DIRECT LASER FABRICATION OF HIGH PERFORMANCE METAL COMPONENTS VIA SLS/HIP

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Abstract

Recent research in the area of direct freeform fabrication of components via selective laser sintering/hot isostatic pressing (SLS/HIP) has focused on the processing of Alloy 625. Alloy 625 is a nickel-based superalloy which provides high temperature strength and corrosion resistance. Alloy 625 test specimens were successfully SLS processed with an integral gas impermeable skin or "can". These samples were subsequently HIPed to high density (>99.5%). Characterization of the test specimens indicated that microstructures similar to conventionally processed P/M materials are achieved in the HIP consolidated "core" region of the parts, while structures similar to those found in cast materials are present in the SLS processed "can" regions. Mechanical analysis of Alloy 625 SLS/HIP parts and production of complex structures will commence shortly.

Introduction

Selective laser sintering/hot isostatic pressing (SLS/HIP) is a hybrid net shape manufacturing technique under development for the production of high performance metal components. SLS/HIP combines the freeform shaping capability of selective laser sintering (SLS) with the full densification capability of hot isostatic pressing (HIP) to produce net shape, high value metal components with significantly reduced costs and lead times in comparison to traditional HIP processing. Components being evaluated for this process include aircraft turbine engine hardware, naval, and submarine components.

A schematic of the SLS/HIP process is shown in Figure 1. As an object is built layer by layer, the laser fuses the metal powder only at the part boundaries to form an integral, gas impermeable skin or "can" to a density exceeding 92% theoretical density. This is the fractional density at which the porosity typically changes from interconnected to closed. The powder in the interior of each layer cross-section can be left unprocessed or optionally laser sintered to an intermediate density. Thus, the component is shaped, canned, evacuated and sealed in-situ. The integrally canned, net shape component produced by SLS is directly post-processed by containerless HIP to full density. A final machining step may be applied if necessary.
SLS/HIP has several advantages over conventional HIP methods. Since an integral skin or "can" is formed of the same material as the component, a secondary canning step is not necessary. Adverse container-powder interactions are eliminated and post-HIP container removal is not required. Tooling and pre-processing steps associated with container fabrication and filling are also eliminated. Therefore, SLS/HIP enables production of complex shapes at reduced cost and in shorter lead-times.

As part of an ongoing research program, SLS/HIP process development is being undertaken for two materials and two demonstration components. Based on a survey of several naval installations, the materials selected for technology development and demonstration are Alloy 625 and Ti-6Al-4V. Prior work on SLS/HIP development for Ti-6Al-4V has been reported elsewhere³. This paper focuses on process development for Alloy 625.

**Experimental**

Alloy 625 is produced by several manufacturers, under a variety of trade names. The fundamental properties and chemical composition are available in test standards such as ASTM B443, B444, and B446. The composition and metallurgy of Alloy 625 is similar to the more commonly used Alloy 718⁴. Its notable properties include high tensile and fatigue strength, as well as excellent corrosion and oxidation resistance. The corrosion resistance is largely provided by the high chromium content of the alloy, while creep strength is provided by formation of a variety of carbides and precipitates (primarily \(\gamma^\prime\)). However, the precise quantity and
distribution of microconstituents can vary greatly depending on the exact composition and thermal history of the material. The supplier selected to provide a powder form of the material was Anval Inc. (301 Route 17 N., Suite 800-T, Rutherford, NJ 07070). The chemical composition and particle size distribution for the supplied powder is listed below.

Table 1. Chemical Composition

<table>
<thead>
<tr>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Fe</th>
<th>Nb</th>
<th>Ti</th>
<th>Al</th>
<th>Si</th>
<th>N</th>
<th>Mn</th>
<th>Co</th>
<th>C</th>
<th>S</th>
<th>P</th>
</tr>
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<td>62.2</td>
<td>21.3</td>
<td>8.22</td>
<td>4.00</td>
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<td>0.27</td>
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<td>0.04</td>
<td>0.028</td>
<td>0.008</td>
<td>0.007</td>
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Table 2. Particle Distribution (Microtrac)

<table>
<thead>
<tr>
<th>Micron</th>
<th>44</th>
<th>31</th>
<th>22</th>
<th>16</th>
<th>11</th>
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<tbody>
<tr>
<td>%&lt;</td>
<td>78.8</td>
<td>40.6</td>
<td>9.4</td>
<td>2.6</td>
<td>1.5</td>
</tr>
</tbody>
</table>

Analysis of the provided powder by SEM and optical microscopy indicates a generally spherical morphology with low internal porosity, as shown in Figure 2.

Figure 2. SEM micrograph of spherical Anval 625 powder (left). Optical Micrograph of polished powder showing low internal porosity (right).

Initial experiments with Alloy 625 were designed to demonstrate the feasibility of constructing integrally canned shapes by SLS suitable for HIP post-processing. To this end, a number of cylindrical specimens were fabricated by SLS. Processing was performed in vacuum using a 250 watt continuous wave Nd:Yag laser. Early test samples showed a high degree of porosity and cracking after processing. The presence of cracks or connected porosity cannot be tolerated in the walls of the part since a gas tight specimen is critical for successful HIPing. Further analysis of the problem indicated that contamination of the powder was a likely culprit. The problem of absorbed gases and other contaminants on powder is well known in traditional powder metallurgy. The removal of contaminants can be accomplished by “degassing” the powder in vacuum. Typically, absorbed water vapor is found in the greatest quantities. The
application of heat to the powder can greatly accelerate the rate of contaminant removal. Measurement of contaminant concentration versus temperature by residual gas analysis (RGA) was performed. Scans for a typical metal powder are shown in Figure 3. A temperature increase as low as 150°C is sufficient to greatly accelerate contaminant removal. Removal of absorbed gases produced a dramatic improvement in alloy flow and densification, as shown in Figure 4.

