Additive Manufacturing of Corrosion Resistant Alloys for Energy Applications

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Agenda

Fundamental Processes in Metals Based Additive Manufacturing
Additive Manufacturing at Penn State
Additive Manufacturing of Corrosion Resistant Alloys
  - Titanium
  - Nickel Base Alloys
  - Duplex Stainless Steels
Fundamental Processes in Additive Manufacturing
Standard Definition of Additive Manufacturing

According to ASTM F2792:

**additive manufacturing (AM), n—a process of** joining
materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies.

Synonyms: additive fabrication, additive processes, additive techniques, additive layer manufacturing, layer manufacturing, and freeform fabrication.

Additive manufacturing of metallic systems may be divided into two categories:
- Directed beam with the addition of a feedstock (powder or wire)
- Scanning of the beam onto a pre-deposited powder bed

Process selection may be based upon build size, build rate, material, and feature definition requirements
AM Processes Allow for the Fabrication of a Wide Range of Geometries and Sizes

Small Complex Components → Large Bulky Components

Powder Bed Fusion → Directed Energy Deposition
**Powder bed fusion, n**—an additive manufacturing process in which thermal energy selectively fuses regions of a powder bed.

**direct metal laser sintering (DMLS), n**—a powder bed fusion process used to make metal parts directly from metal powders without intermediate “green” or “brown” parts; term denotes metal-based laser sintering systems from EOS GmbH - Electro Optical Systems. Synonym: direct metal laser melting.
**Directed Energy Deposition**

**directed energy deposition, n**—an additive manufacturing process in which focused thermal energy is used to fuse materials by melting as they are being deposited. Focused thermal energy means that an energy source (e.g., laser, electron beam, or plasma arc) is focused to melt the materials being deposited.

A “deposition head” is utilized to deposit material onto the substrate.

The substrate can be either a flat plate on which a new part will be fabricated, or an existing part onto which additional geometry will be added.

Deposition is controlled by relative differential motion between the substrate and deposition head.

The laser generates a small molten pool on the substrate as powder is injected into the pool.

The powder is melted as it enters the pool and solidifies as the laser beam moves away.
Complexity of AM Process and Research Needs

- Material Chemistry/Compatibility
- Powder Feedstocks
- Beam Material Interactions
- Melt Pool Dynamics
- Defects
- Post Processing
- Properties

The interrelationships between design, processing, structure, and performance are complicating the development of standards and certification and qualification protocols.

**Design**
- Geometries and Features
- Breakdown of Part Features
- Material Property/Feature Relationships

**Processing**
- Definition of Essential Variables
- Processing/Structure Relationships
- Quantification of Processing Variable Uncertainty

**Material Properties**
- Development of Structure/Property Relationships
- Impact of Features on Material Properties
- Quantification of Material Property Uncertainty

**Product and Performance**
- Assign Reliability to Properties and Analysis
- Validate Global Properties
- Verify Design Requirements
Additive Manufacturing at Penn State
A national resource for additive manufacturing technologies:
university-wide initiative
operated by Penn State’s Applied Research Laboratory, a DoD University Affiliated Research Center (UARC)

An Additive Manufacturing Demonstration Center (AMDF) under the DARPA Open Manufacturing Program

With a mission to:
advanced additive manufacturing technologies,
promote adoption through process and product demonstrations, and
promote and sustain additive manufacturing.

Various Enabling Technologies
Range of Additive Manufacturing Machines Available

- DMG Mori Lasteric 65
- GE Vtomex-M CT
- Stratasys Fortus 400 mc
- ExOne Mlab
- 3D Systems ProX 200
- Optomec LENS
- EOS M280
- 3D Systems ProX320
- Sciaky EBAM
- HP/LS
- DMG Mori Lasertec 65
- GE Vtomex-M CT
- Stratasys Fortus 400 mc
- ExOne Mlab
- 3D Systems ProX 200
- Optomec LENS
- EOS M280
- 3D Systems ProX320
- Sciaky EBAM
- HP/LS
- DMG Mori Lasertec 65
- GE Vtomex-M CT
- Stratasys Fortus 400 mc
- ExOne Mlab
- 3D Systems ProX 200
- Optomec LENS
- EOS M280
- 3D Systems ProX320
- Sciaky EBAM
- HP/LS
Wide Range of Faculty and Research Interests

Dr. Richard Martukanitz  
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Dr. Edward Reutzel  
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Materials Sci. & Eng.

Dr. Sanjay Joshi  
Ind. & Man. Eng.

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Dr. Karen Thole  
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Dr. Pan Michaleris  
Mech. & Nuclear Eng.

