Direct Selective Laser Sintering and Containerless Hot Isostatic Pressing for High Performance Metal Components

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Abstract

A novel net shape manufacturing method known as SLS/HIP that combines the strengths of selective laser sintering (SLS) and hot isostatic pressing (HIP) is presented. Direct selective laser sintering is a rapid manufacturing technique that can produce high density metal components of complex geometry with an integral, gas impermeable skin. These components can then be directly post-processed to full density by containerless HIP. The advantages of *in-situ* HIP encapsulation include elimination of a secondary container material and associated container-powder interaction, reduced pre-processing time, a short HIP cycle and reduction in post-processing steps compared to HIP of canned parts. Results of research conducted on Inconel 625 superalloy, Ti-6Al-4V and Monel are presented. This research is funded by DARPA/ONR contract N00014-95-C-0139 titled "Low Cost Metal Processing Using SLS/HIP".

INTRODUCTION

Selective laser sintering (SLS) is a layered manufacturing technique that can produce freeform three-dimensional objects directly from their CAD models without part specific tooling or human intervention. Parts are built by selectively fusing layers of a powder material using a scanning laser beam. Details on this process are described elsewhere[1,2]. Selective laser sintering technology for prototyping parts in a variety of polymeric materials and for creating investment casting patterns has been commercially developed by DTM Corporation. More recently, RapidTool™ technology for creating prototype injection molding tooling has been introduced[3]. This process is an indirect SLS technique that involves the use of polymer coated metal powders to produce a produce a green shape that is subsequently post-processed by binder burnout and infiltration to produce a fully dense object[4,5,6,7,8,9]. Demand for low volume functional metal prototypes at reduced cost and short lead time has spurred the development of so-called direct fabrication processes. A number of such next-generation direct fabrication processes are under development. These processes typically use a concentrated energy source to consolidate and produce a fully dense or nearly fully dense shape directly from constituent materials. The next generation of selective laser sintering, i.e. direct fabrication of functional metal and cermet components and tooling is under development at the University of Texas[10,11,12]. To produce full density metal parts having complex geometry, a novel net shape manufacturing technique called SLS/HIP is under development at the University of Texas. The idea is to consolidate the interior of a component to 80% or higher density and to fabricate an integral gas impermeable skin or "can" at the part boundary *in-situ*. The SLS processed part can then be directly post-processed by containerless HIP to full density. A final machining step will result in a part having the desired geometry and mechanical properties.

BACKGROUND

The Department of Defense has a number of high value, high performance metal components in service that are produced by hot isostatic pressing. These parts are typically produced by conventional HIP of canned metal powders. Shaped metal cans are commonly used to encapsulate metal powders for HIP. The sheet metal container material is chosen so as to minimize interaction with the powder at processing temperatures. The container material must be removed by machining or by chemical methods after HIP post-processing. Properties of the can material including its melting temperature impose processing limits on the shaped metal encapsulation method.

Complex shapes are typically produced using the ceramic mold process[13] developed by Crucible Materials. This process is similar to investment casting in that dry powder instead of molten metal is poured into a ceramic mold. The production of a near net shape is advantageous because it minimizes scrap losses and machining steps. However, outgassing and heating cycles are long during this process because the ceramic mold is surrounded by a large volume of pressure transmitting medium[14]. The long cycle time and pre-processing steps necessary in the ceramic mold method make it a time consuming and expensive process. In addition, non-metallic contamination is also possible.

In the SLS/HIP process (Figure 1), the component is produced by selectively consolidating a metal powder with a laser beam layer by layer. While producing each layer, a gas impermeable high density skin (> 98% density) is formed at the boundaries of the part. The interior of the part is laser processed to a high density typically exceeding 80%. Thus, the part is shaped and canned *in-situ*. The encapsulated part is evacuated, sealed and 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 part is directly post-processed by containerless HIP. Adverse container-powder interactions are eliminated and post-HIP container removal is not required. SLS/HIP allows production of complex shapes at reduced cost and shorter lead-times.

