Direct Laser Sintering of Metals

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Abstract

The use of a directed laser beam source to selectively sinter multiple layers of binderless metal powder for the purposes of rapid prototyping is described. The work in this paper is restricted to -325 mesh iron powder, which was sintered using a CW 50 W Nd:YAG laser to approximately 35% density. A subsequent post-treatment was performed to achieve a fully dense sample. It is envisioned that such a system can be used to manufacture *functional* metallic prototypes directly from CAD without part-specific tooling.

1 Background

1.1 Rapid Prototyping

The use of rapid prototyping in a concurrent engineering environment results in reduced product development cycle time-a natural consequence of the quick availability of working testable hardware. A functional prototype allows the identification of design deficiencies or areas for design improvement; it allows experimental stress analysis, vibrations testing, and other design performance tests with a very low lead-time. In some cases, experimental analysis of prototypes has proven to be less expensive than computer-based analysis techniques such as finite element analysis [1]. A common characteristic of rapid prototyping methods is that no part-specific tooling, such as a mold or die, is required to make the part. Several reviews of rapid prototyping are available in the literature [2][3][4].

Use of rapid prototyping technology is expected to grow as more materials can be processed using the techniques. Many systems are commercially available; the most popular technique to date is *stereolithography*, which generates a part from a bath of laser-cured photopolymer resin. *Selective laser sintering*, the topic of this paper, is gaining popularity; it generates a part from multiple layers of powder. Commercial systems for SLS are currently limited to ABS plastic, wax, nylon and polycarbonate materials. To date, no commercial freeform fabrication system is available for metallic materials. The preliminary results of an effort to address this issue are described in this paper.

1.2 Selective Laser Sintering (SLS)

SLS generates a part by selectively bonding multiple layers of powder to build a threedimensional part in a layer-by-layer manner. The process has been commercially applied to ABS plastic, nylon, polycarbonate, and wax[5][6][7]. The process is very appealing for applications involving metals and ceramics. It is anticipated that SLS of metals will be less expensive and less time-consuming than conventional production methods for metals, such as sand casting or investment casting, when the number of parts required is reasonably small. Therefore, the system will be used by designers and engineers during the prototyping stages of product development. A complete SLS apparatus will allow designers to quickly generate three-dimensional *functional* prototypes of various parts. The process is also attractive to builders of custom tooling, dies and molds since these industries represent one-of-a-kind applications.

Though there are no commercial systems for applying this technique to metals, research is under way at the University of Texas at Austin.¹ One of their techniques is to coat the metal particles with a binder; the binder is selectively cured with a laser, and the part is later fired to burn out the binder and densify the part. Parts of final densities of 48% [8] and higher have been reported. Other work is ongoing to directly sinter metals by using a liquid-phase metallic material to fill the voids in a solid-phase powder, such as copper in nickel. Higher densities (82%) have been reported using this technique [9].

The approach reported here differs in that the metal powder is sintered directly, without the introduction of a binder or a low-melting-temperature liquid phase. A two-step process is proposed: The laser will not be used to generate a fully-dense final part; instead, the laser apparatus will be used to generate a "green" part that requires subsequent processing such as *Hot Isostatic Pressing* (HIPing), oven firing, or infiltration. The goal of this effort is to assess the process by laser sintering a simple cubic sample of iron, performing a HIP densification treatment, and evaluating the density of the resulting material.

2 Experimental Apparatus

A laboratory scale system was constructed for evaluation of the SLS process. The system is capable of making parts up to 1 in. by 1 in. by 3 in. using laser heating.

¹The SLS process is patented by the University of Texas in Austin and has been reduced to practice by DTM, a subsidiary of B.F. Goodrich.



Figure 1: Powder handling system.

