

FABRICATION OF CERAMIC PARTS USING A DIGITAL LIGHT PROJECTION SYSTEM AND TAPE CASTING

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Abstract

In this paper, fabrication of ceramic parts using projection-based stereolithography by curing the mixture of ceramic and photopolymers was investigated. A stereolithography device with a UV LED projection light source was built. A series of resin and ceramic powders, including alumina and zirconia, were experimented to explore the viscosity of mixture and the resultant part quality. It was found that commercial photopolymers are not suitable for this purpose due to the small cure depth. A customized photopolymer without photoinhibitor and optimized photoinitiator has demonstrated success in producing 3D ceramic green bodies. It was found that the viscosity was very high for all of the mixtures with high ceramic loading which will influence the recoating process. Therefore, tape casting was used to apply the slurry during the build process.

1. Introduction

Ceramic manufacturing produces parts with desirable traits like high hardness, high temperature strength, and low conductivity. Conventional powder metallurgy approach to manufacture ceramic parts involves high tooling cost and long lead time, which is not desirable for small batch prototyping. Additive manufacturing (AM) provides a fast alternative method to fabricate ceramic parts by building layer by layer from a CAD file. Among AM methods, only a few are suitable for producing ceramics, including stereolithography (SL), filament extrusion [1, 2], powder bed fusion, ink writing, and binder jetting [3, 4]. SL process uses ultra-violet radiation from a light source to expose liquid photopolymers to cause cross linking. Under the category of SL, there are three subcategories that describe how the liquid monomer is cured: vector scan, two photon, and mask projection [5]. Digital micromirror device (DMD) based system, invented by SRI International, provides a convenient way to build 3D objects by projecting UV light onto a photopolymer vat using the sliced digital files manipulated by the DMD [6]. A comprehensive review of ceramic SL can be found in reference [7, 8]. Ceramic stereolithography with DMD has been investigated using LED as light source [9].

In ceramic SL, the viscosity of photopolymer is very important. Lower viscosity is beneficial for the slurry to flow and recoat; however higher viscosity is desired to ensure when the ceramic particles are added, the slurry is stable and there is no sedimentation occurred. The typical particle loading in the slurry is between 40% to 55% volume concentrations [9]. There are two types of suspensions: aqueous and resin. Aqueous suspensions are a mixture of acrylamide and N-N'-methylenebisacrylamide dissolved in deionized water [10]. Since it has a water base, the viscosity is low and flows easily, which makes recoating easy. Even though it has a favorable viscosity, it has many other undesirable traits. An example is that the strength after curing is low which makes it delicate. It also doesn't hold the ceramic particles in suspension well because of the low viscosity. The aqueous suspension needs to constantly be agitated to keep the particles suspended. Research from Griffith and Halloran have shown that aqueous acrylamide suspensions have very good fluidity but the depth of cure is not optimal due to the refraction from the powders

[9]. In contrast to the aqueous suspension, resin based suspensions create a much stronger green body after curing but also have a higher viscosity [10]. This type is the common commercial resin used in stereolithography. Brady and Halloran have researched that 200 mPa·s is the lowest viscosity that will ensure the ceramic particles will be stable against sedimentation [11]. Cure depth is another important factor for ceramic SL. The cure depth of the mixture of resin and ceramic powders is dependent on the species of the photopolymer, the ceramic loading, and the UV exposure dose. The ceramic particles change the way the light penetrates and can either scatter or absorb to change the refractive index. Modified Beer-Lambert law has been proposed to model the cure depth in ceramic SL [9]. way

This paper presents a DMD based ceramic SL system that exposes the slurry made of ceramic powders and photopolymers to build green body followed by debinding. A DMD projection system was built in-house for this study. Both commercial resin and customized resin were investigated. The viscosity of the slurry with were characterized.