Based upon on a survey of several naval installations[15], four candidate materials have been selected for SLS/HIP process development. These are Inconel® 625, Ti-6Al-4V, 17-4PH stainless steel and Molybdenum. Table 1 below summarizes demonstration components and their applications.

Material	Component	Application
Inconel 625	Engine Vane	Aircraft turbine engine component
Ti-6Al-4V	Housing base	Tactical missile body component
17-4PH SS	Link assembly	Tactical missile launcher component
Molybdenum	Rotary valve	Torpedo component

Table 1 SLS/HIP demonstration components

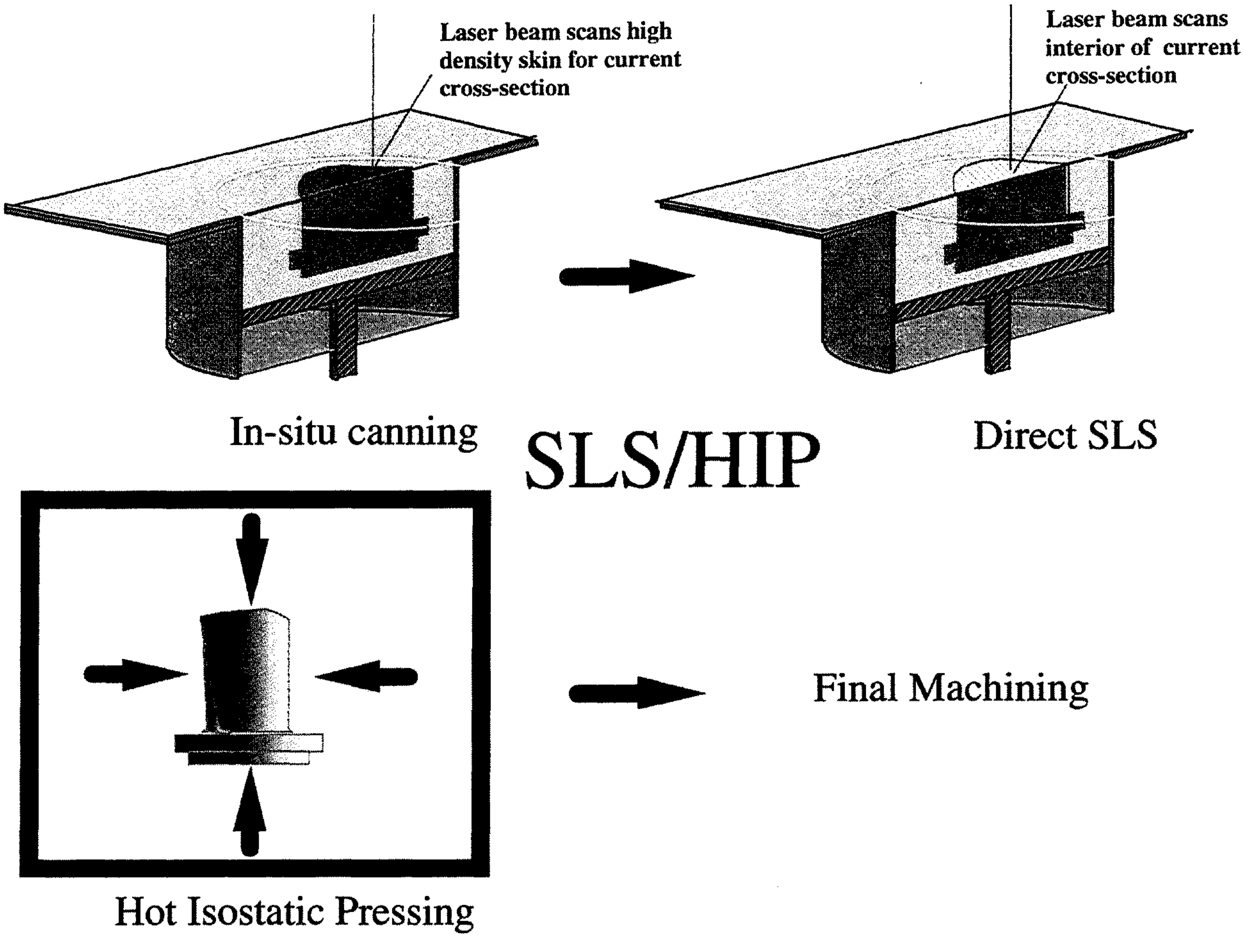

Figure 1 The SLS/HIP Process

EXPERIMENTAL

Candidate powders for screening trials were obtained from Anval Corp. and Nuclear Metals Inc. Anval provided Argon atomized Anval 625 alloy powder (16-44 μm). Ti-6Al-4V (37-74 μm) produced by the PREP method was provided by Nuclear Metals Inc. The compositions of Anval 625 and PREP Ti-6Al-4V are shown in Table 2 and Table 3 respectively.

C	Si	Mn	P	S	Cr	Ni	Mo	Ti	Nb	Al	Co	N	Fe
0.028	0.16	0.04	0.007	0.008	21.3	62.2	8.22	0.27	3.36	0.21	0.04	0.051	4.00

Table 2 Manufacturer's composition of Anval 625

Al	V	Fe	O	C	N	H	Y	Ti
6.35	4.19	0.19	0.19	0.02	0.01	< 0.01	< 0.001	Bal

Table 3 Manufacturer's composition of PREP Ti-6Al-4V

Characterization of the powders used in the SLS/HIP experiments included observation of the powder morphology by SEM and measurement of the powder surface area by gas adsorption surface area analysis (BET method).