2.1 Powder Handling System

The powder handling system is shown schematically in Figure 1. This system includes two stepper motors: one to control the height of an elevator under the working section, another to control the location of a powder shuttle. To add a layer of powder, the elevator is lowered the desired distance, and the shuttle is moved until the actuator opens a powder metering device. A measured amount of powder is dropped through the transfer plate and into the shuttle transport chamber. The shuttle then returns to its home position. As the shuttle returns, a layer of powder is spread over the working section. Excess powder is dumped into the waste bin. The shuttle stops momentarily, completely covering the working section, and the powder is compacted by temporarily raising the elevator.

During a laser sintering experiment, the powder handler is housed in a glove-box with an over-pressure of inert gas or forming gas to avoid oxidation of the metal powder. Controllable parameters include:

- Powder composition, size and shape.
- Laser power (0 to 50 W)
- Laser spot size (>1 mm dia.)
- Scan speed (<20 in./min.)
- Layer thickness
- Process environment (inert gas, forming gas, etc.)

2.2 Laser System

The laser used in this process is a 50 W continuous wave (CW) neodymium:yttriumaluminum-garnet (Nd:YAG) laser with a computer-controlled shutter. The 1.06 μm wavelength laser beam is channeled through a 600 μm single core step index quartz fiber optic to the powder handling system. At the terminal end of the fiber optic, an output coupler and a lens focus the beam to approximately 0.5 mm diameter on the metal powder. The Nd:YAG laser is the better laser choice when compared with a CO₂ laser because the 1.06 μ m heat source couples with metals more efficiently than a 10.6 μ m heat source. See Figure 2. The Nd:YAG laser has the further advantage that, at higher powers, the beam is efficiently transmitted through fiber optics. This allows for more flexible SLS machine design.

The output coupler assembly is mounted on an x-y table, which is translated via two additional stepper motors. The laser's shutter and the four stepper motors (two in the powder handling system and two in the x-y table) are controlled using a single 80386 computer.

2.3 Secondary Processing (HIPing)

The green form generated using the SLS system requires a post-treatment to render a fully dense part. Since the part will, in general, be quite brittle and will possibly contain many small sections, some means of supporting the part during the treatment is required. Furthermore, it is expected that the green structure will contain interconnected porosity that makes conventional gas HIPing impossible. The following technique was successfully implemented to HIP laser-sintered specimens:

- 1. The specimen was suspended in alumina powder in a Pyrex tube.
- 2. The tube was evacuated and baked at 300° C for three hours to drive out all moisture and organic material. The tube was then sealed while still under vacuum.



Figure 2: Wavelength dependence of reflectivity for various metals.

- 3. The sealed tube was HIPed at 15 ksi and 1100° C for 15 minutes. The temperature was linearly increased from room temperature at a rate of 50° C/min. and pressure was kept at 1 atm until a temperature of 900° C was reached.
- 4. After cooling, the Pyrex tube was broken and the metal part was grit-blasted clean.

3 Findings

3.1 Properties

A laser sintered specimen with a corresponding as-HIPed geometry is shown in Figure 3 along with the cross-sectional metallography of each part. This part was made from 44 μm (-324 mesh) iron powder in an environment of 10% hydrogen and argon using 7.5 W of laser power focused to a 0.5 mm spot. A scan speed of 5 in./min. was used with a scan spacing of 0.020 in. Each powder layer was approximately 0.004 in. thick. The as-sintered part is approximately 35% dense, making it a brittle structure; however, it was able to withstand the small amount of handling necessary to place it



(a)



Figure 3: Selective laser-sintered iron sample (a) before and after HIP; (b) and (c) microstructure.

in a HIP vessel. The as-HIPed structure is very dense (> 99.9%) as shown in Figure 3c, with only a small number of remaining voids.

3.2 Environment

The most significant problem associated with SLS of iron encountered in this research effort is the control of the sintering environment and the formation of oxide. For this reason, the powder handling system was placed inside a glovebox and an overpressure of inert or forming gas was applied inside the box. Additionally, the powder was "precleaned" by placing it in a fluidized bed charged with the same inert or forming gas.

