Processing of Titanium Net Shapes by SLS/HIP

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Abstract

SLS/HIP is a hybrid direct laser fabrication method that combines the strengths of selective laser sintering (SLS) and hot isostatic pressing (HIP). SLS can produce complex shaped metal components with an integral, gas impermeable skin. These components can then be directly post-processed to full density by containerless HIP. SLS/HIP is envisioned as a rapid, low cost replacement for conventional metal can HIP processing. The advantages of freeform fabrication combined with *in-situ* HIP encapsulation include the ability to perform containerless HIP, no adverse container-powder interactions, reduced pre-processing time, and fewer post-processing steps compared to conventional HIP of canned parts. SLS/HIP is currently being developed for Inconel® 625 superalloy and Ti-6Al-4V. This paper focuses on microstructure and mechanical properties of SLS processed and HIP post-processed Ti-6Al-4V. SLS/HIP technology for Ti-6Al-4V was demonstrated by fabricating a subscale AIM-9 missile guidance section housing to specification. This work is funded jointly by DARPA and ONR under contract N00014-95-C-0139 titled "Low Cost Metal Processing Using SLS/HIP".

INTRODUCTION

Over the last ten years, SFF technologies worldwide have attained a state of maturity. A variety of SFF technologies are commercially available to produce complex shaped three-dimensional parts and tooling in a variety of materials including plastics, paper, polymers, wax, sand, ceramics and metals. The next major advance in SFF research and development is taking place in the area of direct fabrication processes, especially for low volume production of functional metal, cermet and ceramic components or tooling. Direct fabrication implies layerwise shaping and consolidation of feedstock (e.g. powder, wire, ingot, paste or melt) to complex shapes having full or near full density without the use of intermediate binders, furnace densification cycles, or secondary infiltration steps. These methods typically require minimal thermomechanical post-processing or machining to obtain desired geometry, structure and properties. There is a growing demand amongst industrial and government users for direct SFF processes that enable production of small lot or "one of a kind" functional metal, cermet and ceramic components. A number of direct fabrication methods are under development[1,2,3,4,5,6,7]. The materials systems investigated by these methods include steels, nickel base superalloys, titanium and its alloys, refractory metals, bronze-nickel and cermets.

Selective laser sintering (SLS) is a SFF technique that creates three-dimensional freeform objects directly from their CAD models. An object is created by sequentially fusing thin layers of a powder with a scanning laser beam. Each scanned layer represents a cross section of the object's mathematically sliced CAD model. In direct SLS, a high energy laser beam directly fuses a metal or cermet powder to high density (> 90%), preferably with minimal or no post-processing requirements.

As part of ongoing direct SLS research efforts at the University of Texas, a hybrid net shape manufacturing technique called SLS/HIP is being developed for high performance metal components[8,9]. SLS/HIP combines the freeform shaping capability of SLS with the full densification capability of HIP to produce net shape, high value metal components at significantly reduced costs and shorter lead times. Such components include fighter aircraft turbine engine hardware, naval and submarine components. Since direct SLS had previously demonstrated the potential to consolidate metal powders to net shapes with densities in excess of 80% theoretical density[10], SLS/HIP is envisioned as a natural combination of SLS and containerless HIP to obtain full density metal parts.

The working principle of SLS/HIP is to consolidate the interior of a component to 65% or higher density and to fabricate an integral, fully dense, gas impermeable skin or "can" at the part boundaries *in-situ*[11]. A component is produced by selectively consolidating a metal powder with a laser beam layer by layer. Unlike other direct laser fabrication processes that strive to fuse powder to full density across each layer, in SLS/HIP the laser beam 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 or surface connected to closed[12]. The powder in the interior of each layer cross-section can either be left unsintered so as to provide 65% packing density or be optionally laser sintered to an intermediate density typically exceeding 80% of theoretical density. Thus, the net shape P/M part is shaped, canned, evacuated and sealed *in-situ*. The encapsulated part is then 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 part, a secondary canning step is not necessary. The integrally canned, net shape component produced by SLS is directly post-processed by containerless HIP to full density. 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. SLS/HIP enables production of complex shapes at reduced cost and shorter lead-times.