Morphology

Both the Inconel 625 and Ti-6Al-4V powders were quite spherical, as would be expected for gas atomized and PREP powders. The high degree of sphericity provided excellent flow characteristics. A free flowing powder is important for SLS since the powder layers must be smooth and uniform. The presence of some particle rearrangement as material is drawn into the melt pool beneath the laser beam has also proven beneficial. The micrographs indicate a dendritic surface structure for both powders, but the presence of some segregation is not problematic since the material is melted and resolidified during the SLS process.

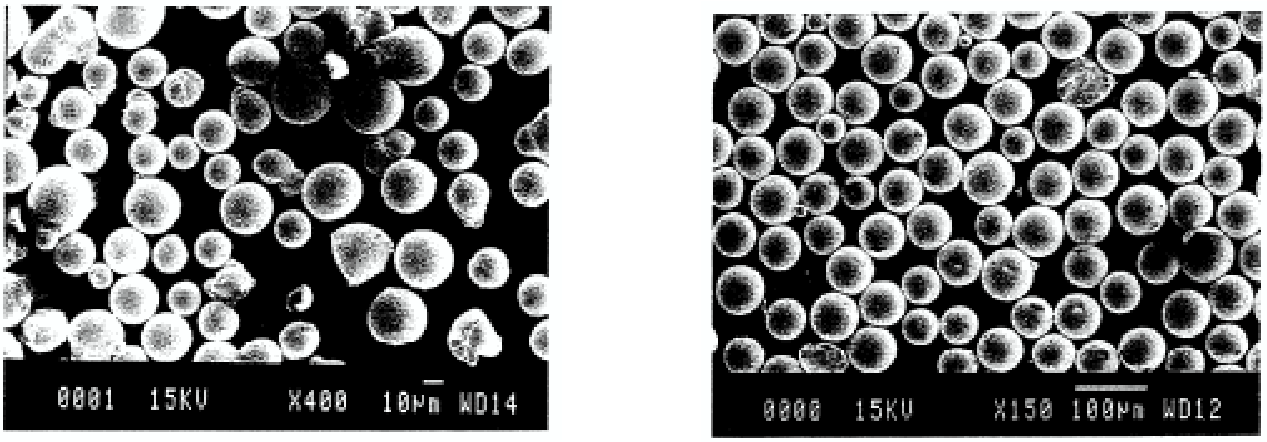

Figure 2 SEM micrographs of -325 mesh Anval 625 (left) and -200 mesh PREP Ti-6Al-4V (right)

Surface Area Analysis

The surface area of the powder is of greater concern, since the material must be thoroughly degassed prior to SLS processing. There are indications that the presence of oxide formation during SLS processing is a significant factor in influencing both the mechanical properties and the part density following the SLS stage. A 5-point BET surface area analysis (Table 4) was performed on the powder using a Micromeritics ASAP 2010 Surface area Analyzer. Samples of Inconel 625 and Ti-6Al-4V weighing 26 gm and 18 gm respectively were degassed at 350° C for 24 hours.