![Figure 3. Residual Gas Analysis (RGA) data showing water desorption from metal powder with increasing temperature.](image)

Following optimization of the processing parameters, production of cylindrical HIP specimens commenced. A typical specimen, shown in Figure 5, has an outer diameter of 0.5 inches with a wall thickness of 0.125 inches and an unprocessed powder core. A portion of a transverse cross-section of one such cylinder is shown in the photomicrograph of Figure 6. The figure shows a section of the powder core, the right side-wall and the base of the cylinder. The
fractional density varies from 60% at the axis to more than 98.5% at the side-wall. Shown on the left in Figure 7 is a photomicrograph of the interface between the cylinder side-wall and the interior powder core while the photomicrograph on the right shows the interface between the fully dense side-wall and the porous outer layer formed by sintering due to local heat transfer.

Figure 5. A typical as-SLS processed Alloy 625 cylinder.

Figure 6. Transverse cross-section segment of a typical SLS processed Alloy 625 cylinder at the interface between bottom-cap, side-wall and core.

Figure 7. Interface between skin and core (left), between skin and outer layer (right).
Microstructure

Sample parts were sectioned, mounted, and polished for metallographic inspection. Grain structure was revealed by a mixed acids etchant\(^7\) (15 ml HCl, 5 ml acetic acid, and 5 ml HNO\(_3\)). The photomicrograph on the left in Figure 8 shows the grain structure of as-SLS processed fully dense skin in a cross-section transverse to the powder layer orientation. The image height is the equivalent of approximately 7 powder layers, each of thickness 125\(\mu\)m. The microstructure consists of elongated columnar grains oriented vertically in the build direction. The grain orientation indicates that the solidification front originates at the remelted surface of the previously solidified layer, which provides significantly better heat conduction than the loose powder surrounding the part along the sides. No distinct boundaries between individual powder layers are visible. The photomicrograph taken parallel to the powder layers exhibits the expected equiaxed grain structure, as shown on the right in Figure 8.

Figure 8. Etched microstructure of cylinder side-wall, transverse section (left) and longitudinal section (right).

SLS processed Alloy 625 cylinders were post-processed using the following HIP cycle: ramp from ambient temperature to 1150\(^\circ\)C at 15\(^\circ\)C/min, pressurize to 138 MPa (20,000psi) over 30 min, hold at 1150\(^\circ\)C and 138 MPa for 3 hours, ramp down to ambient at 30\(^\circ\)C/min. Figure 9 shows photomicrographs of the powder core taken prior to and after HIP. Image analysis of the unprocessed core indicates a density of 60% prior to HIP. Actual density may be slightly higher, in the range of 65%, due to errors caused by particle pullout during sectioning and polishing of the sample. Following HIP, a core density of 99.5% was achieved. Etching reveals grain boundaries formed largely along prior particle boundaries. The generally spherical geometry of the particles is preserved in the grain structure shown in Figure 9. While this is the expected microstructure for HIP processed parts, the effect of the prior particle boundaries (PPBs) on the mechanical behavior of the parts has not yet been established. The presence of contaminants or undesirable precipitates at PPBs can substantially reduce ductility and tensile strength. Because there is no additional cold working to disrupt the boundaries following HIP, the presence of a continuous impurity film can be quite detrimental. Previous researchers have identified the presence of an Al-rich oxide film as limiting mechanical and corrosion properties in improperly processed material. While great care has been taken during SLS processing to minimize the possibility of contamination, tensile test specimens will be produced in the near
future to verify the mechanical properties of the SLS/HIP processed material.

A second type of cylinder was also processed by SLS. This cylinder, fabricated with an identical 0.5 in. diameter, was processed to near full density across the entire diameter. This type of processing is necessary to fabricate "end-caps" for an integrally canned object made by SLS suitable for HIP post-processing. Metallography revealed that the density in this type of specimen exceeded 98.5% of theoretical density. The etched microstructure, shown on the left in Figure 10 reveals equiaxed grains. This microstructure is quite comparable to that obtained in conventionally cast or annealed wrought materials\(^9\). The SLS processed specimen was post-processed by a HIP cycle consisting of 3 hours at 1240°C and 172 MPa (25000 psi). The microstructure in the HIP post-processed specimen (with density exceeding 99.5% of theoretical) is shown on the right in Figure 10.

Figure 10. Etched microstructure of 98.5% dense SLS processed (left) and 99.5% dense SLS/HIP processed (right) Alloy 625.
Conclusions

Feasibility of fabricating integrally canned complex shapes by SLS for containerless HIP post-processing was demonstrated in Alloy 625. Simple cylindrical shapes were fabricated by SLS and consolidated to full density by HIP. Preliminary evaluation of microstructure reveals that material processed by the SLS/HIP technique is equivalent to conventionally processed P/M material in the part core. The small volume of material processed directly by SLS that forms the can structure is similar to conventional cast structures. Future work will focus on building a part with complex geometry as a technology demonstration for Alloy 625, further characterization of structure/property relationships as well as optimization of processing parameters for build speed, skin thickness and HIP cycle time.

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References