Benefits of Tailoring
Hot Isostatic Pressure/Powdered Metal (HIP/PM) and
Additive Manufacturing (AM)
To Fabricate Advanced Energy System Components

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2016 NETL Crosscutting Research Review Meeting
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Presented by:
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in collaboration with:

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U.S. Department of Energy
National Energy Technology Laboratory
Demonstrate how tailoring HIP/PM, coupled with advances in AM (also known as 3D printing or 3DP) has specific, measurable benefits for fabricating advanced energy (AE) system components.

Goals:

- Validate that AM, in combination with HIP, offers a viable method of producing A-282 components
- Provide key information about cost, manufacturing challenges/opportunities and lead-times when compared to other methods including traditional HIP/PM and casting.
Relevance to Fossil Energy

For expensive, high nickel alloy components, EIO activities have shown advantages of HIP/PM over other methods such as casting and forging.

- Savings up to 40% in raw material costs (vs. casting)
- Eliminates difficulties resulting from reactivity of these materials in the molten state
- Facilitates manufacture of large size requirements associated with FE/AE
- Net shape & porosity free parts require less post processing including machining & weld repair

Work in AM suggests further advantages…
Potential Significance of the Results of The Work

- Many new advanced alloys for Fossil Energy will require new manufacturing methods
- Supplier Availability will determine the rate for adopting Clean Coal technologies
- Castings, Forgings, and Extrusions are *THE* “pinch points”
- Current Supply Base is Mostly Off-Shore
- Saturated with Long Lead Times

Creates opportunity for evolving US industrial base
Project Approach

Commercial Relevance

Project utilizes a Westinghouse gate valve

- Modified to ¼ scale
- 3” x 4” x 2”
- ~ 2.7 lbs
- Wall thickness range ¼” – ¾”

Valve selected for the complexity of its shape & crosscutting applications to other AE systems, including nuclear
Three new methods of manufacturing advanced alloys are under evaluation:

1. Directly built AM parts;
2. AM cans for HIP/PM; and
3. AM cans produced in the final part material.

Project is utilizing

- Binderjet technology (fastest metal 3DP technique, coupled with an alloy specific sintering profile to produce a sufficiently dense part for final HIP
- Haynes 282, a high nickel material capable of withstanding the severe operating environments required in AE systems

Project is being conducted in 3 Phases
Primary Tasks

1.0 – Project Management & Planning

2.0 – Atomization of A-282

3.0 – Material Characterization & Sintering Methodology (MC/SM) for A-282

4.0 – Produce 2 Valve Components via AM Cans & HIP/PM Manufacturing

5.0 – Produce 1 Valve via AM/3DP and HIP

6.0 – Post Processing Analysis

7.0 – Outreach & Technology Dissemination
Task 1.0 – Project Mgmt & Planning

Team Leader:
Energy Industries of Ohio

in collaboration with

Carpenter
ExOne
Bodycote
Energy Industries of Ohio

- Non Profit 501(c) 3 Corp
- Facilitate Technology Development for Ohio’s Base load Generation
- Implement Efficiency Projects for Energy Intensive User Industries
  - 9 Industries use 30% of all energy
  - Ohio is first for 3 & in the top five for others
- Foster Collaborations & Teams
  - Federal, State, University, National Laboratories & Private Industry
  - Exploit Synergies between supply and demand sectors
Our Workforce and Skills Challenge

A Two Decade Gap for Coal; Three Decades for Nuclear

Source: EIA AEO’07 reference case and Annual Energy Review 2006

2/18/2008
EIO’s Role in US Manufacturing

- Traditional manufacturing +
  - EIO is working with heavy manufacturers (castings, forgings, fabrications etc) to enhance their traditional manufacturing processes
  - Automation, energy efficiency and innovations help to offset higher labor charges domestically.

- Advanced Manufacturing
  - EIO is working on R&D projects involving both new materials and new methods of manufacturing

RESULTS: Not only are we re-shoring for US opportunities, we are also getting foreign companies approaching us with export opportunities
Technical Background/Project Motivation

National Compact Stellarator Program

- EIO charged with prototyping and providing large, high strength Nuclear Castings
- Staff experienced for working with Nuclear supplier industry, Nat’l Labs and Producers
Technical Background/Project Motivation

EIO is Prime Contractor for $50M Advanced-UltraSuperCritical Materials Program

- Pulverized Coal CCS technology
- \( \uparrow \) Efficiency \( \downarrow \) Emissions
- Consortium of All U.S. Boiler and Turbine Manufacturers and EPRI
- Goal: 5000psig, 1300\(^\circ\)F main steam and above for net plant efficiency >45%
- New Materials (nickel-based alloys) and designs
- Supply Base is key to commercialization
A-USC Consortium Members

- U.S. Department of Energy
- Energy Industries of Ohio
- EPRI
- Oak Ridge National Laboratory
- GE
- B&W
- ALSTOM
- RILEYPower
- Foster Wheeler
- Siemens

*Phase 1 only*
A progressive increase in steam conditions has been taking place worldwide.