Material	Predicted Area (m^2/g)	Measured Area (m^2/g)
Inconel 625	0.035	0.052
PREP Ti-6Al-4V	0.034	0.0686

Table 4 BET Surface area analysis

Leak testing

To screen specimens produced by SLS for impermeability, a leak testing apparatus and procedure was adapted from the Metals handbook article on containerless HIP[16]. For a specimen to be acceptable for HIP, a gas impermeable barrier must be provided. A helium leak rate less than 1 X 10^{-9} standard cm^3/s is considered acceptable. Such low leak rates are required because the leak rate at typical a HIP pressure of 1000 atm (100 MPa) will be five orders of magnitude greater than that during leak testing at 1 atmosphere.

Feasibility demonstration

To demonstrate feasibility of constructing a thin walled can acceptable for HIP, a rectangular geometry was chosen. The procedure established to qualify specimens and to refine SLS processing parameters is shown in the flowchart of Figure 3. A schematic of the test geometry is shown in Figure 4.

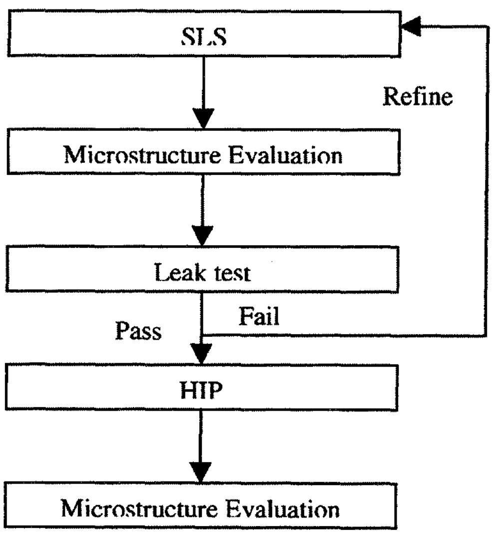

Figure 3 Specimen qualification procedure

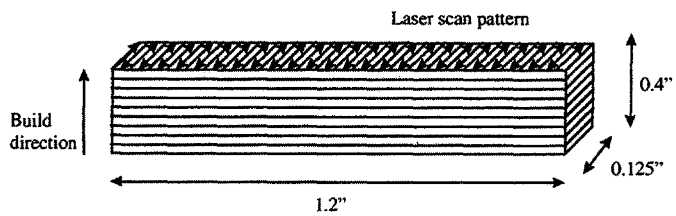

Figure 4 Thin wall specimen schematic

SLS apparatus

SLS trials were conducted on a high temperature selective laser sintering machine designed and built at the University of Texas. This machine is equipped with a 250 Watt Nd:YAG laser, powder preheating capability up to 600° C and controlled atmosphere.

HIP apparatus

HIP trials were conducted on a ABB model QIH-3 equipped with a graphite heating element. Temperatures up to 2000° C and pressures up to 200 MPa are attainable with the graphite element in inert atmosphere. Argon gas was used as the pressure transmitting medium.

RESULTS AND DISCUSSION

To demonstrate feasibility of constructing gas impermeable thin walls by SLS, specimens conforming to the geometry of Figure 4 were produced. These were then leak tested and screened according the procedure in Figure 3. Shown on the left in Figure 5 is the cross-section of a thin wall specimen produced by SLS. This specimen passed the leak test with a helium leak rate of less than 1 X 10 $^{-10}$ standard cm^3/s. This specimen was post-processed by a HIP cycle consisting of 1 hour at 1100 °C and 30 mTorr pressure followed by 45 minutes at 1100° C at 66 MPa. A cross-section of the HIPed specimen is shown on the right. Average porosity was measured over a metallographic montage in as SLS processed and HIPed specimens revealing a decrease in average porosity from 4.5% to 3.7%.