Under the ongoing research program, SLS/HIP process development is being undertaken for two materials. Based on a survey[8] of several naval installations, the materials selected for technology development and demonstration are the alloys Inconel® 625 and Ti-6Al-4V. The component selected for SLS/HIP technology demonstration of Ti-6Al-4V is the AIM-9 Sidewinder missile guidance section housing, shown in Figure 1.

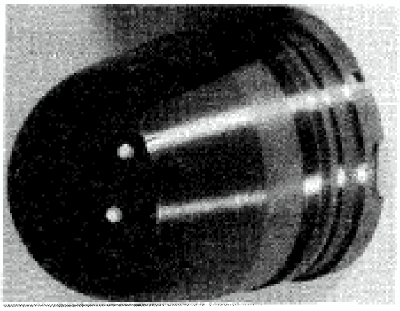

Figure 1. Guidance section housing base for the Sidewinder missile, baseline component for Ti-6Al-4V SLS/HIP technology demonstration.

SLS/HIP PROCESSING

To demonstrate the feasibility of constructing integrally canned shapes by SLS suitable for HIP post-processing, a number of cylindrical specimens, 0.5 inches diameter, 2 inches long with 0.100 inches skin wall thickness were produced by SLS. A typical integrally canned cylindrical specimen is shown in Figure 2. These specimens were post-processed to full density by a HIP cycle consisting of 4 hours at 925° C and 93 MPa (15000 psi).

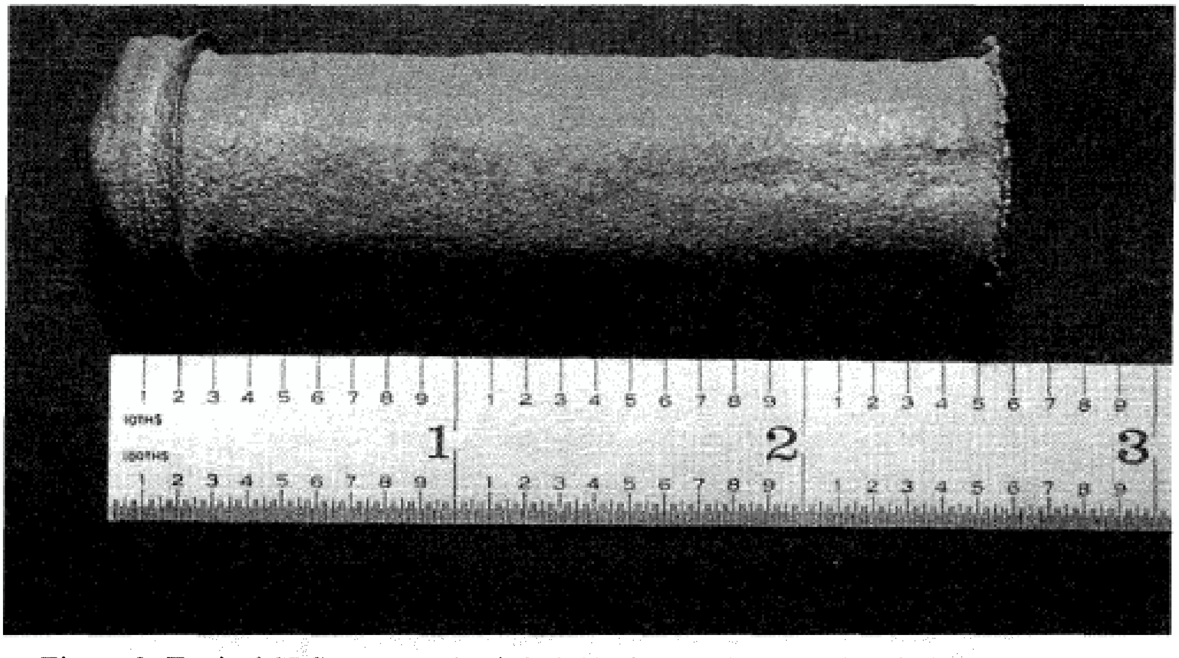

Figure 2. Typical SLS processed, Ti-6Al-4V integrally canned cylindrical specimen.