2. EXPERIMENT

2.1 EQUIPMENT SETUP

The exposure system consists of a 5.5 W LED UV light source, a motherboard with DMD from Texas Instruments, and optics (Wintech Lightcrafter PRO4500, Wintech Inc. TX). The UV LED releases 405nm wavelength of light in a field of view of 65.6mm × 41mm. The DMD has a resolution of 912 × 1140 (WXGA) with diamond pixel. The focal distance is 92mm. The control board communicates with a computer through the Texas Instruments provided graphical user interface (GUI) that controls the image upload and exposure time with a HDMI interface. The STL file was sliced using Freesteel Slicer and each individual slice was uploaded to the firmware and stored on the external boards. Exposure time of each layer is controlled by a computer connected to the control board.

The platform system moves the build downwards in the z direction between each exposure. A XY-stage was used to control the movement. The resolution in the z direction was 10 microns. The platform is made of an L shaped sheet aluminum that the resin adheres to and was attached to the XY- stage. The platform was merged in a vat filled with photopolymer.

The recoating was achieved using an eccentric motor to provide vibration which would cause the resin to flow off to cover the finished layer. When the ceramic material was applied to the resin, it made the viscosity increase significantly. It was found that it is difficult for the slurry to flow over. Instead, a tape casting method with squeegee and a template were used for recoating. The final configuration is shown in Figure 1.



Figure 1 Final stereolithography 3D printer with all subcomponents mounted

2.2 EXPERIMENTS

2.2.1 MATERIALS

The exposure was done with the instrument developed in section 2.1. Two resins have been used in the experiments: a commercial photopolymer (AlphaSense Inc. DE), and a customized resin (Spectra Group Ltd., Millbury OH). Three powders were investigated: alumina with particle size of 3.0 μm (Hudson Supply, OH), zirconia with particle size of 40 nm (TZ-3Y-E, Tosoh Corp. Japan), and alumina with particle size of 500 nm (Ceralox APA-0.5, Sasol GmbH).

2.2.2 PROCESS

The powders were mixed with resin in a container first and then transferred to the vat which is underneath the projection lens. The viscosity of the slurry was measured using a viscometer (Brookfield DV-I). The sliced layer image was sent to the projector and exposed the slurry. After one layer was exposed, the platform lowered down and a blade was used to recoat another layer until all layers were completed. The green body was removed from the platform and then debinded in a furnace (SunFire 10, Neytech Inc).

A series of experiments have been conducted: (1) commercial resin only to test the functionality of the instrument; (2) commercial resin with 3.0 μm alumina and 40 nm zirconia; (3) customized resin with 500 nm alumina powders. The experimental details are described in section 2.2.3 to 2.3.5.

2.2.3 COMMERCIAL RESIN ONLY EXPERIMENT

The first experiment was done to validate the system functionality with resin only. A commercial transparent yellow photopolymer (AlphaSense Inc. DE) was used. The viscosity of the resin was measured using a Brookfield DV-I viscometer, with a cylindrical spindle. The viscosity was found to be 37.6 cP at room temperature. The exposure was 20 seconds and the recoating time was 40 seconds where the plate would step down and the resin would flow to recoat the plate. Simple rectangle and oval shapes were used as the test parts with a 0.1mm slice thickness. During the exposure, the liquid resin visibly cured when the image was projected.

2.2.4 COMMERCIAL RESIN WITH ALUMINA AND ZIRCONIA

The first test with alumina and the AlphaSense resin slurry has a particle loading of 50% by volume. The alumina powder has a particle size of 3.0 micron (Hudson Supply, OH). This mixture resulted in a very powdery end product that was not holding together which is not suitable for the exposure. The second batch has a 40% particle loading by volume. The viscosity of the slurry was measured to be around 100,000 cP.

For the zirconia mixture, a lower volume percentage of 26% particle loading by volume was used to ensure the right viscosity suitable for recoating. The Zirconia has a particle size of 40 nm (TZ-3Y-E, Tosoh Corp. Japan) which is expected to yield a deeper cure depth.

