Selective Laser Sintering of High Performance High Temperature Materials

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Abstract

Hot Isostatic Pressing (HIP) of high performance metal parts is currently done using either a shaped metal container or a pre-fabricated ceramic mold depending on the part material and geometry. An alternative method of HIP encapsulation that allows complex part geometry, short cycle time and minimal potential for container-powder bed interaction is desired. Integral, fully dense metal skins with complex geometry can potentially be constructed by direct selective laser sintering (SLS). The advantages of in-situ HIP encapsulation by direct SLS include the elimination of a secondary container material and any associated container-powder bed interaction, reduced pre-processing time and a short HIP cycle. Single and multi-layer specimens of Inconel 625, Ti-6Al-4V and 17-4 PH stainless steel were produced by direct SLS. Closed porosity in Inconel 625 and 17-4 PH stainless steel samples ranged from 0 to 12% and area porosity from 0.5 to 20%, depending on the laser energy density. Direct SLS samples of Inconel 625 were subjected to helium leak testing and found to be impervious, with a leak rate less than $1x10^{-10}$ atm cc/s. These samples met the criteria for containerless hot isostatic pressing.

OBJECTIVE

An in-situ canning technique using direct selective laser sintering (SLS) is being developed for hot isostatic pressing (HIP) of high performance components. The process developed should be an integral canning technique with near net shape capability for complex parts and require minimal processing steps. The integral can must be leak free and cannot interact adversely with the powder bed. The process should not be limited by the can properties. The objective of this research is to produce highly dense skins of high performance metals acceptable for containerless HIP. Containerless HIP requires an outer shell with a helium leak rate[1] of less than 10^{-9} std cc/sec.

CONVENTIONAL HIP TECHNIQUES

Shaped Metal Can

Shaped metal cans are commonly used to encapsulate metal powder for HIP. They are largely made of low carbon steels, stainless steels, titanium and titanium alloys based on the powder to be processed and processing conditions. The container material is chosen such that minimal interaction occurs between the container and the powder at the processing temperature. Encapsulation containers are processed from sheet metal using standard metal working processes[2]. Welded joints demand special attention, as they are a common point of failure during the HIP cycle. Container fabrication becomes expensive for part geometry more complex than a simple cylinder, thus sheet metal encapsulation is limited to generally simple designs. After HIP, the container must be removed by either machining or chemical methods. The process limits imposed by the properties of the canning material, such as its melting temperature, are a drawback to the shaped metal encapsulation method.

Ceramic Mold Process

More complex shapes are typically produced using the ceramic mold process[3]. This process is similar to investment casting, except dry powder is poured into a ceramic mold instead of molten metal. 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[4]. The long cycle time and pre-processing steps necessary in the ceramic mold method make it a time consuming and expensive process. Non-metallic contamination is also a possibility.

SLS/HIP

In the proposed direct SLS/HIP process, an impermeable high density skin is formed around the shape of a complex part by selective laser sintering. The interior of the part is laser sintered to intermediate density. The encapsulated partially sintered part is evacuated, sealed and processed by HIP to full density. This process can be followed by final machining, if necessary. Figure 1 shows a schematic of the process.