MICROSTRUCTURE AND MECHANICAL PROPERTIES

To compare the microstructure of SLS/HIP processed Ti-6Al-4V against conventionally canned and HIPed Ti-6Al-4V, a reference specimen from the same lot of powder as that used to fabricate cylindrical specimens was encapsulated in glass and post-processed using the same HIP cycle. The results, shown in Figure 3, indicated that the microstructure of SLS/HIP processed Ti-6Al-4V in the bulk is the same as that of conventionally canned and HIPed Ti-6Al-4V. The skin region of a SLS/HIP processed Ti-6Al-4V part exhibits a microstructure different from that seen in the bulk. This is expected because during SLS, the skin region is completely melted by the laser and undergoes rapid solidification, whereas the interior of the part is left unprocessed, and undergoes densification during HIP post-processing. The micrograph in Figure 4 shows the interface between the skin and the core. The difference in microstructures is clearly evident with the skin region exhibiting a high aspect ratio Widmänstatten type microstructure while the core exhibits a coarser microstructure that results from the coalescence of prior particle grain boundaries.

Figure 3. Etched microstructure of glass encapsulation HIP Ti-6Al-4V (left) and SLS/HIP processed Ti-6Al-4V (right)

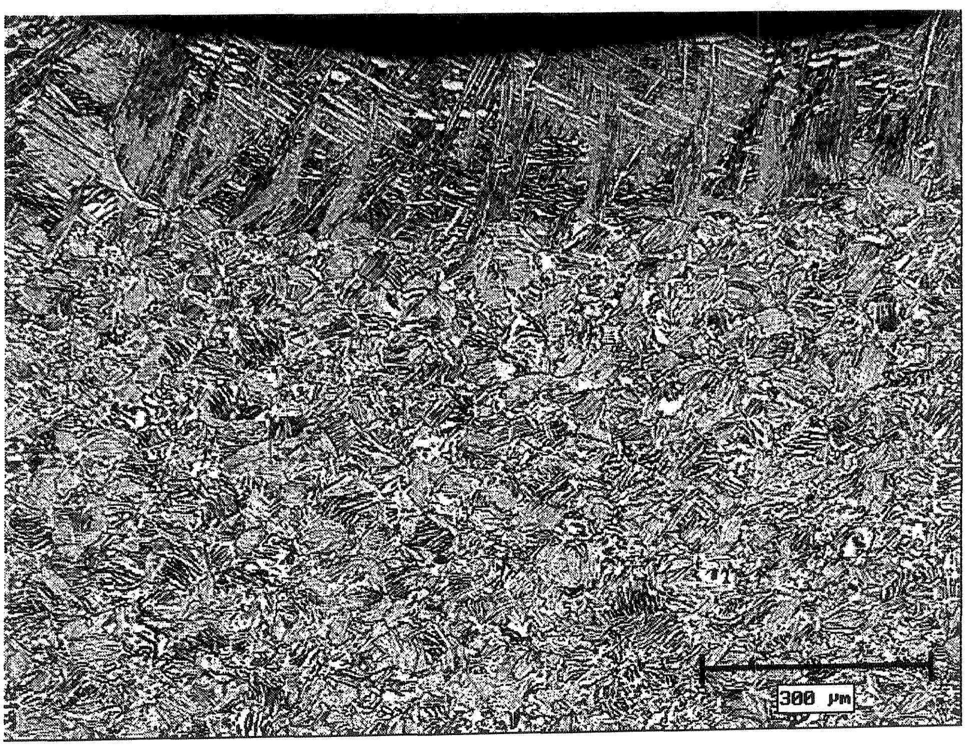

Figure 4. Can-core interface

Hardness was evaluated and compared against conventionally processed material. SLS processing of the skin to full density resulted in material having hardness values comparable to cast material. Hardness values for the SLS/HIP processed core agreed well with values for conventionally canned and HIPed material and did not differ much from the hardness values for the SLS processed skin region.