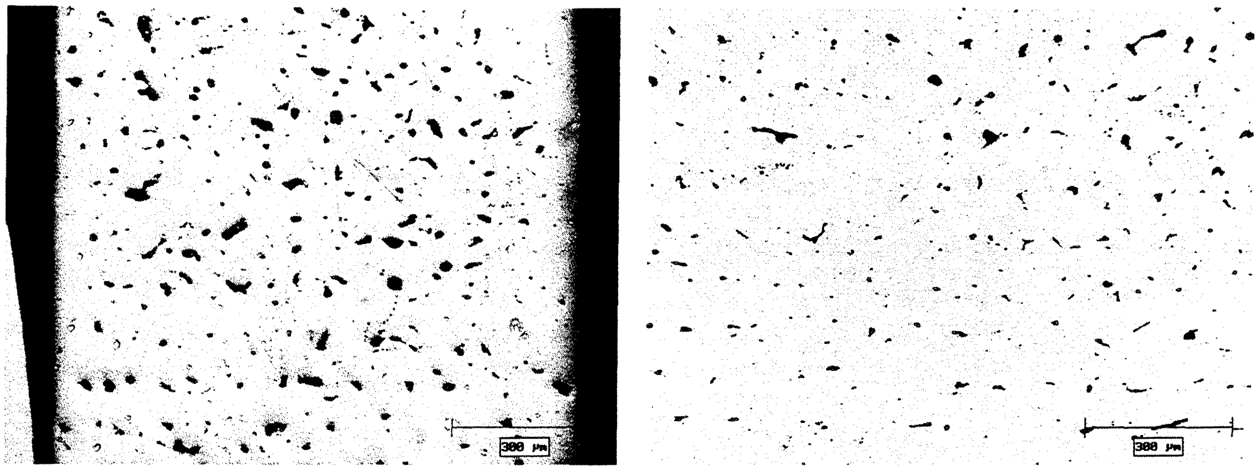

Figure 5 Inconel 625 Specimen 121496, as SLS processed (left) and HIP post-processed (right)

The next step in SLS/HIP process development was to demonstrate capability of constructing a simple shape. A 0.5 inch diameter, 0.5 inch long cylinder was chosen for this purpose. Shown in Figure 6 is an axial cross-section of an Inconel 625 cylinder processed by SLS to 98.5% density. This specimen was post-processed by a HIP cycle consisting of 3 hours at 1240 ° C and 25000 psi. This cycle resulted in nearly full densification to 99.5%. A cross-section of the HIPed specimen is shown in Figure 6 Specimen 051797, as SLS processed, 1.5% porosity . Temperature and pressure HIP maps (Ashby) were generated using software developed by one of the authors (Bourell). The objective was to correlate experimentally observed density with predicted density. These maps show that for Inconel 625 parts with initial density in excess of 98%, the dominant mechanism of densification at temperatures higher than 1100° C and pressures in excess of 100 MPa (15 ksi) is power law creep. The maps predict that HIPing conditions of 1100° C and 100 MPa should be sufficient to densify Inconel 625 in excess of 98% density in 1 hour. To ensure full density, a more aggressive HIP cycle consisting of 3 hours at 1240^0 C and 165Mpa (25ksi) was employed.