3.2.1 Inert Environment

When using -325 mesh iron powder, it was found that a good single layer could be generated in an inert environment of argon. Unfortunately, the bond *between* the layers was generally *inadequate* for the following reasons:

- The free energy of formation for oxide increases with decreasing temperature [10]. Thus, in cooling to room temperature from a high homologous temperature, a period of high oxidation growth can be expected. This oxide is particularly undesirable in the SLS process because it prevents effective sintering or wetting.
- During the sintering of each layer, the temperature of the powder was brought to a value above the melting temperature of the metal. Each laser scan was performed relatively quickly. Thus, adjacent scans occurred before the material cooled and simultaneously oxidized. For this reason, the bonds within the layers (between scans) were acceptable.
- Between layers, the material was allowed to return to room temperature. Thus, each layer ran through a temperature cycle that is very conducive to oxide formation as discussed above. (Oxide was observed in SEM EDAX probing.) Though the atmosphere was free of oxide, enough residual oxygen was present in the powder to form an oxide layer on the upper surface and prevent good bonds between the layers.

3.2.2 Reducing Atmosphere

A common reducing atmosphere for ferrous materials is a mixture of hydrogen and nitrogen; this mixture is often called "forming gas." Laser sintering in this atmosphere proved to be unsatisfactory because of the formation of nitrides between each layer, preventing inter-layer bonds. (Nitriding is a common surface hardening technique for steels.)

When the same iron material was sintered in a reducing atmosphere of 10% hydrogen and argon, excellent bonds between the layers were achieved. It should be noted that all other process parameters (laser power density, scan spacing, layer thickness, etc.) were kept constant between the tests in argon, nitrogen and hydrogen, and those in argon and hydrogen.

3.3 Post-Treatments

From the materials standpoint, HIPing proved to be satisfactory for these preliminary tests-it achieved a dense sample with a predicted reduction in the dimensions of the specimen. From the standpoint of FFF, HIPing is not attractive without a model that gives a "process compensated" geometry for the sintering operation. A model of the HIPing process is required to define an "as-sintered" part geometry. This model is not straight-forward because, for example, holes and cavities which are temporarily filled with alumina powder will densify at a different rate during HIPing than the adjacent metal.

3.4 Warping

Because the heat is applied at the top surface, the top densifies to a greater extent than does the bottom surface of each layer. Additionally, the upper surface cools from a higher temperature than material below, causing more thermal contraction on the upper surface. for these reasons, each layer of sintered powder tends to warp upward, which is undesirable from a powder-handling standpoint because the thickness of each layer is not uniform. The solution adopted for this work was to build an "anchor" of thin sintered layers onto which the actual structure was constructed. This thin frame gives some stiffness to the overall structure and avoids warping in many situations.

Various other techniques are possible:

- Heating the powder bed. This method has been implemented [11] for laser sintering of metals with good success; it reduces the thermal strain by reducing the temperature excursion. This method has the added benefit that it helps prevent oxide build-up between layers.
- "Knitting" the structure in such a way that the loose powder is allowed to flow to fill the gaps left by the densified material.
- Bonding the prototype to a rigid sample of the same material during the first stages of the sintering process, effectively building an anchor. This may be acceptable for some applications where the anchor can become an integral part of the laser-sintered structure.

4 Conclusions

The proof-of-concept goal that stimulated this work effort was successfully achieved. Binderless iron powder was sintered into a cubic shape of 35% density in a reducing atmosphere using a directed laser beam. The cube was later HIPed to full density with a predicted reduction in overall dimensions. It was demonstrated that the control of the sintering environment is critical for successful laser sintering. In this particular case, an environment of 10% hydrogen and argon yielded the best inter-layer bonds.

5 Acknowledgments

The work and advice of several people is acknowledged: Leon Beha, Jerry Harrison, Jim Resue, Steve Rutkowski, Paul Siemers, Cosimo Montanaro, and Mark Brun.

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