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ARL Penn State

Dr. Judith Todd  
Supporting the Innovation Ecosystem for AM

Undergraduate and graduate education

Founding member of America Makes

Industry Practicums and Technology Exchanges

Close collaboration with numerous government organizations

Healthy commercial R&D portfolio

Engaged in several governing agencies on standards:
  - ASTM F42 Subcommittee
  - AWS C7 and D20 Committees
  - Metallic Materials Properties Development and Standardization (MMPDS)
  - Metallic Materials Properties Development & Standardization
  - ASME Design, Materials, and Manufacturing Segment
Additive Manufacturing of Corrosion Resistant Alloys
Titanium Alloys
Wide range of mechanical properties reported for AM fabricated Ti-6Al-4V.

Even with nominally similar processing conditions, mechanical properties reported for AM fabricated Ti-6Al-4V still vary, particularly for elongation.

Changes in Microstructure with Height on Build Wall

- **Thin Wall**
  - 80 mm
  - 60 mm
  - 40 mm
  - 20 mm

- **Thick Wall**
  - 80 mm
  - 60 mm
  - 40 mm
  - 20 mm
The tensile strengths decreased linearly with increasing height for all the wall structures and for both the longitudinal and transverse orientations.

The elongation measured from the longitudinal tensile samples extracted from the thick wall structures increased linearly with increasing height.

The height was measured as a distance along the build direction between the substrate and where the tensile sample was extracted.

Orientation Has Statistically Significant Impact on Mechanical Properties

Longitudinal oriented samples exhibited 40 MPa higher average tensile strength (1072 ± 33 MPa) than transverse oriented samples (1032 ± 31 MPa).

The longitudinal samples also exhibited a significantly lower elongation (17.0 ± 4.3%) compared to the transverse oriented tensile samples (19.3 ± 4.4%).

Wall Thickness Also Has Statistically Significant Impact on Mechanical Properties

Thick wall structures exhibited a higher average tensile strength of 1066 ± 36 MPa compared to the thin wall structures which averaged 1032 ± 31 MPa.

In addition, the thick wall structures averaged a slightly higher elongation (19.4 ± 4.7%) than the thin wall structures (17.1 ± 3.9%).

Characterization of Prior Beta Grain Formation Shows Impact of Changing Geometry

Prior Beta Grain Measurements Show Trends With Tensile Strength

The orientation dependence on the mechanical properties may be the result of the transversely oriented columnar prior β grains.

The higher number of prior β grain intercepts in the longitudinal direction may have resulted in a higher amount of grain boundary strengthening.

Nickel Base Alloys
### Wide Range of Allowable Compositions in Inconel® 625

<table>
<thead>
<tr>
<th>Element</th>
<th>Standard</th>
<th>Powder</th>
<th>Deposit</th>
<th>Powder</th>
<th>Deposit</th>
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<tbody>
<tr>
<td>Ni</td>
<td>&gt; 58.0</td>
<td>64.8</td>
<td>64.06</td>
<td>61.3</td>
<td>60.62</td>
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<tr>
<td>Cr</td>
<td>20.0 – 23.0</td>
<td>21.0</td>
<td>21.59</td>
<td>21.3</td>
<td>21.46</td>
</tr>
<tr>
<td>Fe</td>
<td>&lt; 5.0</td>
<td>1.02</td>
<td>1.07</td>
<td>4.34</td>
<td>4.14</td>
</tr>
<tr>
<td>Mo</td>
<td>8.0 – 10.0</td>
<td>8.73</td>
<td>8.83</td>
<td>8.70</td>
<td>8.96</td>
</tr>
<tr>
<td>Nb</td>
<td>3.15 – 4.15</td>
<td>3.43</td>
<td>3.47</td>
<td>3.83</td>
<td>4.11</td>
</tr>
<tr>
<td>Si</td>
<td>&lt; 0.50</td>
<td>0.37</td>
<td>0.39</td>
<td>0.035</td>
<td>0.051</td>
</tr>
<tr>
<td>Mn</td>
<td>&lt; 0.50</td>
<td>0.31</td>
<td>0.28</td>
<td>0.010</td>
<td>0.085</td>
</tr>
<tr>
<td>C</td>
<td>&lt; 0.10</td>
<td>0.008</td>
<td>0.009</td>
<td>0.005</td>
<td>0.006</td>
</tr>
<tr>
<td>Ti</td>
<td>&lt; 0.40</td>
<td>0.019</td>
<td>0.033</td>
<td>0.19</td>
<td>0.21</td>
</tr>
</tbody>
</table>

**Low Fe Content**

**High Fe Content**
Fabrication of Inconel® 625 Wall Structures to Study Impact of Composition

Processing Parameters

Technique: Laser-based Directed energy deposition
Power: 2000 W
Scan speed: 10.6 mm/s
Hatch spacing*: 2.54 mm & 2.29 mm
Nozzle standoff: 10 mm

• Selected Inconel® 625 builds fabricated were also subjected to a standard HIP cycle

HIP Conditions
1160°C for 4 hours at 130 MPa

Tensile specimens are extracted at various heights and in different orientations
Fe Content Has Minimal Impact on As Deposited Microstructure

Both solidification structures had blocky, irregularly shaped morphologies for the secondary phase constituents.