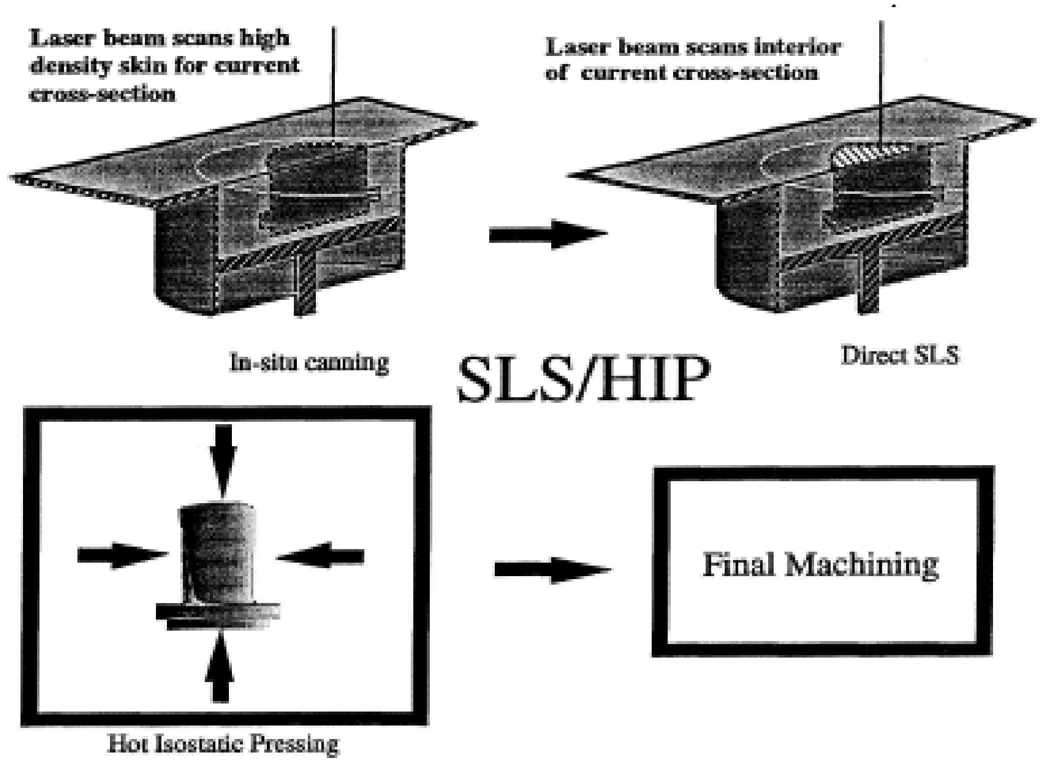

Figure 1. SLS/HIP Process

The direct SLS/HIP method has several advantages over conventional HIP methods. Since an integral skin is produced, the subsequent HIP process is considered containerless. There are no secondary canning steps, no adverse container-powder reactions and no container removal steps. A long out-gassing/heating cycle is not necessary. SLS/HIP allows production of complex shapes that cannot be achieved through standard sheet metal processing for a reasonable cost. As in the ceramic mold method, a near net shape is produced, minimizing scrap and machining steps. SLS/HIP has clear advantages over both the shaped metal can and ceramic mold HIP techniques.

EXPERIMENTAL

A computer controlled, high power Nd:YAG laser was used to process metal powders by selective laser sintering. Materials used in screening trials included spheroidized Inconel 625 (Anval Corp.), a flaked elemental blend of Ti-6Al-4V (Micron Metals, Inc.) and a spheroidized 17-4 PH stainless steel powder (Ametek Corp.), each with a -325 mesh particle size.

Laser energy density[5] was varied between 1000 J/cm^2 and 2500 J/cm^2 as defined by the Andrew number equation,

$$A_N = \frac{P}{v \cdot \delta} (J/cm^2)$$

where

P is the incident laser power (Watts)

v is the laser scan speed (cm/s)

δ is the scan spacing (cm)

Samples prepared by direct SLS were cross-sectioned and prepared for optical microscopy. Closed porosity measurements were made using an AccuPyc 1330 gas pycnometer by comparing measured density to theoretical density. Area porosity was calculated from the optical micrographs.

To successfully HIP an object to full density, a barrier must be provided to prevent pressurized gas from entering the porous body. Due to high pressures involved in HIP (typically of the order of 15 ksi), openings as small as 0.1 μm can produce unacceptable leak rates. Leak testing is commonly performed on samples prior to HIP to ensure successful compaction. The impermeability test screening and procedure for integral can SLS parts is based on the Metals Handbook article on containerless HIP[6]. As a general guideline, the maximum helium leak rate through the barrier must not exceed 10^{-9} cc/s at atmospheric pressure. Such a low leak rate is required because at typical HIP pressures, the leak rate will be 1000 times greater than that at ambient pressures. Shown below is a schematic of the leak test apparatus used to test laser sintered Inconel 625 samples.

