

# **Mechanical Property Enhancement Via HIPing of Polymer-based SLS Objects**

Jose M. Perez, Jr.  
School of Engineering and Engineering Technology  
Penn State Erie, The Behrend College  
5091 Station Rd  
Erie, PA 16563, USA

Rapid Prototyping Center  
Milwaukee School of Engineering  
1025 N Broadway  
Milwaukee, WI 53202, USA

Faculty Advisor: Vito Gervasi

## **Abstract**

Solid Freeform Fabrication technologies have the ability to produce parts with complex geometries relatively quickly without the need for costly tooling. Advances in the field are creating a market in which these parts can go directly into a service environment, thus evolving into Digitally Driven Manufacturing (DDM). However, the limited material strength that DDM parts possess is keeping DDM from seeing its full potential. Although parts produced using DDM can be built with uniform densification, they are not fully densified. The application of a densification process used in metallurgy known as Hot Isostatic Pressing (HIPing) to polymer-based parts produced through the Selective Laser Sintering process is the focus of this research. The HIPing process places an object under heightened isotropically applied pressures through a pressure-conveying medium at an elevated temperature in order to more fully densify it via creep and plastic flow. An internally applied vacuum facilitates the removal of any gasses trapped inside the part and aids to further increase its density and ultimately improve its mechanical properties.

## **1. Introduction**

### **1.1. solid freeform fabrication**

Solid Freeform Fabrication (SFF) (often referred to as Rapid Prototyping (RP)) is the term used to describe the process of producing solid parts, models, and prototypes without the use of tooling. A computer generated solid-model is used as the basis for these parts and is broken down into two-dimensional horizontal slices which, when stacked, form the third dimension of height. These objects are then produced via an additive process that builds subsequent layers upon one another to form the actual part. Different machines produce these layers using a variety of techniques ranging from the controlled deposition of adhesives into a powder media to the use of a UV laser to cure a thin film of a photopolymer. The SFF process allows for the construction of parts with incredibly complex geometries both internally and externally that would be impossible to make in a single build using traditional manufacturing techniques. These parts can then be used as prototypes, mold plugs, or finished one-off parts.

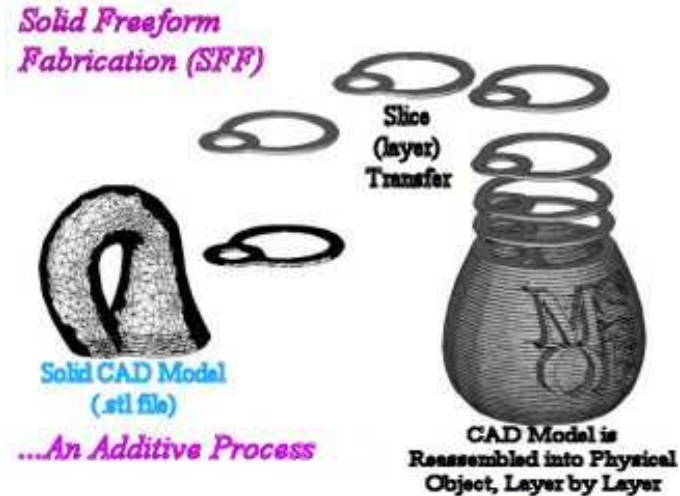


Figure 1 Solid Freeform Fabrication Example

## 1.2 selective laser sintering

Selective Laser Sintering (SLS) is one of these SFF processes and was the one used to produce parts for this research. The SLS process uses a CO<sub>2</sub> powered laser to precisely fuse thin layers of a powdered polymer together to form the individual slices. The temperature inside the machine is raised to a point where the laser can be run at a low wattage and only has to increase the temperature of the powder a small amount (between 10-15°C) to fuse it together. After each pass of the laser a roller mechanism (Figure 2) spreads the subsequent layer of the powder across the build bed. Then laser then fuses this next slice in the exact pattern of the solid model and the heat from the process also fuses it to the previous layer. The process repeats itself until the completed part is produced. The part is then removed from the build bed and any unused powder is removed from the part. These parts can then go directly into final usage but may require secondary processing to seal their otherwise porous surface.

