Technical Development of Slurry Three-Dimensional Printer

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Abstract. The aim of this paper is to review the technical development of slurry three-dimensional printer (3DP) which based on photo-polymerization and constrained surface method. Basically, slurry consists of ceramic powder, resin and photo-initiator. The light engines for solidifying the photo-curable slurry can be classified as laser, liquid crystal panel (LCD), digital light processing (DLP). The slurry can be reacted and solidified by selective ray according to the reaction spectrum of photo-initiator. Ceramic powder used in this study is zirconia oxide. Experimental results show that ceramic particle size affects the viscosity of slurry severely resulting in low accuracy and the occurrence of micro crack in the layer casting procedure. Therefore, the effect of particle size on the curability and accuracy of built green part is discussed. A single dental crown is proposed to be fabricated by these three light engines as a benchmark for comparison. In addition, the cost and the limitation are compared in the aspect of dental crown fabrication. Consequently, the lowest cost is LCD-type slurry 3DP system. DLP-type slurry 3DP can produce green body with the fastest fabrication time. The volumetric error of sintered part that made by these three fabrication methods is similar because the composition of slurry is the same.

1. Introduction

The three dimensional printer (3DP) can produce parts with complex shapes using various materials. The vat photo-polymerization, one of 3DP, uses light to induce polymerization of a liquid monomer into a solid polymer. It is also known as stereolithography and divided into the top-down and the bottom-up methods as shown in Figure 1 [1]. Many 3DP developed different methods to produce 3D ceramic parts over several years. The top-down stereolithography (SL) [2], selective laser sintering (SLS) [3], fused deposition modelling (FDM) [4] and powder ink-jet printing (IJP) [5] are the typically system to fabricate the production of ceramic particles without moulds or tooling. The top-down SL can produce high accuracy green part among the others but the viscosity of slurry needs to be range between 2 and 5 Pa.s to ensure satisfactory layer recoating [6]. Furthermore, the major disadvantages of top-down SL are the high volume shrinkage ratio and unavoidable waste of slurry.

The advantages of bottom-up SL are resin savings, cost reduction and better controllable layer thickness [7]. The development sequence of light engine for SL is laser for point scan [8], optical fiber array for line scan [9], liquid crystal panel [10] and digital light projection (DLP) [11] to generate the
layer pattern for layer exposure. However, the bottom-up SL based on DLP method has not been used to produce ceramic parts yet.

The aim of this paper is to compare the cost and accuracy of a single dental crown fabrication. The method used is the bottom-up SL with vary light engines consisting of laser scan, layer pattern exposure using LCD and DLP.

![Figure 1. Two fabrication method in the stereolithography system (Left: Top-down SL system [7]; Right: Bottom-up SL system).](image)

2. Material and methods

2.1. Slurry preparation

This study used a micro-sized zirconia-yttrium (called ZrO₂) powder with an average particle size of 1 µm. The composition is list in Table 1. The slurry consisted of four components: 70 wt% ZrO₂, 25 wt% HDDA (1,6-hexandediol diacrylate), 8.5 wt% acrylated monomer and 1.5 wt% photo-initiator (Trimethylbenzoyl diphenylphosphine oxide, TPO). The mixture slurry is then ball milled at 200 rpm for 2 hours. Finally, it is degassed in a vacuum chamber for 10 minutes to remove the air bubble that may be generated during the ball milling process. A viscometer (DV-III, ULTRA) is used to measure the viscosity because the particle size affects the viscosity of slurry and the fluidity during casting a layer. As a result, the viscosity is 3.89 Pa.s.

|        | ZrO₂ | Y₂O₃ | Al₂O₃ | SiO₂ | Fe₂O₃ | Na₂O | TiO₂ |
|--------|------|------|-------|------|-------|------|------|
|        | >94  | 5.2±0.2 | 0.25±0.05 | <0.02 | <0.002 | <0.005 | <0.002 |

2.2. Fabrication method

The fabrication method of this study bases on bottom-up SL system with three different light engines which are laser scanning, pattern exposure by using liquid crystal panel (LCD) and digital light processing (DLP) as shown in Figure 2. Layer pattern is scanned by laser or exposed by image projection by LCD or DLP through a transparent resin vat to cure layers that are hung upside-down on the building platform. Besides, the bottom of the resin vat is coated with a thin PDMS (polymethylsiloxane) film to facilitate the separation of newly cured layers.

2.3. Specifications of the used system

Table 2 lists the specification of the three types of 3DP. The laser type of 3DP is Form 2 that is commercialized by Formlab Co. The resolution bases on the spot size of laser beam. The resolution of LCD depends on the pixel size as shown in Figure 3. No literature reports how to calculate the exact resolution in 3DP application using DLP. Therefore, this study proposed a general calculation method bases on the effective forming area. The DLP projector used is 720P that has a 1280 × 800 resolution. In this study, the effective forming area has a 144 wide along the X-axis. Therefore, the width (144
mm) is divided by 1280 and the exact resolution in X-axis is 0.1125. Then, the exact resolution in X-axis can be obtained by 0.1125 multiply the height resolution (800) as 0.081. As a result, the exact resolution for DLP in forming process is 113 × 800 µm. The price also lists in Table 2. The lowest price is LCD-type 3DP.