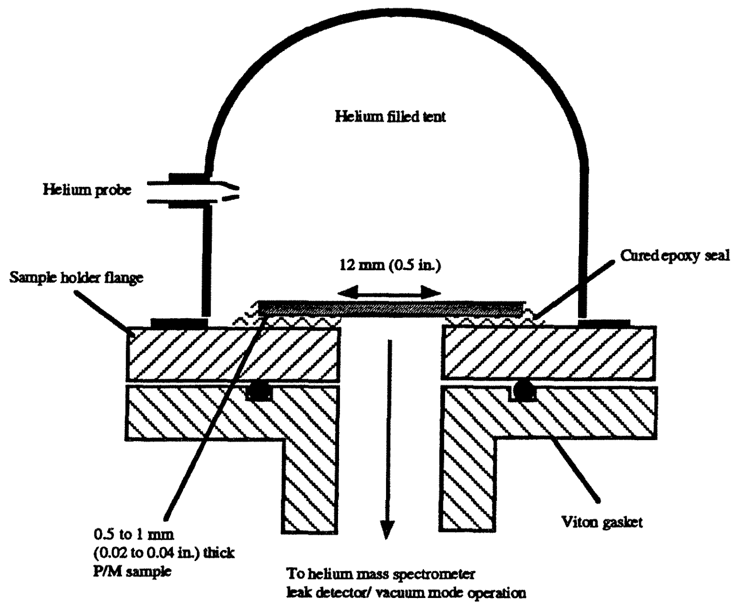

Figure 2. Leak Testing Apparatus

RESULTS AND DISCUSSION

Inconel 625

Closed porosity on the Inconel 625 coupons ranged from 0% to 10%. Area porosity measurements ranged from 4% to 20%. A small amount of closed porosity is acceptable in the integral skin because it does not contribute to leakage through the skin. Micrographs of the samples sintered under a highly inert environment are shown in Figures 3 and 4. The energy density used to sinter the sample is listed in the caption.

Leak testing showed that the Inconel 625 specimens are impervious to helium, with a leak rate of less than 1×10^{-10} atm cc/s. This leak rate is an order of magnitude lower than that acceptable for containerless hot isostatic pressing.

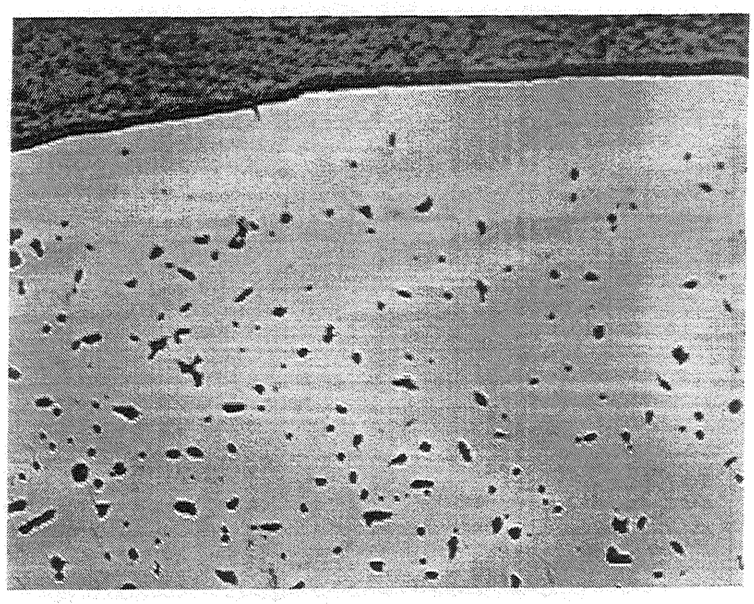

Figure 3. Inconel 625, Energy Density = 2230 J/cm^2, 1.8 mm viewfield, 100X

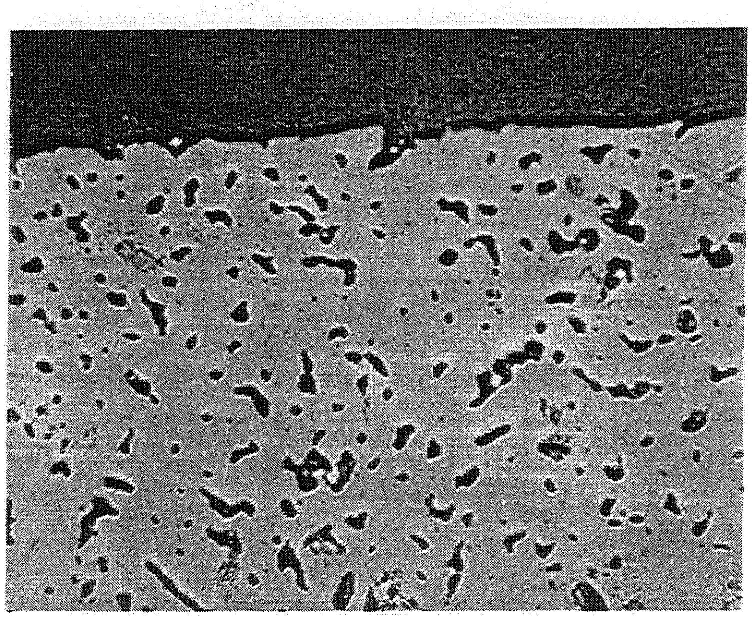

Figure 4. Surface of Inconel 625, Energy Density = 2134 J/cm^2, 1.8 mm viewfield, 100X

Ti-6Al-4V

The titanium alloy tended to ball during SLS processing. Rather than melting into a smooth layer, the material tended to form clumps of dense regions. A micrograph of a dense region is shown in Figure 5.