Material	Processing	Hardness
Ti-6Al-4V	SLS processed skin	36 HRC
Ti-6Al-4V	Cast (grade C-6)[13]	36 HRC
Ti-6Al-4V	Cast (grade C-5)[13]	39 HRC
Ti-6Al-4V	SLS/HIP processed core	35-37 HRC
Ti-6Al-4V	Glass encapsulated HIP	34-36.5 HRC

Table 1. Ti-6Al-4V hardness values.

Tensile specimens conforming to ASTM E 8, round tension test specimen (small-size specimens, 0.25 in. nominal diameter) were machined from integrally canned Ti-6Al-4V cylinders fabricated by SLS and post-processed to full density by HIP. The results indicated that the tensile strength specification for the Sidewinder housing demonstration component was exceeded while the minimum elongation was barely met. Future testing will establish correlations between tensile properties and oxygen levels in SLS/HIP processed parts.

Processing	Tensile Strength (ksi)	Elongation (%)
SLS/HIP	162	5
Sidewinder specification	111	5
Annealed[14]	130	10
Solution treated[15]	150	8
Cast (grade C-5)[13]	130	6
Cast (grade C-6)[13]	115	8

Table 2. Ti-6Al-4V tensile properties.

CHEMICAL ANALYSIS

Oxygen levels in commercially available titanium feedstock and finished product are critical from performance considerations. The oxygen levels can vary from 0.08% to 0.40% by weight depending on the particular application. The strength typically increases with increasing oxygen content at the expense of ductility. Lower oxygen levels improve ductility, fracture toughness, stress-corrosion resistance and resistance to crack growth.

Oxygen and nitrogen levels in SLS/HIP processed Ti-6Al-4V specimens were determined by ASTM E 1409-97 inert gas fusion technique[16]. The levels were compared against levels in the starting powder as well as against specifications for the Sidewinder housing component. The results of analyses conducted by two analytical labs are shown in Table 3. All three SLS/HIP specimens were taken from the core region of each part. The results indicated that the oxygen specification on the Sidewinder housing was barely met. However, it is to be noted that both starting powders (PREP™ and Argon atomized) have 0.19% by weight oxygen and the powder used to fabricate each of the three SLS/HIP specimens had been recycled at least 6 times. This indicates that on average 100 ppm oxygen pickup occurred during each SLS run. This is an extremely encouraging result since it corresponds to the typical oxygen pickup observed by Crucible Research in conventional metal can Ti-6Al-4V HIP processing[17].

To lower oxygen levels in SLS/HIP processed Ti-6Al-4V as well as to increase powder recyclability, ELI (Extra Low Interstitials) grade Ti-6Al-4V powder with 0.099% by weight or lower oxygen may have to be used. In addition, protocols will be established for mixing recycled powder with virgin powder so as to maintain oxygen levels well within specification. During the establishment of such protocols, tracking chronological changes in oxygen levels of parts produced sequentially from the same lot of powder will be an important criterion. ELI grade Ti-6Al-4V powder will be tested in future SLS trials.