*No change along build height
**Mechanical Properties Displayed Significant Differences With Fe Content**

Low Fe content specimens exhibited higher yield and tensile strengths

High Fe content specimens exhibited higher elongations

Properties did not change with height or orientation

<table>
<thead>
<tr>
<th>Fe Content</th>
<th>Orientation</th>
<th>Number of Tensile Specimens Tested</th>
<th>Yield Strength (MPa)</th>
<th>UTS (MPa)</th>
<th>Elongation (%)</th>
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</thead>
<tbody>
<tr>
<td>Low Fe</td>
<td>Longitudinal</td>
<td>6</td>
<td>531 ± 7</td>
<td>874 ± 25</td>
<td>32 ± 3</td>
</tr>
<tr>
<td></td>
<td>Transverse</td>
<td>6</td>
<td>508 ± 10</td>
<td>846 ± 24</td>
<td>40 ± 4</td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td>12</td>
<td>520 ± 12</td>
<td>860 ± 27</td>
<td>36 ± 5</td>
</tr>
<tr>
<td>High Fe</td>
<td>Longitudinal</td>
<td>6</td>
<td>460 ± 35</td>
<td>763 ± 27</td>
<td>40 ± 7</td>
</tr>
<tr>
<td></td>
<td>Transverse</td>
<td>6</td>
<td>439 ± 6</td>
<td>743 ± 27</td>
<td>49 ± 8</td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td>12</td>
<td>450 ± 27</td>
<td>753 ± 25</td>
<td>44 ± 9</td>
</tr>
</tbody>
</table>

**Mechanical Properties Graph:**

- **High Fe**
- **Low Fe**

**Graph Details:**
- UTS
- Yield strength
- Height (mm)
- Orientation: Longitudinal Specimens
- Dimensions: 150 mm x 100 mm x 13 mm
Addition of HIP Produces Lower Strength and Higher Elongation

Low Fe as deposited

Low Fe HIP

High Fe as deposited

High Fe HIP
Duplex Stainless Steels
Directed Energy Deposition of Duplex Stainless Steels

- Laser Power: 2 kW
- Travel Speed: 635 mm/min
- Step Over Distance: 2.5 mm
- Standoff Distance: 10 mm
- Laser Spot Size: 4 mm
- Powder Nozzle Diameter: 2 mm
- Powder Flow Rate:
  - Duplex 2205: 13.5 g/min
- Shielding Gas: 100% Argon
- Shielding Gas Flow Rate: 47.2 L/min
Increases in Build Height show a Reduction in Ferrite Bands
Increases in Build Height Showed Increases in Austenite

Austenite: 27% ± 1%

Austenite: 47% ± 2%

Austenite: 56% ± 4%
Three Types of Austenite Are Observed and High Levels of Intragranular Austenite

Ferrite = Red
Austenite = Blue
2205 DSS HIP Microstructure is Homogeneous and Well-Balanced
AM Microstructure Had Less Segregation Than Wrought And HIP Conditions

AM + HIP
Ferrite
Austenite
Wrought
AM
Microstructures Vary Across Different Grades of DSS

2101 DSS

As-Deposited

HIP

Wrought

2205 DSS

As-Deposited

HIP

Wrought

2507 DSS

As-Deposited

HIP

Wrought
Back up Slides
Functional Grading of Materials in AM
Use of AM to Produce Functionally Graded Joints

Directed Energy Deposition

Design of Joint to Minimize Carbon Diffusion

Controlling Microstructure

Photo courtesy of PSU ARL
The AM community faces a number of cross-cutting challenges impeding the development of new material systems.

These characteristics can add a dimension of intricacy to materials development by exploiting the unique processing advantages of AM to create state-of-the-art multi-materials that combine more than one type of material.

The five strategic thrusts in this roadmap acknowledge the fundamentally unique AM processing considerations of these material classes and call for research needed to create new AM-based metal, polymer, and ceramic materials.
What are Multi-Materials?

A growing research and development topic in AM is the ability to additively manufacture components made of several different materials (e.g., metals, ceramics, and polymers).

Pre-process feedstock blending and in-situ alloying are used to formulate intricate AM parts from combinations of metal-metal, metal-ceramic, polymer-ceramic, and polymer-metal materials.

The ability to blend different feedstock powders not only gives designers the freedom to customize materials that meet end-user requirements, but also offers the potential to vary the material composition during processing to fabricate high-performance, functionally-graded materials (FGM).

While multi-materials are currently produced in a rudimentary nature, the approach holds great promise for delivering performance not currently possible.

Manufacturing FGM parts with microstructural gradients exhibiting unique or unusual properties and functionalities represents a fundamentally new paradigm in the selection and design of advanced material systems.