Table 2. The specification comparison of the three types system.

| Resolution (µm) | Average price (USD) |
|----------------|---------------------|
| Formlab Form 2 (Laser scan) | Laser spot size: 140 Z-axis: 20-100 |
| LCD pattern (layer exposure) | Pixel dot: 117 × 93.6 Z-axis: 20-100 |
| DLP pattern (layer exposure) | Exact resolution: 113 × 90 Z-axis: 20-100 |
|                |                     | 3499          |
|                |                     | 1599          |
|                |                     | 3488          |

2.4. Dental model benchmark

Single crown is an obvious aesthetic device in dental restoration device. Conventional fabrication method use CAD/CAM technique but it frequently induces the visible unavoidable surface micro-crack resulting in lower mechanical strength after sintering treatment. Therefore, this study proposes a general single crown model as a benchmark as shown in Figure 3. The dimension of this model is 10.4 × 11.5 × 6.4 mm³. Hence, three types of 3DP will fabricate the green part of the proposed model.

Figure 2. Fabrication method (a) laser scanning, (b) LCD light engine and (c) DLP light engine.

Figure 3. The proposed benchmark.

2.5. Sintering treatment
All green parts are carried out sintering treatment for obtaining zirconia dental crown with high strength and dense. Literature represents that two-stage sintering process can avoid the occurrence of micro-crack [12]. Therefore, the green parts are heated to 600 °C and maintained for 1 hour as the first stage for burnout the resin. The heating at this stage is 5 °C/min. Then, the temperature rises to 1450 °C with a heating rate of 10°C/min as the second stage. The holding time is 4 hours to allow the grain growth.

2.6. Microstructure observation and hardness measurement

The raw materials of ZrO₂ powder with fine particle size and high tetragonality could be sintered to dense at the temperature above 1400°C but crystallinity and microstructure are function of particle size and sintering parameters. Therefore, a field emission microscope (FE-SEM) is used to observe the particle size of green part and microstructure of sintered part. Furthermore, the hardness of sintered parts is measured using a Vicker test to compare with standard specimen for evaluating the hardness requirement of the 510K regulations.

2.7. Volumetric measurement

The volumetric control software is used to automatically measure the volume of each sintered benchmark. The ratio of each Form 2, LCD light engine and DLP light engine replica benchmark volumes are compared to that of the proposed benchmark CAD model.

3. Results and discussion

3.1. Green body fabrication

Figure 4(a) shows the result of green body fabrication. The layer can be seen and the thickness of each layer is 50 µm. Fabrication times for the LCD and DLP light engines are 15 minutes but for laser scan is about 28 minutes. That means laser scan needs more times to fabricate a single benchmark and depends on the numbers of fabrication piece and layer. However, fabrication time for layer exposure only depends on the number of layers. This reveals that layer exposure has higher efficacy than laser scanning. The green body is not easy to measure the dimension because the strength is low. Therefore, the volumetric measurement will be carried out by sintered part.

3.2. Sintering treatment

Figure 4 (b) represents the result of sintered part. The layer cannot be seen because the resin is burnout and shrank. Experimental results show that the volumetric shrinkage rate is about 51%. Therefore, CAD model was enlarged according to the volumetric shrinkage rate resulting in all green bodies were larger than the require benchmark. After sintering treatment, the sintered parts were volumetric measurement.
3.3. Volumetric measurement

Figure 5 shows the result of volumetric measurement. The maximum volumetric error is about 0.53 mm that occurs at the marginal line. However, the error should be less than the measured magnitude because the sintered part was painted with black due to the appearance color is white and high reflector index. The paint layer is about 0.12 mm thickness. Therefore, the exactly volumetric error is about 0.41 mm.

Because the composition of slurry is the same for the used three types of forming method, the result of volumetric error is similar. However, the fastest fabrication time for green body is the DLP type because the light intensity (58 mW/cm\(^2\)) from projection is higher than that of the LCD-type (14 mW/cm\(^2\)). As a result, the curing time for one layer of DLP-type is 2 seconds but that for LCD-type is 8 seconds.

![Figure 5. The volumetric tolerance of sintered part.](image)

4. Conclusion

This study fabricates green bodies of single crown by three different types of additive manufacturing including laser scanning, LCD exposure and DLP exposure successfully. Before sintering treatment, zirconia particles are adhered by cured resin to form the green body. The volume of green body is pre-enlarged according to the volumetric shrinkage rate of sintering treatment. These three types can fabricate the green body of benchmark successfully. After sintering, the maximal volumetric error is 0.41 mm. These three methods have the similar volumetric error because the composition of slurry is the same. Comparing the equipment cost, LCD-type slurry 3DP system is the lowest. However, DLP-type slurry 3DP system can produce higher accuracy of sintered part.

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