Specimen	Oxygen (wt. %)	Nitrogen (wt. %)
Sidewinder specification	0.25 max	-
PREP™ Ti-6Al-4V	0.19	0.01
Ar atomized Ti-6Al-4V	0.196	0.02
SLS/HIP 040898 (Lab A)	0.250	0.019
SLS/HIP 040898 (Lab B)	0.255	0.037
SLS/HIP 042998 (Lab A)	0.2485	0.073
SLS/HIP 042998 (Lab B)	0.200	0.016
SLS/HIP 050198 (Lab A)	0.251	0.038
SLS/HIP 050198 (Lab B)	0.167	0.040

Table 3. Oxygen and Nitrogen levels in Ti-6Al-4V.

TECHNOLOGY DEMONSTRATION

A subscale version of the guidance section housing for the AIM-9 Sidewinder missile was processed by SLS. This demonstration component, shown in Figure 5 was built at 90% scale. The typical length scale on the component shown is 3 inches. Although this component was selected for SLS/HIP technology demonstration, metallographic evaluation of the "as SLS processed" component revealed full density, as shown in the micrographs of Figure 6. This microstructure is consistent with cast Ti-6Al-4V.

Figure 5. SLS processed Ti-6Al-4V demonstration component.

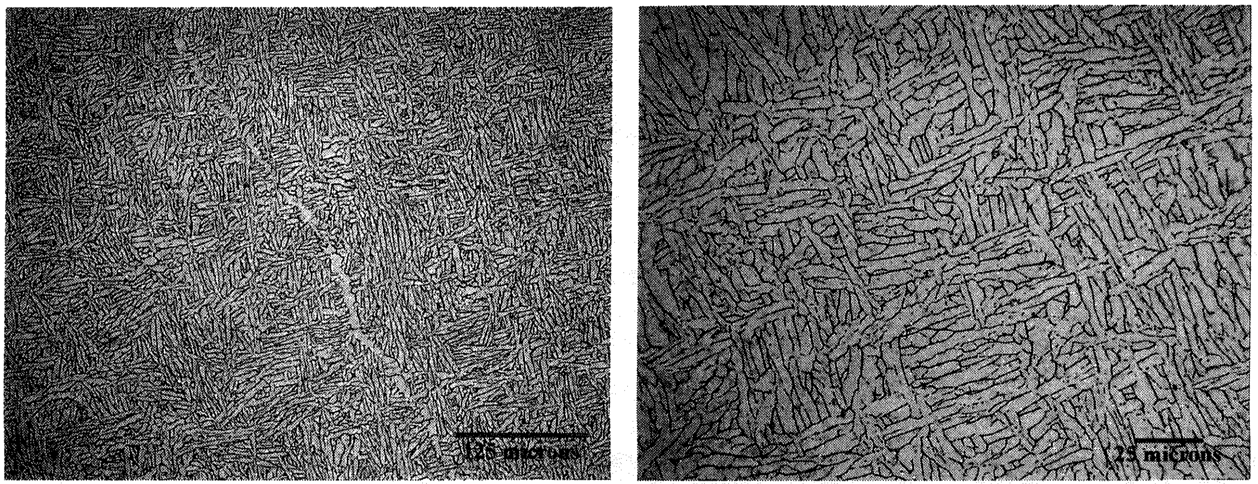

Figure 6. Microstructure of fully dense, SLS processed Ti-6Al-4V demonstration component.

CONCLUSIONS

Feasibility of fabricating integrally canned complex shapes by SLS for containerless HIP post-processing has been demonstrated for Ti-6Al-4V. Simple cylindrical shapes were fabricated and consolidated to full density by HIP. Microstructure and mechanical properties (hardness and tensile) reveal that material processed by the SLS/HIP technique is equivalent to conventionally processed material. Oxygen levels in SLS/HIP processed Ti-6Al-4V meet specification on the component chosen for technology demonstration. A simplified version of a Ti-6Al-4V demonstration component was successfully fabricated.

Future work will focus on demonstrating SLS/HIP technology for Inconel 625, further characterization of structure/property relationships as well as optimization of processing parameters for build speed, skin thickness, oxygen content and HIP cycle time.

ACKNOWLEDGEMENTS

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