Closed porosity was measured as 1% to 3% for the entire sample. Area porosity was measured as 2.5% in a dense region.

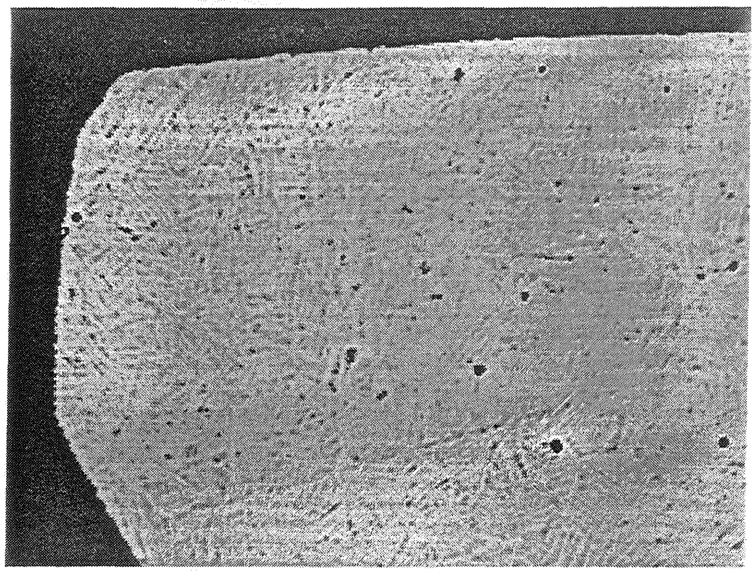

Figure 5. Ti-6Al-4V, Energy Density = 1061 J/cm^2, 1.8 mm viewfield 100X

17-4 PH Stainless Steel

Closed porosity measurements on 17-4 PH SS coupons prepared by direct SLS ranged from 3.2% to 12.2%. Area porosity was measured as 0.9% and 3.6% for a band across the sample. The porosity level in the 17-4 PH samples is acceptable for the production of an integral skin because it is mostly closed porosity. A typical micrograph of one of the samples is shown in Figure 6.

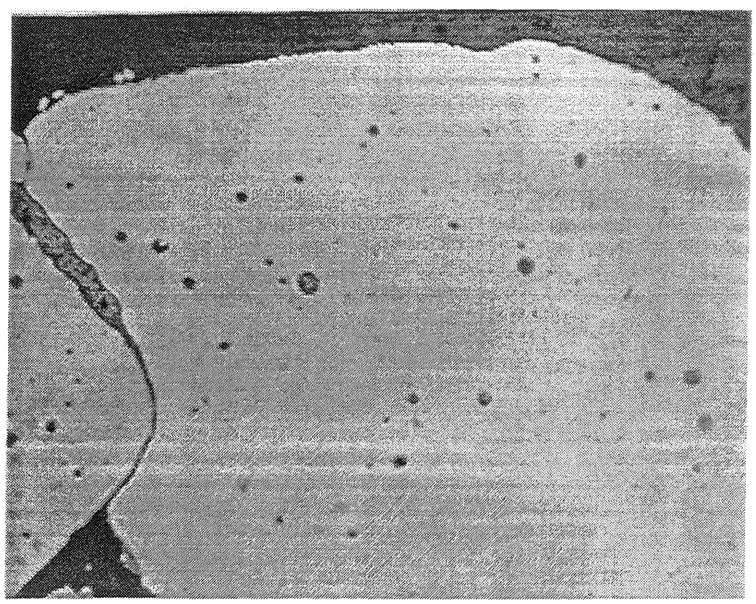

Figure 6. 17-4 PH SS, Energy Density = 1045 J/cm^2, 1.8 mm viewfield, 100X

The micrograph shows that several scan line widths of material flowed together to form larger bands. These bands are almost fully dense, exhibiting only 0.6% area porosity.

CONCLUSIONS

Preliminary materials screening studies to date on Inconel 625, Ti-6Al-4V and 17-4 PH stainless steel indicate the feasibility of constructing integral HIP containers by direct SLS. Samples of Inconel 625 produced by direct SLS meet the leak rate requirements for containerless hot isostatic pressing. Optical micrographs from the screening trials for Inconel 625 and 17-4 PH stainless steel show mostly closed porosity.

ACKNOWLEDGMENTS

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