2.2.5 CUSTOMIZED RESIN WITH FINER POWDERS

The cure depth from the experiment in section 2.2.2 is not enough with current settings. To increase the cure depth the resin and ceramic material were changed. A custom resin was made for this purpose by Spectra Group Ltd. (Millbury OH). The resin is an acrylic monomer base with a viscosity of 85 cP at 25°C. It has no photo inhibitors added in order to maximize the cure depth. When this resin is exposed to UV light by itself, it can reach a cure depth of up to one inch depending on the light source power.

The alumina powder has a particle size of 500 nm (Ceralox APA-0.5, Sasol GmbH). The alumina has 500 ppm magnesia as dopant to help minimize agglomeration and maximize dispersion. The small particle size and magnesia were chosen to help keep the viscosity lower once it is added to the resin. Two batches of slurry were prepared: 35% and 40% particle loading.

2.3 DEBINDING

The green parts were sintered in a furnace (SunFire 10, Neytech Inc.). This has different ramp features and holds that are programmable in order to get the desired sintering process. The first phase for our test was the resin burnout. For this, the furnace was ramped up at 25°C/min to 732°C and held for 20 minutes.

3. RESULTS AND DISCUSSION

3.1 VISCOSITY OF SLURRY

The viscosity of the slurry is important and will influence the recoating and stability of the slurry. As discussed earlier, in order to have a successful SL process with vat recoating, the viscosity needs to be below 3000cP to flow properly and above 200cP to limit sedimentation. A rheometer (DHR-2, TA Instruments) was used to characterize the viscosity. All testing was done at 25°C. The flow sweep tests the viscosity at different shear rates taking the average of the measurements over 60 seconds for each data point. The range of shear rates varied between 0.01 sec⁻¹ and 10 sec⁻¹. This lower shear rate range was used because it is close to the conditions during SL. The results are show in Figure 2.

The pure customized resin (from Spectra Group) exhibits Newtonian fluid behavior because the viscosity remains relatively constant with a changing shear rate. It shows a viscosity around 100cP which is close to the manufacturer's specification of 85cP. All of the samples with 500 nm alumina powders (from Ceralox) however seem to show a shear thinning behavior which means that as the shear rate is increased, the viscosity decreases. This is probably due to the alumina being a suspension in the resin rather than a complete uniform dispersion. One important point in Figure 2 is where the viscosity passes the 3000cP measurement. This is between the 10%

and 20% particle loading which is much lower than the optimal loading. A loading closer to 50% is desirable in order to produce a green body that can be sintered to full densification. This means that for the Ceralox alumina and resin mixture, a vat recoating system will not work because the viscosity gets very high with the higher loading percentages. In order to resolve this, the recoating will use a tape casting method so the alumina particles can be loaded to a higher percentage. Another observation when mixing these different test samples was the amount of sedimentation at different levels. After mixing the compounds and letting them settle for a minute, there was a very thin layer of alumina at the bottom. Once the mixture got to about 30% particle loading there was no noticeable sedimentation. This is due to a flocculated powder bed that entered the compaction phase of powder sedimentation. When the particle loading went above 40%, the viscosity was too high to be measured. It was a very thick sticky peanut butter consistency and would hold its shape without flowing.