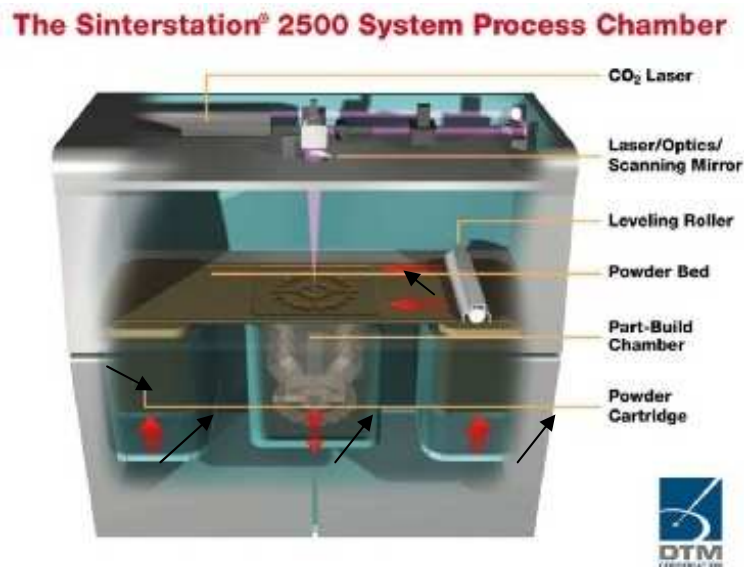


Figure 2 SLS Machine Components

### 1.3 hot isostatic pressing

The Hot Isostatic Pressing (HIPing) process is one that was developed for use in the Metallurgy industry to fully densify parts made using the powder manufacturing process and to remove the microporosity found on the surface of parts made using other manufacturing process. Parts that go through the HIPing process are exposed to elevated pressures via a pressurizing medium, either liquid or gas, at an increased temperature. The pressure works to collapse internal voids and more fully densify the parts by way of creep and plastic deformation of the material.

### 1.4 applications

Potential applications for HIPing as a secondary process in SFF are almost endless. As was stated earlier, the ability to build products that are not possible to manufacture using conventional methods and without the added cost and time of tooling are the two biggest advantages of SFF. With a significant increase in mechanical properties, SFF could make the following possible: one-of-a-kind optimized structures for the aerospace and medical industries would go from concept to feasible end-user products, artificial joints could be made to perfectly fit a patient *before* surgery, significantly reducing surgical time and chance of infection by eliminating the time currently spent modifying the implants during the surgery, and lastly, the ability to build replacement parts for any number of machines on-site without the down-time that comes from waiting on a vendor to supply parts or the cost and space spent stocking a large variety of replacement parts that may never be used would be a serious benefit to the manufacturing community at large. The other benefit to an effective method for mechanical property improvement would be lower operating costs on the SLS machine. If parts that are made at lower laser powers can be made stronger using a secondary process such as HIPing, then, providing the HIPing process is time and cost effective, there is an incentive to run the SLS machine at lower laser wattage which has been linked to lower degradation of the SLS powder and subsequently longer powder life. Essentially, this process could decouple the build parameters from the final mechanical properties.

### 1.5 research objective

The primary objective of this research was to study the possibility of improving the mechanical properties of polymer-based parts created on the SLS machine through the application of a HIPing process. The creation of a specific process was not the goal so much as proof of this process' potential through a substantial gain in mechanical properties (greater than 10%).

## 2 Properties of Polymer-based SLS Objects

When the polymer powders used in the SLS process is fresh, its individual grains are spherically shaped. Repeated exposure to the elevated temperatures inside the machine itself and in the powder bed when in proximity to previously built parts clumps individual particles together to form larger particles with irregular shapes. To account for this, all powder is sifted through a sieve between processes to get a more homogenous mixture of similarly sized particles within the powder bed before a build. Larger individual particles require more power to sinter and their irregular shape scatters the laser in a less predictable manner and reduces the layer thickness that can be used and still maintain the degree of accuracy required in these builds. Also, these larger irregularly shaped particles cannot fit exactly next to one another and will trap air between themselves and any adjacent particles. This trapped air remains even after the build is completed. This trapped air inside of objects produced using the SLS results in brittle parts with incomplete densification and poorer mechanical properties in the finished parts. It is this incomplete densification that is the primary focus of this research and the improvement of it to improve the mechanical properties of parts produced using the SLS process.