Cost Effective Materials Have Been Critical to Achieving Increased Efficiency

US A-USC Goal

- 5400/1350/1400
- 5400/1300/1325/1325

R&D ongoing Europe, Japan, U.S. (Ni-based Materials)

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Materials Selection for A-USC Alloys (Boiler Superheater/Reheater Tubing Strength)

A-USC Technology Requires Nickel-Based Alloys

Today’s SC & USC Technology Limited by Steel Alloys
UltraSuperCritical (USC) Materials Project – Potential Show-Stoppers

Product Form and Size Limitations

The U.S. domestic boiler and turbine manufacturers are working to confirm the materials technology and component fabrication feasibility for advanced USC plant components.

The production capabilities of raw material suppliers and foundries must also be assessed for:

- Large, heavy wall pipe
- Castings
- Forgings

The ultimate plant unit size and other design aspects will be influenced by the size and product form limitations of domestic and worldwide suppliers (i.e. foundries, forges, etc.) capable of working with these new, high-strength materials.

images from Japan Steel Works
Technical Background/Project Motivation (Cont.)

- Both Programs involved locating suppliers
- Found Castings, Forgings, and Extrusions are **THE** “pinch points”
- Found Supplier Base is limited, saturated and foreign
- Found Supplier Base for Coal/Nuclear Overlaps
- Found Supplier Availability will impact the rate for introducing both Clean Coal and Nuclear Systems

Opportunity for Supply Chain Development
Ohio/TechBelt Opportunity for an Advanced Energy (AE) Supplier Program

- These needs are traditional TechBelt products
- EIO has direct relationships with these industries and their affiliated organizations FIA, OCMA, OSC, etc.
- Knowledge that their current markets are declining
- Knowledge that they are looking for new markets
- Knowledge that they are capable of transition into AE markets
  - But…They don’t know of Advanced Energy opportunities
  - And…Power Gen potential customers don’t know of them

EIO could connect the dots!
EIO Approach in Ohio & TechBelt

EIO employed a different (bottoms-up) model

1. Develop the specific “needs envelope” (sizes, alloys, etc.) of target “pinch point” items
   * Worked with key customers from fossil and nuclear
2. Use Industry organizations to ID candidate suppliers
3. Conduct on-site visits to assess interest & ability
4. Facilitate customer interaction and teaming opportunities
5. “Champion” needs for transitional assistance
6. Pursue technology development & demonstration
EIO Approach in Ohio & TechBelt

Program Outcomes

✓ A Catalogue listing Ohio Suppliers that can meet the AE Power Gen Industry needs – Project was expanded to include Pittsburgh Region

✓ Promoting the Catalogue to OEM’s & Customers

✓ Cultivation of HUBS around pinch points & market/export opportunities

✓ Advanced Research, Prototype Development & Industry Expansion
Technical Background/Project Motivation

Under the Ohio Program, EI O conducted Research & Technology Development Using A-282

- Produced World’s largest Step Casting followed by an AE Valve using A-282

- Working with Carpenter & Bodycote - Duplicated A-282 Step Component using HIP/PM
Technical Background/Project Motivation

- Additive Manufacturing is a logical progression in seeking new methods for producing FE/AE components.
- Dialogue and collaboration with our colleagues at Carpenter, ExOne and Bodycote focused on finding ways to make AM and HIP/PM more competitive.
- Potential advantages, including reduced costs and leadtimes, of combining AM with HIP/PM resulted in this project proposal.
- Carpenter, ExOne and Bodycote are all highly respected companies in their fields, with facilities in the TechBelt.
Atomization of A-282 PEP – Carpenter Powder Products

Manufactures a broad range of gas atomized loose metal powders and consolidated powder forms