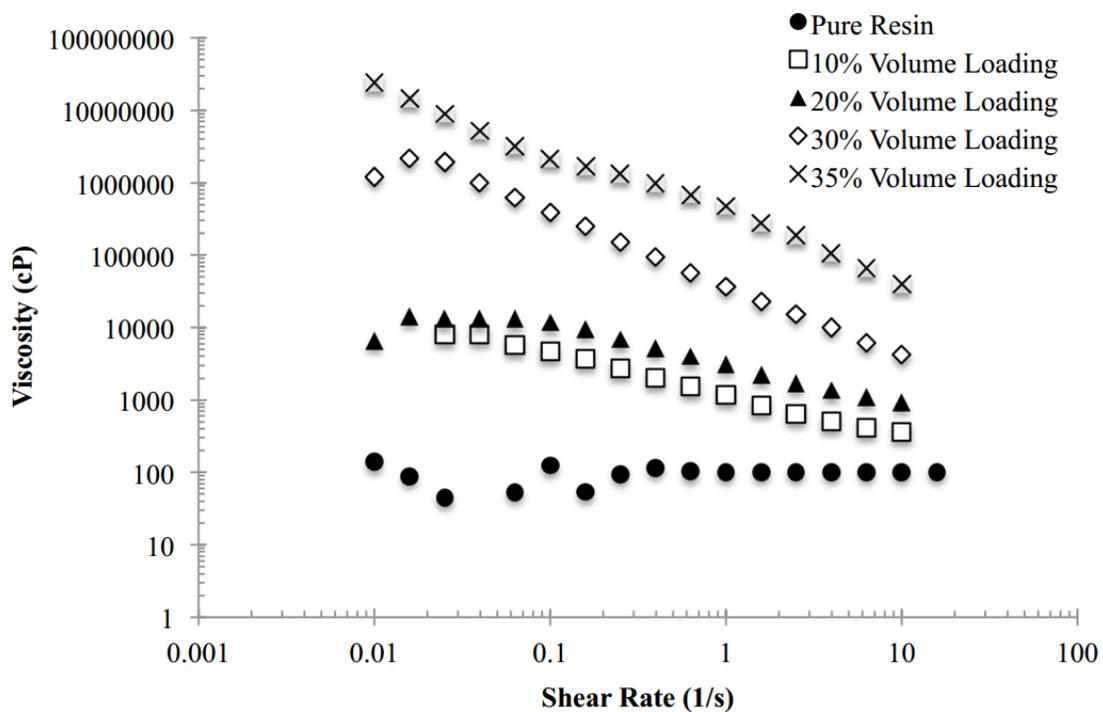


Figure 2 Viscosity test of customized resin with alumina in varying volume percentages

3.2 CURE DEPTH

The curing of pure resin (AlphaSense resin) has been cured by 20 seconds exposure. During the exposure, the liquid resin visibly cured when the image was projected. Even though the UV light was on for 20 seconds, the layer was cured in about 5 to 10 seconds. The extra exposure time does not affect the final part; it just slows down the print time.

However with the slurry made of 40% 3.0 micron alumina, after the first exposure of 40 seconds (trying to get the maximum cure depth), the result was a thin film of about 50 microns. This cure depth was not sufficient because it did not allow subsequent layers to bond to the previous one. This resulted in the parts being pushed around as each layer was applied which ultimately would not yield a viable part. When exposure time was increased, the cure depth did

not increase. This was because the reflection of particles reduced the UV light absorption. The grey color of the alumina powder itself also partially contributes to the less absorption.

The cure depth of this zirconia mixture was much higher than the alumina at about 0.25mm and each subsequent layer was successfully bonding with the previous. From exposure point of view this is desired but from sintering perspective, the particle loading was too low at 26%, which would not be sufficient to provide a fully ceramic part after sintering.

In order to successfully obtain a green body with reasonable relative density and also ensure the cure depth is enough, the resin must be optimized. The commercial resin usually has photoinhibitor, which is to prevent excessive curing depth in conventional SL. For the purpose of curing mixture of particle and resin, the concentration of photoinhibitor must be reduced. In the customized resin, the photoinhibitor was completely removed to maximize the cure depth. At the same time, the photoinitiator must also be optimized to increase photo polymerization. The percentage of photoinitiator (H-NU 400 IL, Spectra Group, OH) was optimized for the 405 nm wavelength. Figure 3 shows the absorption with different concentration. The absorption of the resin used in this study was about 7%. Smaller particle size also helps to increase cure depth; therefore, alumina powders with finer particle size (500 nm, Ceralox) was chosen to make slurry with this customized resin.