The specific material used for this research was DuraForm® PA Polyamide powder made by 3D Systems, Inc., specifically for use in SLS machines. 3D Systems, Inc. provided the following material data:

Density	0.59 g/cc
Avg. Particle Size	58 μm
Specific Gravity	0.97
Melting Point	184°C

Tensile Strength	44MPa
Tensile Modulus	1600MPa
Elongation at Break	9%

### 3 Method

#### 3.1 part build parameters

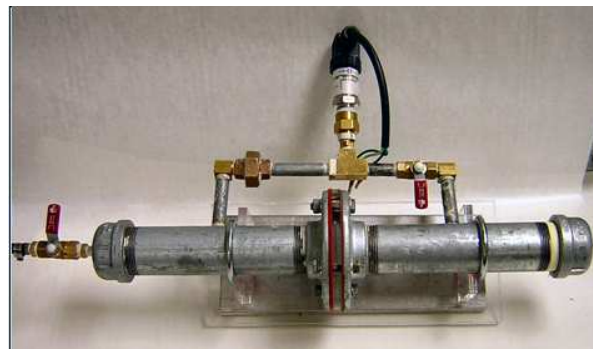
The parts used for this experiment were built on July 17, 2004 on a DTM, Inc. SinterStation 2500*plus* SLS apparatus. The part shape is a modified version of the ASTM D638 tensile bar. The primary difference was that the part has a thickness of ¼” and has a 0.190” diameter, 1” deep hole in one end. This hole was designed into the part to allow for a vacuum line to be inserted during the HIPing process. All parts were built using a laser power of 8 watts, one of the lowest settings available on the SinterStation 2500*plus*. A lower outline power was also chosen as well as a layer thickness of 0.004”. These settings were chosen because they produce parts that are less dense and have a higher amount of porosity. The mentality was that improvements would be more noticeable if going from a weaker part to a stronger part as opposed to trying to strengthen an already relatively strong part. If these results can be mapped then the process would only need to be extrapolated to work with parts that already have stronger mechanical properties.

#### 3.2 density measurements

To quantify the amount of densification a simple gas pycnometer (Figure 3) was constructed using two section of 1 ½” ID steel pipe, a series of fittings and valves, and an argon gas cylinder. Equations based off of the Ideal Gas Law (Equation 1) were used to derive the volume of the test samples. A high-accuracy pressure transducer was used to monitor the pressures (P) within the pycnometer and connected to a computer to record and graph the resulting data. The pycnometer is a dual chamber device with the part to be measured in one chamber and a predetermined amount of an inert gas in the other. The volume (V) of each of the two chambers was determined by measuring the amount of water each displaced. Using the ideal gas law equation (shown below) the change of pressure that occurs when the valve that separates the two chambers is opened can be used to derive the amount of volume displaced by the actual part. The Ideal Gas Law formula is shown here:

$$PV=nRT \tag{1}$$

The temperature (T) inside the pycnometer was monitored using a K-type thermocouple connected to a digital thermometer, the universal gas constant ( $R=82.0575 \text{ (mL*atm)/(K*moles)}$ ) was used, and the n-value was derived using basic algebra. Traditional methods of measuring density such as water displacement were not used in this study because the water absorbed by the test samples, due to the hygroscopic nature of the Nylon powder used in these parts, would have affected their mechanical properties.



**Figure 3 Gas Pycnometer**

### 3.3 pressurizing medium

Compressed air was chosen as the pressurizing medium for this test. Availability, convenience, and cost were the primary deciding factors in using it over any alternatives. Other products considered for use included a low melt bismuth alloy produced by the Cerro Metal Products Company and Argon gas. The use of bismuth was shelved because of problems with it adhering to and infiltration into the SLS parts even at lower pressures. The argon was not used for lack of a proper argon compressor. The compressed air was provided by a standard oil-less compressor and was piped into the pressurizing chamber using reinforced high-temperature silicone tubing. To prevent infiltration from the pressurizing medium and to distribute the pressure in a more isostatic manner each part was coated using a two-part high-temperature RTV silicone before HIPing. Each part also had a brass rod inserted approximately 1" into one end and had 1atm of vacuum applied to it through a manifold to evacuate the trapped gases inside the part.

### 3.4 pressure chamber

A stainless steel pressure chamber was built as the HIPing chamber for this project. The chamber is shown in Figure 4. Stainless steel was chosen for its corrosion resistance at the higher temperatures this vessel would operate in. The primary chamber has a volume of approximately 710mL with a depth of .248m. A pair of tubes, each with a 0.190" ID, extend approximately 16" from either side of the chamber's base. The first was used as the source line for the pressurizing medium and the second was used to feed a K-type thermocouple into the chamber to monitor temperatures internally. The lid was held in place by a set of 8 bolts and was sealed from the main chamber using a high-temperature silicone gasket. A single hole was drilled and tapped in the center of the lid to accommodate a 1/8" NPT fitting that allows the vacuum line to access the parts inside the chamber.