Manufacturing: PA, RI and Sweden

R&D: Reading, PA
PEP – Carpenter Powder Products

Leader in Gas Atomization Technology

Bridgeville, PA
- Air Induction
- VIM (2)
- Ar and N

Torshalla, Sweden
- Air Induction (2)
- N

Woonsocket, RI
- Protected atm.
- Ar and N

Stainless steels, nickel/cobalt base, fine powders, tool steels
Capacity – 20,000 Tons
PEP – Carpenter Powder Products

Program Task 2: Powder Manufacturing

Melting → Screening → Particle Size Determination → Blending → Chemical Analysis
A Global Supplier of Industrial Additive Manufacturing Equipment

- 50-year history of developing and implementing nontraditional manufacturing processes.
- Invested >$80 million in the development and implementation of three-dimensional printing (3DP) since the early 1990s.
- Offers both the services and the equipment for applying 3DP technology for molds / cores used for sand castings and direct metal parts.
- ExOne Production Service Centers are located throughout the United States, Germany and Japan.
- ExOne systems are able to print in a variety of industrial materials with the largest available build sizes.
Direct Metal Technology

1. Spreading new layer of metal powder
2. Powder Printing
3. Print-Bonded Particles
4. Particles agglomerated in one droplet (Voxel)
5. Parts Stilted for Infiltration
6. Sintered Particles
Direct Metal Technology

1. Spread layer of metal powder
2. Selective dispensing of binder using inkjet printing technology
3. Binder drying
4. Repeat step 1 to 3
5. Extraction and cleanup of green part
6. Binder burnout and densification
3DP is Basic Powder Metallurgy

Product Forms

Bonded

Partially Sintered

Infiltrated

Highly Sintered

Powder metallurgy is the process of blending fine powdered materials, pressing them into a desired shape or form (compacting [printing]), and then heating the compressed material in a controlled atmosphere to bond the material (sintering). The powder metallurgy process generally consists of four basic steps: powder manufacture, powder blending, compacting
ExOne – Materials Research

Material Form
- Bonded
- Porous
- Matrix
- Solid

Silica Sand
- Chromite
- Cerabeads
- Ceramsite
- FeCrAl Alloy
- Inconel 625
- Iron / Bronze

Carbon
- SiC
- Hybrid
- Tungsten Carbide
- Haynes 282
- 17-4 PH SS
- Rene 80
- Cobalt Chrome
- Ti 6-4 Ti Cp
- Carbon

Zircon
- Alumina
- Plaster
- Glass
- Silica
- Inconel 718
- Maraging Steel
- Aluminum
- Magnesium
- Copper
- Stellite
- Hastelloy
- M2 or H13
### 3DP Binder Jetting Parts – Rapid Production

<table>
<thead>
<tr>
<th>System</th>
<th>Speed</th>
<th>Build Rate</th>
<th>Layer Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>M-Print</td>
<td>75 seconds per layer</td>
<td>2052 cm³/hr (125 in³/hr)</td>
<td>Variable with minimum of 0.15 mm (0.006 in)</td>
</tr>
<tr>
<td>M-Flex</td>
<td>30 seconds per layer</td>
<td>1200 cm³/hr (73 in³/hr)</td>
<td>Variable with minimum of 0.1 mm (0.004 in)</td>
</tr>
</tbody>
</table>
Build Volume:
15.5 x 9.5 x 9.5 in.
394 x 241 x 241 mm

Layer Thickness:
100 or 180 microns

Accuracy:
+/- 0.5%

System includes
depowdering station
and curing oven
Build Volume:
29.5 x 15 x 15.75 in.
750 x 380 x 400 mm

Layer Thickness:
100 or 180 microns

Accuracy:
+/− 0.5%
ExOne Technologies

Sand Casting Molds and Cores - Without a Pattern

Micro Holes and Features with Advanced Laser Machining

Industry Class Additive Manufacturing Equipment

Functional and Accurate 3D Printed Metal Parts
Direct Metal Technology – S Max

PRODUCTION PRINTERS

S-MAX Tech Specs

The S-Max, suited for sandcasting foundries, creates complex sand molds directly from CAD data, eliminating the need of a physical core or mold. The ability to cast in hours without hard tooling in casting process chain.

Job Box
- for building and unloading process
- on motorized roller conveyor
- additional Job Box optional

Operating Panel

Control Cabinet

Fluid Connectors
for safe handling of liquid media

BINDER SYSTEMS
Furan

BUILD VOLUME
l x w x h 70.9 x 39.4
(1800 x 1000 x 700)

BUILD SPEED
2.12 to 3.00 ft³/h
(60–85 L/h)

l x w x h 271.7 x 138.6 x 112.6 in.
including one job box, right - standard (6900 x 3520 x 2860 mm)
Bodycote operates a global HIP business with the largest equipment network in the world.