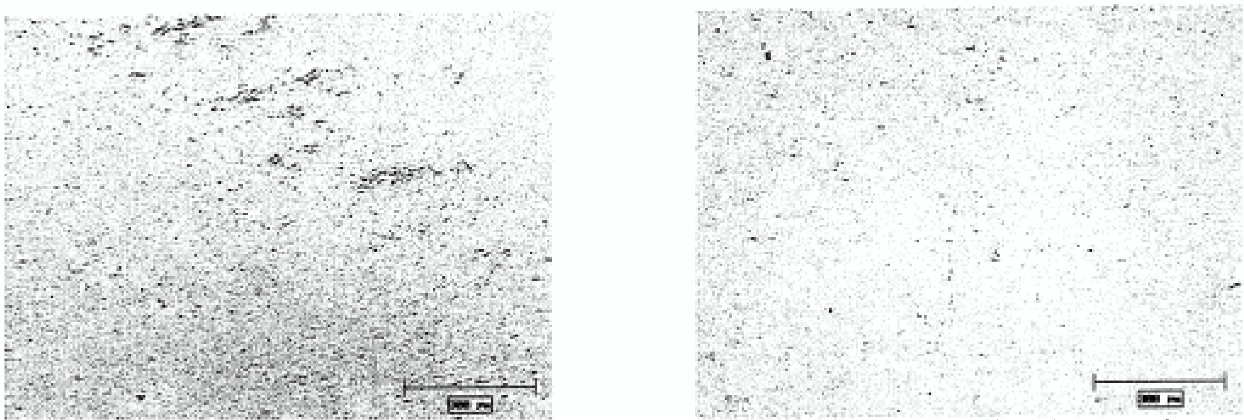

Figure 6 Specimen 051797, as SLS processed, 1.5% porosity (left) and HIPed, 0.5% porosity (right)

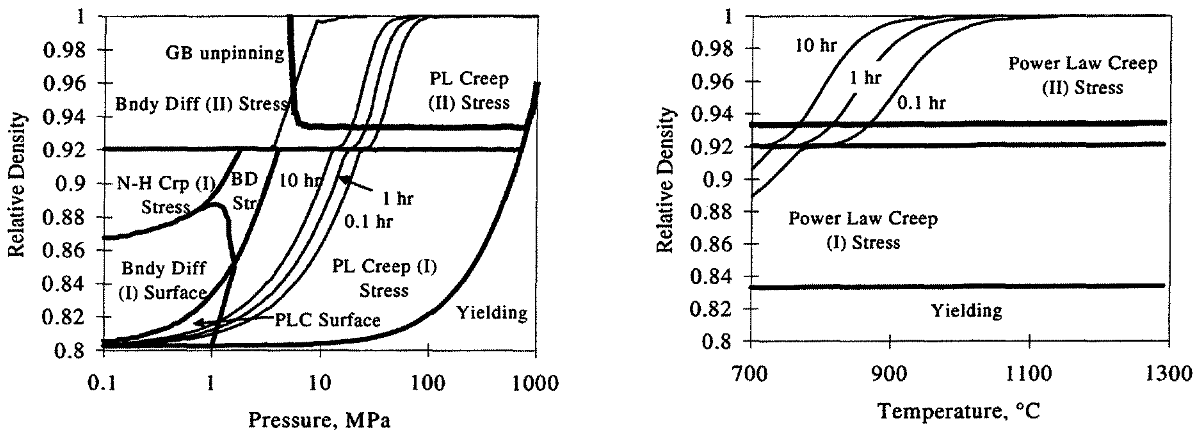

Figure 7 Pressure HIP map (at 1240° C) for Inconel 625 (left) and temperature HIP map (at 165 MPa) for Inconel 625 (right)

Single layer screening trials were conducted on candidate materials Ti-6Al-4V, Monel (65%Cu-35%Ni) and Ti-14Al-21Nb, a Titanium Aliminide. Shown in Figure 8 is a cross-section of a PREP Ti-6Al-4V single layer (0.15 cm thickness) produced by SLS. This specimen successfully passed the leak test with a helium leak rate less than 1 X 10^{-10} standard cm^3/s. Shown in Figure 9 are cross-sections of Ti-14Al-21Nb and Monel single layer specimens demonstrating full density capability.