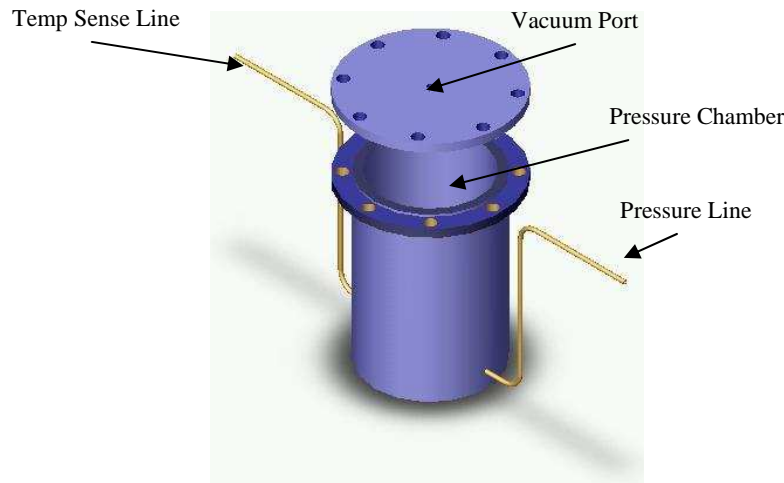


Figure 4 HIPing Chamber

### 3.5 mechanical testing

The mechanical properties of the parts used in this experiment were measured on the MTS Tensile testing apparatus along with a 2" extensometer. The resulting data was outputted to a Microsoft® Excel spreadsheet, which was then used to create the stress-strain curve and calculate the ultimate strength ( $S_U$ ), yield strength ( $S_Y$ ), tensile modulus, and Elongation at Break.

### 3.6 optical comparison

A small section (about  $\frac{3}{8}$ " long) at the end of each tensile bar was milled down approximately  $\frac{1}{16}$ " and a series of 5 holes was drilled into this thinner section using a straight pin as a drill bit (figure 5). The diameter of each of the 5 holes was measured before and after the parts underwent the HIPing process using a Scherr-Tumico Optical Comparator. These measurements were recorded, averaged, and compared to one another to determine a percentage of change as a result of the HIPing process. The holes were made in a thinner section to prevent them from being deformed during tensile testing.

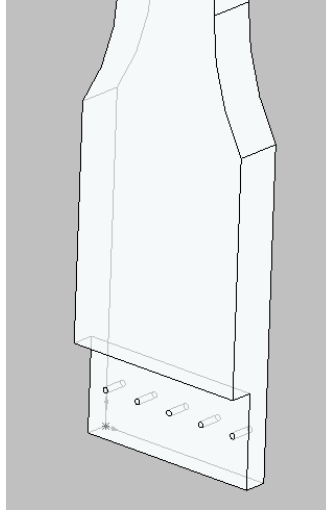


Figure 5 Example of Part Notch

## 4 Conclusion

### 4.1 ineffectiveness of low pressure HIPing

After conducting a series of HIPing processes on the test samples, examination of the physical and mechanical properties of the first batch of parts caused the entire series of tests to be aborted. Density and pinhole diameter changes were along the magnitude of .5-1.5%, well within the expected margin of error for the measurements themselves. These minimal changes in the part density directly correlated with negligible changes in mechanical properties and an essential re-evaluation of the process. Essentially, the use of HIPing pressures below 150psi has proven ineffective.

### 4.2 process improvements

The basic premise of improving the mechanical properties of a polymer-based part through the application of a secondary HIPing process remains valid. The specific method and equipment used in this research is what must be modified to prove the validity of this process. The first step would be to use a combination of higher pressures and temperatures to force the material closer to and possibly higher than its melt temperature. In essence, the part should become soft, without becoming molten, and therefore flow and densify better all while maintaining its shape. Additionally, the HIPing chamber should be modified to include an internal heating element. Temperature monitoring during the HIPing trials showed that the internal temperature of the HIPing chamber didn't reach the target temperature for almost four hours in the case of the higher temperature tests and for just over three hours in the lower temperature test.

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