Bodycote has over 50 HIP vessels of varying sizes in multiple locations and is able to accommodate large volumes of small products as economically as large individual components.

Bodycote provides two major HIP routes for customers:

- HIP Services, providing porosity removal through HIP densification.
- HIP Product Fabrication, for the manufacture of components through powder metallurgy and diffusion bonding.
Hot Isostatic Pressure (HIP) combines high temperatures (up to 2,000° C) with isostatically applied gas pressures (up to 45,000 psi) – comparable to the Mariana Trench 11,000m deep in the Pacific Ocean.
Hot Isostatic Pressing (HIP) combines very high temperature and pressure to eliminate porosity in castings, and consolidate encapsulated powders to give fully dense materials.

Dissimilar materials can be bonded together to manufacture unique, value-added components.
**HIP Process**

Both manufacturing processes require HIP
- AM Cans are filled with PM then HIP’d.
- AM Valve is also HIP’d to achieve full density

**HIP Trials** were conducted to determine if the AM cans and AM component could be run through a Coach* HIP Cycle

<table>
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<th>Temperature</th>
<th>Time</th>
<th>Pressure</th>
<th>Atmosphere</th>
</tr>
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<tbody>
<tr>
<td>2125° F +/- 25° F</td>
<td>240 +15/-0 minutes</td>
<td>14.75+/- .25 KSI</td>
<td>argon</td>
</tr>
</tbody>
</table>

*Successful use of the Coach HIP cycle, (as opposed to requiring a customized furnace profile), contributes to the cost competitiveness of the overall manufacturing processes when compared to other manufacturing processes such as casting and forging. Parts can be batched or combined with other orders to reduce cost and leadtime.
Progress on Primary Tasks

1.0 – Project Management & Planning

2.0 – Atomization of A-282 ✔

Phase 1

3.0 – Material Characterization & Sintering

   Methodology (MC/SM) for A-282 ✔

Phase 2

4.0 – Produce 2 Valve Components via AM

   Cans & HIP/PM Manufacturing ✔

5.0 – Produce 1 Valve via AM/3DP and HIP ✔

6.0 – Post Processing Analysis

Phase 3

7.0 – Outreach & Tech Dissemination
Task 2 - Atomization of 282

- For the HIP process – powder screened to roughly ~ 250 microns
- For the AM process – powder screened to max size 22 microns

SEM photomicrographs of the AM powder are shown in Figures 1 & 2.
Task 3 – Material Characterization & Sintering Profile

- Testing of the A282 was conducted in a high vacuum furnace producing a vacuum below 3.0x10^{-5}
- Three pump downs and backfills with inert gas (96% nitrogen & 4% hydrogen) were done before each run to ensure any moisture was removed from the chamber
- Burnout Temperature 600° C (to remove binder)
- Variables in the test runs included:
  - Furnace Temperature (min 1290° C to max 1325° C)
  - Max Temperature Hold Time (1 hour – 1.5 hour)
  - Ramp-up and Ramp-down Rates (5° C/min to 1° C/min. The ramp rate down from 1315° C to 800° C was also changed from 5° C/min to 1° C/min)
  - Number of samples in a run (ranged from 1 – 3)
  - Size of sample

FINDING: Achieved 99.6% density before machining, with no distortion or cracks!
In the optimized run, the maximum temperature was 1295°C, held for one hour. The details of the profile used for this test are shown in the table at right. 99+% density was achieved.

Result: Sample was 99.6% dense with no distortion before machining. The sample is shown at right after machining.
Task 4 – Produce 2 Valve Components via AM Cans & HIP/PM Manufacturing

EIO Team produced two fully dense* AM cans of differing wall thicknesses (.125” & .150”) in A-282, the same material as the final part, to explore potential benefits of producing the cans from the final part material.

Pictured below: varied angles of the sintered 0.150 inch walled can.

*Achieving full density allows cans to be filled with PM without first having to run through a HIP cycle.
Task 4 – Produce 2 Valve Components via AM Cans & HIP/PM Manufacturing

AM Cans were filled with Powdered A-282 in preparation for HIP

Both the valve HIP cans as well as the AM valve component were run in a coach HIP cycle.