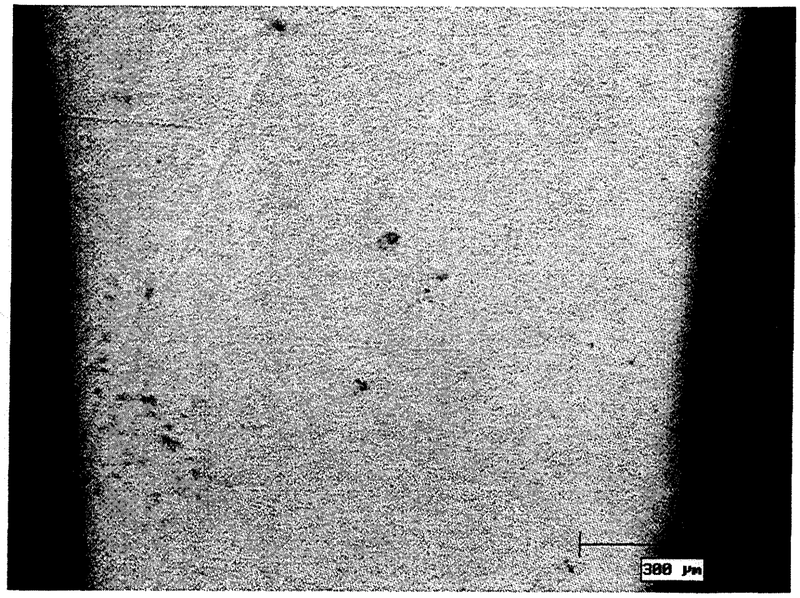

Figure 8 PREP Ti-6Al-4V, single layer, as SLS processed

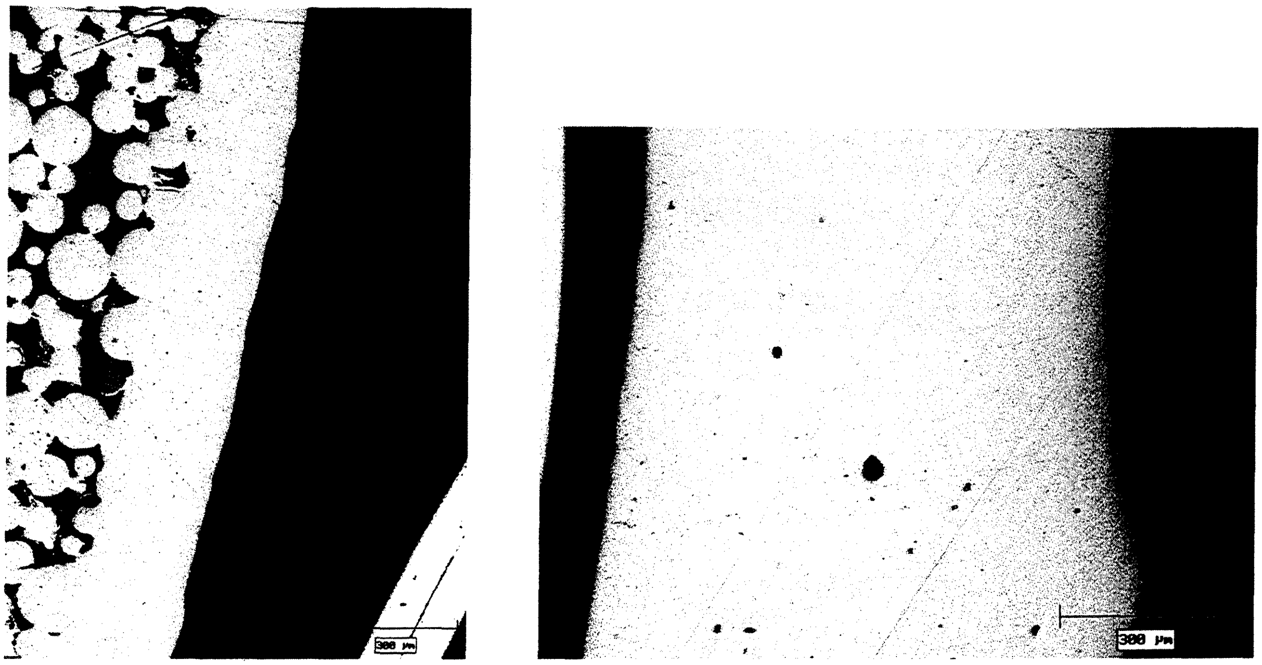

Figure 9 Single layer specimens of PREP Ti-14Al-21Nb (left) and Monel (right)

CONCLUSIONS

The feasibility of SLS/HIP has been successfully demonstrated for a simple cylindrical shape made of Inconel 625 superalloy. Preliminary screening trials on PREP Ti-6Al-4V indicate that a gas impermeable skin can be produced by SLS. Research is ongoing to qualify additional materials including Monel, Ti-14Al-21Nb, Molybdenum and 17-4PH stainless steel. The next step in SLS/HIP process development will be to demonstrate a complex geometry part. SLS/HIP has tremendous potential for rapid, net shape manufacture of high performance metal components at reduced cost and shorter lead times.

