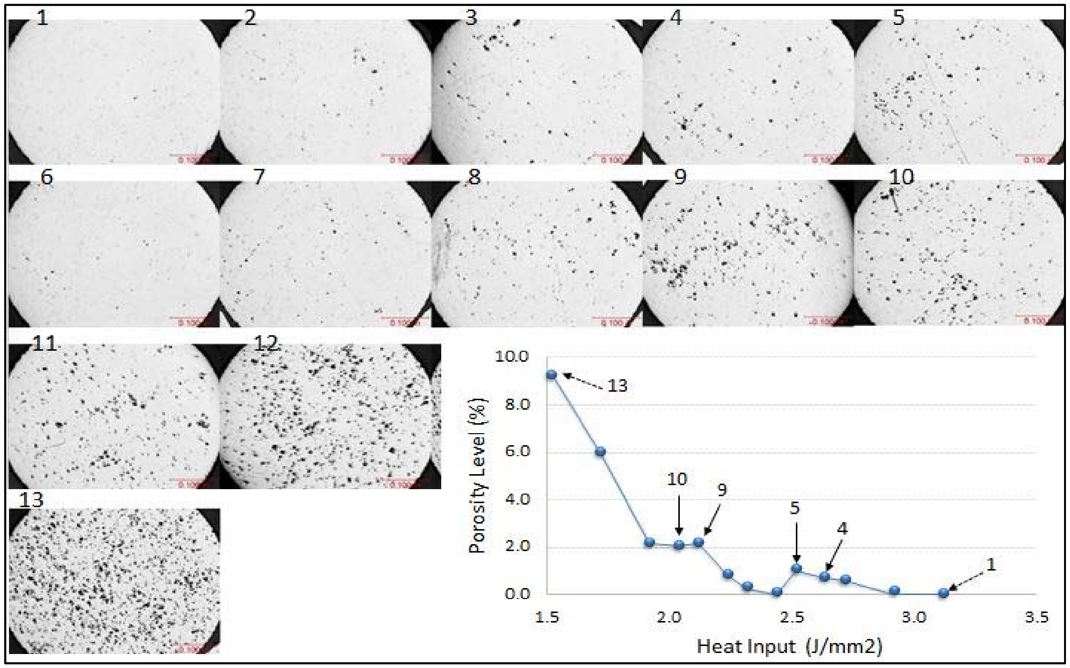
Task 2

- **Challenges:**
  - Powder leaking through seal (S-C powder)
    - Handling of free-flowing coarser powders was challenging due to leaking
    - Loss of a considerable amount of S-C powder during the fabrication of the mechanical test walls
  - A new seal replacement technique was developed at Solar Turbines to avoid this problem
  - Extra S-C powder was purchased.

Project Technical Risk Management

- **Challenges:**
  - Excessive porosity due to the poor sealing
    - Disruption of the argon air knife performance due to leaking of the front door sealing system
  - Solar Turbines fixed the front door sealing issue, and rebuilt the P-C walls.



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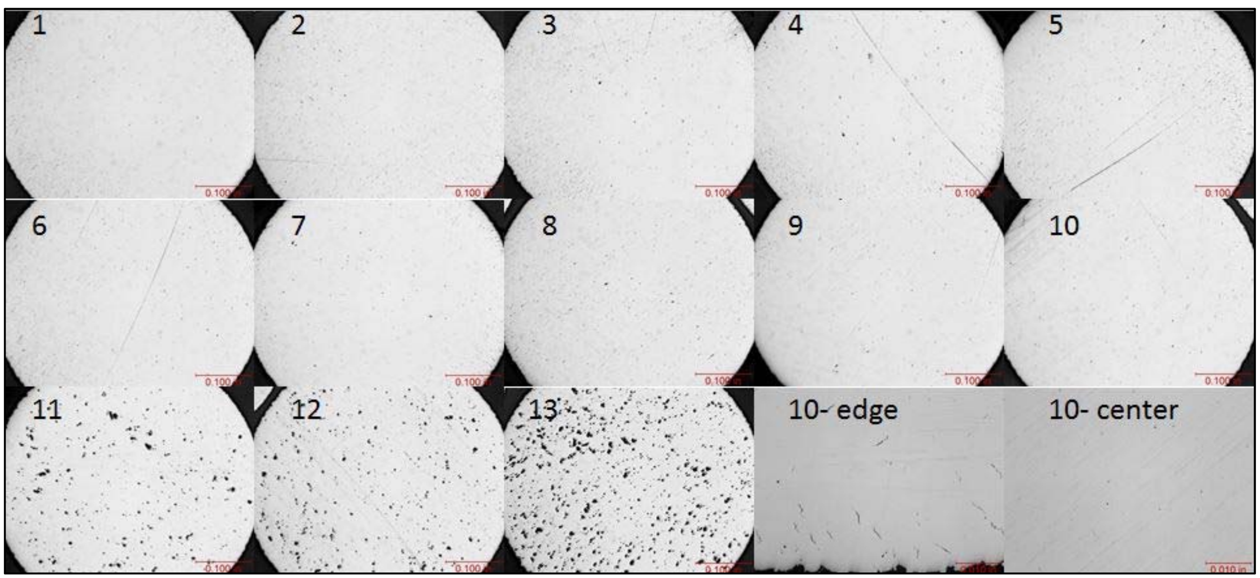
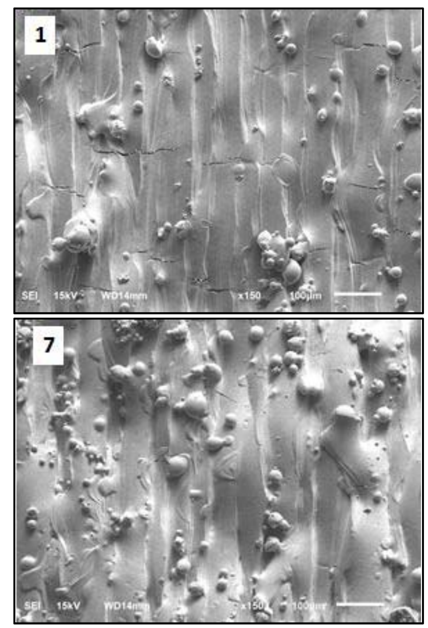
Project Technical Risk Management

- **Challenges:**
  - Cracking
    - Microcracks mostly formed around the edges





Project Technical Risk Management

- **Challenges:**
  - Cracking
    - Geometry
    - Process parameters
    - Chemical composition
      - Savage and Krantz[1] showed that an increase in the amounts of Si and Mn reduced the cracking tendency in Hastelloy X
      - Tomus et al.[2] showed that lower amounts of Si and Mn reduced the cracking tendency
      - Harrison et al.[3] showed that a minimum amount of Si and Mn is needed to minimize/avoid cracking.

[1]*Savage, W.F., and Krantz, B.M., "Microsegregation in autogenous Hastelloy X welds",* Rensselaer Polytechnic Inst., *Troy, NY, 1971.*

[2]*Tomus, D., Jarvis, T., Wu, X., Mei, J., Rometsch, P., Herny, E., Rideau, J.F., and Vaillan,t S., "Controlling the microstructure of Hastelloy-X components manufactured by selective laser melting",* Physics Procedia, *Vol. 41, pp. 823-7, December 31, 2013.*

[3]*Harrison, N.J., Todd, I., and Mumtaz, K., "Reduction of micro-cracking in nickel superalloys processed by selective laser melting: A fundamental alloy design approach",* Acta Materialia, *Vol. 94, pp. 59-68, August 1, 2015.*

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