Heat Treatment of AM Parts by Hot Isostatic Pressing

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Isostatic Pressing

- Isostatic pressing is a forming process that applies equal pressure in all directions on a product, compacting the workpiece uniformly from all sides
  - This results in maximum uniformity of density and microstructure without the geometrical limitations of uniaxial pressing

- Hot Isostatic Pressing (HIP) is used to fully consolidate parts at elevated temperatures.
  - Temperatures are usually 500-2,000ºC (932-3,632ºF)
  - Pressures are usually 100-200 MPa (15,000 to 30,000 psi)
  - Gas used is typically argon

- The densification process is a combination of
  - Plastic deformation
  - Creep
  - Diffusion
Design of Quintus® pressure vessels - Core Technology

- Wire wound vessel gives a reduced footprint whilst decreasing the frequency needed for safety inspections
- The Patented Quintus URC® furnace technology includes a fan or nozzle for forced convection cooling
- High cooling rates allow combined heat treatment cycles

URC® and URQ® are registered trademarks of Quintus Technologies AB
What is Hot Isostatic Pressing used for?

- Refining a product
  - HIP is used as an extra process step to achieve better material properties

- Producing a product
  - HIP is used to produce the product ready for final post processing steps such as grinding or machining

- Densification of products produced by
  - Casting
  - Metal Injection Moulding (MIM)
  - Additive Manufacturing / 3D printing
  - Cladding
  - Diffusion bonding

- Compaction of powder
  - Powder billets
  - Tool steel
  - Near-Net-Shape (NNS)
  - Sputtering targets
Hot Isostatic Pressing

As-cast / MIM / 3D print

Powder Metallurgy

Near-Net-Shape Product

Pores Eliminated
HIP of components with residual porosity

- HIP is used to remove porosity in metal parts
  - Internal porosity in Castings is always present after solidification due to material shrinkage
  - Micro porosity in MIM and 3D-printed components

- The combination of high temperature and high isostatic pressure will remove residual porosity
  - Temperature typically 80-90% of melting temperature
  - Pressure up to 2070 bar of Argon gas
  - At these pressures the gas acts as a liquid
As-printed material

• Internal defects as-printed
  • Lack-of-fusion porosity
  • Gas porosity etc.
  • Oxides
  • Micro cracks

• Internal defects means
  • Stress concentrations
  • Crack initiation points

• Negative influence on
  • Fatigue
  • Ductility
  • Fracture toughness
Why HIP?

- Elimination of internal defects gives:
  - Elimination of stress concentrations and crack initiation points
- Superior material properties
  - Drastically increased fatigue life 10 – 100x
  - Increased ductility and fracture toughness
  - Reduced scatter
    - More predictive material properties
    - Increased lowest design values
- Improved machined/polished surface quality
  - Increased fatigue, corrosion, optical, sealing and esthetic properties

Variations in AM - Variables

Feedstock / Material
- Chemistry
  - Starting Chemistry
    - Variation from lot to lot
    - Variation within lot
    - Macro / micro segregation
  - Recycling
    - Humidity or other Environmental Factors
      - Contamination, sieves, seals, brushes, etc.
    - Size distribution and morphology changes
  - Chemistry change, bulk and surface
- Bed Density
  - Particle Size Distribution
  - Apparent / Tap Density
  - Sintering

Thermal Profile
- Process Parameters
  - Start Temperature
    - Bottom thermocouple placement critical
  - Preheat Temperature
    - Based on geometry
  - Beam Focus value (not calibrated)
  - Line Order
  - Raster Speed
  - Beam Current
  - Melt Scheme
    - Scan Strategy, line, point, area melting
    - Beam current, speed, focus, etc. changes with geometry due to physics
  - Post heat
    - Same as preheat, but incorporates melt, geometry and overall current
    - Between parts or at the end of layer

Execution
- Machine to Machine Variability
  - Different software version
  - Different power supplies, filaments, e- gun designs, focusing coils, etc.
  - Operating environments – temperature, humidity, magnetic fields, etc.

- Process Variability
  - Operator
    - Variation in personnel
    - Steps in Magics Software
      - Support Structure Generation
      - Additional parts for heat sinks
      - How builds are loaded in machine
    - Order
    - Grouping of parts
  - Location
  - Maintenance
    - Different Support Staff from Arcam

Courtesy of Oak Ridge National Laboratory
Variations in AM – Build plate position

- Example (EBM T64):
  - Sample position on the build plate =>
  - Variation in porosity

![Sample images](image-url)
Typical AM operations

Pre-Processes
- Design
- Shrinkage calculation
- Powder selection
- Powder sourcing
- Powder handling
- Cleaning of AM system
- Evacuation / Purging
- Pre-heating

Printing
- Spreading powder
- Printing contour
- Printing hatch pattern
- In-situ monitoring
- Recycling of powder

Post processes
- (Curing)
- (De-binding)
- (Sintering)
- Stress relief
- Part removal
- Support removal
- Recycling of build plate
- Cleaning
- HIP
- Heat treatment
- Machining
- Chemical milling
- Abrasive flow machining
- Sand blasting
- Short peening
- Coating

Quality control
- CT scanning
- Inspection
- Powder management
- Property checks

(Binder Jet Process)
High Pressure Heat Treatment

Savings in:
- Lead time
- Cost
- Resources
- Capital investment
What does this mean for AM?
Typical AM process flow

**EBM**
- Pre-Processes
- Printing
- De-caking / part removal
- HIP\(^1\)
- Heat treatment\(^2\)
- Aging\(^3\)
- Cleaning and Surface finishing
- Part inspection
- Finished Part

\(~99.9\%\) (100\%)