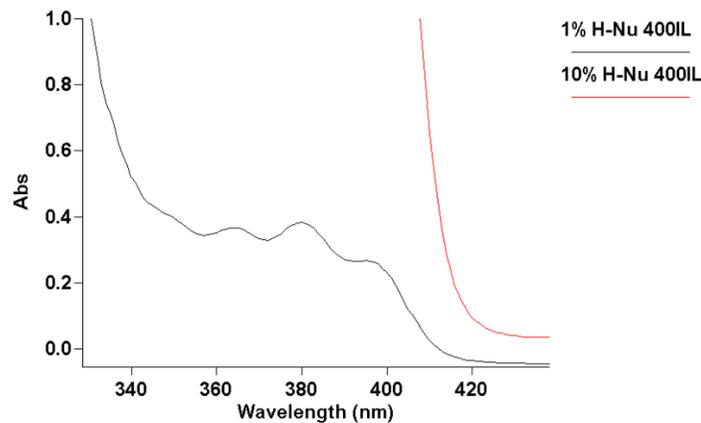


Figure 3 Absorption of resin with different concentration of photoinitiator [courtesy of Spectra Group]

In order to test the cure depth of this mixture, a particle loading of 35% was printed. The step depth between each exposure was 0.2mm and a tape casting method was used to apply the slurry. Rather than dipping it into the bath, the resin mixture was squeegeed onto the plate. Each exposed layer adhered to the previous layer so the cure depth was sufficient with this resin. The resultant green body has low relative density that handling is difficult. Therefore another batch with particle loading increased to 40% was prepared.

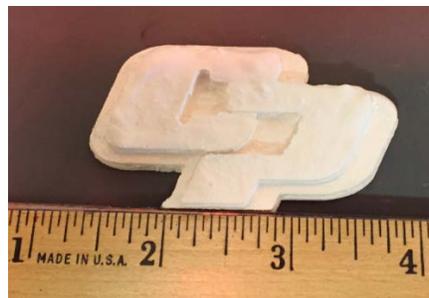


Figure 4 40% particle loading of alumina with customized resin

Figure 4 shows the final green part with 40% concentration of Ceralox alumina. Each layer has very high accuracy with regards to the features. The small disconnect between the “C” and “P” was kept separate throughout the build. The one discrepancy visible is the change between each height. As the platform lowers, the layer height gets thicker in some places and sometimes didn't settled directly on the previous layer. The thicker portions are due to the tape casting and not getting a perfectly smooth layer application. The disconnection between layers is due to the platform moving between layers while building. In order to resolve this, another bolt can be added to prevent any rotation on the printing plate. Another experiment was conducted with this improvement. Three different parts with different sizes were successfully exposed to examine the feature's fidelity with part size. The results is shown in Figure 5. Each subsequent layer had very good placement on the previous which confirms that the print plate no longer has rotational movement to take away from the accuracy. The parts are a quarter inch tall and the detail was held through each layer. The smallest mustangs produced fine details. The gap between the front hoofs is 0.01 inch and it was held separate in all the builds which shows very good layer accuracy.



Figure 5 Exposure with different feature size

Figure 6 shows the part during resin burnout at approximately 400°C. The black is the resin carbonizing and burning out of the part. After it reached 732°C, the carbonizing was over and the part went back to white showing that the resin was gone.



Figure 6 Resin burnout initial stages showing carbonization

4. CONCLUSIONS

1. Higher viscosity is desired to prevent particle sedimentation during ceramic SL, but make it difficult to recoat. Tape casting method demonstrated success with good layer uniformity. 40% particle loading by volume has been achieved in this study which yields higher green body density for post sintering.
2. Commercial photopolymers are not suitable for ceramic SL due to the cure depth is too low. The resultant green body doesn't have enough adhesion between layers.
3. A customized photopolymer without photoinhibitor and optimized photoinitiator, has been demonstrated successfully producing alumina ceramic green body with very good resolution.

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