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</tr>
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</table>
Task 4 – Produce 2 Valve Components via AM Cans & HIP/PM Manufacturing

Two cans of differing thicknesses were produced via Additive Manufacturing.

Fill or Feed Tube for PM was welded on to AM can.

The can on the left is the .125” can prior to HIP.

Feed tube is removed post HIP.

The can on the right is the .150” post HIP.
Task 4 – Produce 2 Valve Components via AM Cans & HIP/PM Manufacturing

The completed components from the HIP’d .125 and .150” cans are shown below:
Task 4 – Produce 2 Valve Components via AM Cans & HIP/PM Manufacturing

More distortion on the .125” can was detected as compared to the .150” can (pictured below). Future testing could be pursued under a follow-on grant to ascertain optimal can wall thickness and other strategies to overcome distortion.
Task 4 – Produce 2 Valve Components via AM Cans & HIP/PM Manufacturing

Cross section shows significant minus material on .125” can
Task 4 – Produce 2 Valve Components via AM Cans & HIP/PM Manufacturing

Unsupported flanges “sagged” during HIP on both .125” & .150” cans. No sag in 3D-Printed valve
Task 5 – Produce 1 Valve Component via AM/3DP & HIP

3DP Valve Successfully Produced in A-282

The support cylinders on the flange shown in the bottom view image (blue arrows highlight two of them) were added in an attempt to allow the part to shrink uniformly and minimize distortion during sintering.

The AM valve is subsequently HIP’d to achieve full density.
Task 5 – Produce 1 Valve Component via AM/3DP & HIP

Final AM valve component after HIP and Heat Treat
EIO Has Commenced Phase 3

→ Task 6 - Post Processing Analysis
EIO Has Commenced Phase III

Activities Include:

➤ Task 6 – Post Processing Analysis
  ✔ Argon Analysis
  ➤ Metallography
  ✔ Photography of Finished Parts
  ✔ Section Final Components for Test & Evaluation
  ➤ Conduct Chemical and Physical Property Tests

➤ Task 7 – Outreach & Technology Dissemination
  ✔ Outreach Plan
  ➤ Dissemination Activities Are On-going
Task 6 – Post Processing Analysis

- The varied parts were all subjected to Heat Treatment (HT)
- New Heat Treat protocol was established by experts at ORNL based on previous work
  1. Precipitation age harden 1850 F for 2 hours then air cool
  2. Reheat to 1450 F for 8 hours.
- Heat Treat was performed by Bodycote in Cincinnati, OH
Task 6 – Post Processing Analysis

Chemical and Physical Property Tests will be conducted and results will be compared with published and gathered data.

<table>
<thead>
<tr>
<th>Process</th>
<th>Physical Properties (RT)</th>
<th>Chemical Composition, Weight % (4)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.2% YS (ksi)</td>
<td>UTS (ksi)</td>
</tr>
<tr>
<td>Published (1)</td>
<td>103.7</td>
<td>166.4</td>
</tr>
<tr>
<td>Powder (2012 - Carpenter)</td>
<td>bal</td>
<td>19</td>
</tr>
<tr>
<td>Cast Step (2011)</td>
<td>58.29</td>
<td>19.345</td>
</tr>
</tbody>
</table>

(1) solution annealed & age hardened plate
(2) Nickel as balance
(3) Maximum
(4) Trace elements < .005% not listed
Task 6 – Post Processing Analysis

Finished Components were sectioned for evaluation by multiple sources
### Summary: Progress To Date (4/19/16)

#### 1.0 - Project Management & Planning

- ✔ 2.0 – Atomization of A-282

#### Phase 1

- ✔ 3.0 – Material Characterization & Sintering Methodology (MC/SM) for A-282

#### Phase 2

- ✔ 4.0 – Produce 2 Valve Components via AM Cans & HIP/PM Manufacturing
- ✔ 5.0 – Produce 1 Valve via AM/3DP and HIP

#### Phase 3

- ✔ 6.0 - Post Processing Analysis
- ✔ 7.0 - Outreach & Tech Dissemination
Tailoring HIP/PM with advances in AM provides specific, measurable benefits for fabricating advanced energy (AE) system components.

Three new methods of manufacturing advanced alloys under evaluation include:

1. Directly built AM parts;
2. AM cans for HIP/PM; and
3. AM cans produced in the final part material.

Potential advantages include lower manufacturing costs, ability to produce more complex designs, improved production efficiency & readily transferrable technology.

Post Processing Analysis will identify future R&D direction.
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