**SLM**
- Pre-Processes
- Printing
- Stress Relief\(^2\)
- Support removal / Removal from build plate
- HIP\(^1\)
- Heat treatment\(^2\)
- Aging\(^3\)
- Cleaning and Surface finishing
- Part inspection
- Finished Part

\(~99.9\%\) (100\%) \(~99.9\%\)

**BJ**
- Pre-Processes
- Printing
- Curing
- De-binding
- Sintering
- HIP\(^1\)
- Heat treatment\(^2\)
- Aging\(^3\)
- Cleaning Surface finishing
- Part inspection
- Finished Part

\(~60-70\%\) \(~99.5\%) 100\%\)

\(^0\) Removal of stresses created by printing process
\(^1\) Removal of porosity and improvement of fatigue resistance
\(^2\) Adjustment of material properties
\(^3\) For specific alloys
Possible process flow

EBM
- Pre-Processes
- Printing
- De-caking / Part removal
- High Pressure Heat Treatment\(^1\) and aging\(^2\)
- Cleaning and Surface finishing
- Part inspection
- Finished Part

Traditional
- ~99.9%
- ~94%

High Speed
- 100%
- 100%

SLM
- Pre-Processes
- Printing
- Stress Relief\(^0\)
- Support removal / Removal from build plate
- High Pressure Heat Treatment\(^1\) and aging\(^2\)
- Cleaning and Surface finishing
- Part inspection
- Finished Part

Traditional
- ~99.9%
- ~96%

High Speed
- 100%
- 100%

BJ
- Pre-Processes
- Printing
- Curing
- De-binding
- Sintering
- High Pressure Heat Treatment\(^1\) and aging\(^2\)
- Cleaning and Surface finishing
- Part inspection
- Finished Part

- ~70%
- ~93%
- 100%

\(^0\) Removal of stresses created by printing process
\(^1\) Removal of porosity and improvement of fatigue resistance
\(^2\) Adjustment of material properties
\(^3\) For specific alloys
Laser PBF Factory of the Future

Today
- Pre-Processes
- Printing
- Stress Relief
- Support removal / Removal from build plate
- HIP
- Heat treatment
- Aging
- Cleaning and Surface finishing
- Part inspection
- Finished Part

Tomorrow
Parts off build plate
- Pre-Processes
- Printing
- Stress Relief
- Support removal / Removal from build plate
- HIP, High Pressure Heat Treatment and aging
- Cleaning and Surface finishing
- Part inspection
- Finished Part

Tomorrow
Parts on build plate
- Pre-Processes
- Printing
- Stress Relief
- Support removal / Removal from build plate
- Cleaning and Surface finishing
- Part inspection
- Finished Part

0. Removal of porosity and improvement of fatigue resistance
1. Removal of stresses created by printing process
2. Adjustment of material properties
3. For specific alloys
Heat Treatment Examples
Comined HIP and heat treatment cycle for In718

- The typical HIP cycle for PBF IN718 according to ASTM F3055-4a that has been combined with the typical heat treatment for this alloy including solutionizing, rapid cooling and a dual temperature ageing according to AMS5663N. As can be seen the full process can be performed in the HIP.
  - It is also possible to take the parts out after the combined HIP, solutionize and rapid cooling step and perform the 18+ hour ageing in another furnace if that is more efficient.
- Micro-hardness measurements showed a benefit of combining the cycles
Recent results confirm the same

<table>
<thead>
<tr>
<th>Post-treatment</th>
<th>Cycle</th>
</tr>
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<tbody>
<tr>
<td>HIP</td>
<td>1200°C / 120 MPa / 4 h / RC (approximately 42 °C/min)</td>
</tr>
<tr>
<td>HIP + HT</td>
<td>1185°C / 170 MPa / 3 h / FC (approx. 13 °C/min) to solutionizing temperature (980 °C)</td>
</tr>
</tbody>
</table>

Solution treatment (ST): 980 °C / 157 MPa / 1 h / RC to HT
Aging: 740 °C / 138 MPa / 8 h / FC to 635 °C / 131 MPa / 10 h / RC to RT

Note: RC, RC, and RT denote furnace cooling, rapid cooling, and room temperature, respectively.

Fig. 17. Microhardness of as-built and post-treated specimens, where BD and TD denote build and transverse direction, respectively.

Effect of post-treatments under hot isostatic pressure on microstructural characteristics of EBM-built Alloy 718

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Direct quench from HIP-temperature from β-region

- Direct quench from HIP-temperature from β-region
  - Heat treatment specifications for TiAl6V4 suggest water quench for maximum strength.
  - Literature and CCT-diagram suggest quench rates in the range of 500-3000 K/min during the phase transition (900° → 750°C)

- Experiment:
  - TiAl6V4 samples were quenched by URC®/URQ®. Gas quench rates in the range of 300-6000 K/min were recorded.
  - Fine acicular ferrite with UTS >1000MPa was reproduced reliably.
HIP above β-transus temperature HIP for Ti-6Al-4V

- Anisotropic microstructure and properties
- Possibility to erase variations and anisotropy with above β-transus HIP (e.g. 1100 °C)?
  - Yes, but fast cooling needed for mechanical properties
- Ongoing work at ORNL

As-printed columnar structure

After HIP with quench equiaxed structure
Conclusions and discussion
Conclusions and discussion

• Heat treatment cycles can be included in the HIP cycles used for AM parts.
• The total AM build process time can be reduced using high pressure heat treatment.
• Improved mechanical properties and the predictability of processes facilitates a robust move from prototyping to production.
• Lean AM Manufacturing™ can be facilitated through the reduction of process steps and improved document handling with modern HIP equipment at the centre of the production chain.
Thank you for your attention

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