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REFERENCES

[i] Beaman, Joseph J. and Deckard, Carl R., *Solid Freeform Fabrication and Selective Powder Sintering,* 15th NAMRC, North American Manufacturing Research Conference Proceedings, 1987, pp. 636-640.
[2] Deckard, C.R., Ph.D. Dissertation, Department of Mechanical Engineering, The University of Texas at Austin, 1988.
[3] *Rapid Prototyping and the Selective Laser Sintering Process : Tooling*, product literature, DTM Corporation, Austin, Texas.
[4] Vail, N.K., *Preparation and Characterization of Microencapsulated, Finely Divided Ceramic Materials for Selective Laser Sintering*, Ph.D. dissertation, Department of Chemical Engineering, The University of Texas at Austin, 1994.
[5] Vail, N. K., Barlow, J.W. and Marcus H.L., *Silicon Carbide Preforms for Metal Infiltration by Selective Laser Sintering of Polymer Encapsulated Powders*, Solid Freeform Fabrication Symposium Proceedings 1993, The University of Texas at Austin, pp. 204-214.
[6] Deckard, Lucy and Claar, Dennis T., *Fabrication of Ceramic and Metal Matrix Composites from Selective Laser Sintered Preforms*, Solid Freeform Symposium Proceedings 1993, The University of Texas at Austin, pp. 215-222.
[7] Stucker, Brent E., Bradley, Walter L., Norasetthekul, Somchin (Jiab) and Eubank, Phillip T., *The Production of Electrical Discharge Machining Electrodes Using SLS: Preliminary Results*, Solid Freeform Fabrication Symposium Proceedings 1995, The University of Texas at Austin, pp. 278-286.
[8] Bampton, C.C. and Burkett, R., *Free Form Fabrication of Metal Components and Dies*, Solid Freeform Fabrication Symposium Proceedings 1995, The University of Texas at Austin, pp. 342-345.
[9] Wohlert, M. and Bourell, D., *Rapid Prototyping of Mg/SiC Composites by a Combined SLS and Pressureless Infiltration Process,* Solid Freeform Fabrication Symposium Proceedings 1996, The University of Texas at Austin.
[10] Fuesting, T., L. Brown, S. Das, N. Harlan, G. Lee, J. J. Beaman, D. L. Bourell, J. W. Barlow and K. Sargent, *Development of Direct SLS Processing for Production of Cermet Composite Turbine Sealing Components-Part I,* Solid Freeform Fabriaction Symposium 1996 Proceedings, pp. 39-46.
[11] Fuesting, T., L. Brown, S. Das, N. Harlan, G. Lee, J. J. Beaman, D. L. Bourell, J. W. Barlow and K. Sargent, *Development of Direct SLS Processing for Production of Cermet Composite Turbine Sealing Components-Part I,* Solid Freeform Fabriaction Symposium 1996 Proceedings, pp. 47-55.

[12] Das, S., N. Harlan, J. J. Beaman and D. L. Bourell, *Selective Laser Sintering of High Performance High Temperature Metals,* Solid Freeform Fabrication Symposium 1996 Proceedings, pp. 89-95.
[13] *Hot Isostatic Pressing of Metal Powders,* Metals Handbook, Vol. 7, 9th Edition, pp. 425-426.
[14] Atkinson, H. V., and B. A. Rickinson, *Hot Isostatic Pressing*, Adam Hilger: Bristol, England, 1991, pp. 64-65.
[15] Knight, Ronald, Joe Wright, Joseph Beaman, and Douglas Freitag, *Metal Processing Using Selective Laser Sintering and Hot Isostatic Pressing (SLS/HIP),* Solid Freeform Fabrication Symposium 1996 Proceedings, pp. 349-353.
[16] *Hot Isostatic Pressing of Metal Powders,* Metals Handbook, Vol. 7, 9th Edition, pp. 436.