

# High Quality, Fully Dense Ceramic Components Manufactured Using Fused Deposition of Ceramics (FDC)

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## Abstract

Solid Freeform Fabrication (SFF) is a technology that produces physical solid components or parts from computer design models. This technology has the potential of reducing functional ceramic product development cycle time in terms of reducing design iteration and production time, minimizing extra post processing, and therefore reducing cost. A commercially available Fused Deposition Modeling (FDM<sup>TM</sup>) 3D Modeler was altered for use with ceramics. This newly developed method referred to as Fused Deposition of Ceramics (FDC) is capable of fabricating complex shape, functional ceramic components.

We have investigated issues related to hardware, software, feed material, and build strategy which are required to achieve high quality, fully dense green ceramic parts. In this paper, we report recent improvements made in the FDC process, including hardware modifications, software improvements, feed material standardization, as well as build strategy/condition control. We also report the current FDC status for making complex functional parts. Our goal is to optimize the FDC condition to ensure its robustness for producing defect free green ceramic parts consistently and without interruption.

## 1. Introduction:

Solid Freeform Fabrication (SFF) is a technology that produces real physical solid models from 3D computer-aided design (CAD) model data, electronic scan data and model data created from 3D digitizing systems for rapid prototyping applications such as testing of form/fit/function. Because SFF is able to quickly go from 3D electronic data to an arbitrarily complex shaped 3D part, it allows design engineers to make design corrections very early in the product development process, and reduces the number of design cycle iterations and cost dramatically. The solid freeform fabrication technologies also have an advantage for small quantity production. This is especially true for structural ceramic components, because the cost of manufacture of a structural ceramic component is a direct function of part complexity and production quantity.

Fused Deposition of Ceramics (FDC), developed at Rutgers University, is an SFF technique [1-6], based on the commercial Fused Deposition Modeling (FDM) process for fabrication of ceramic and metal (FDMet) objects. FDC uses a polymeric binder system to bond ceramic powders together in forming wire-like filaments which are melted in the liquefier delivery head which moves in the X and Y directions and is controlled by computer. The material is then

extruded from the heated head and deposited on a layer-by-layer basis. The extrusion process shears the material which quickly solidifies while reheating and bonding to the previous layers.

For plastic and wax parts manufactured by the FDM process, the primary concern is defects on the part's surface. Defects such as internal voids have not been a top priority for FDM, because plastic and wax parts do not necessarily require full density for their applications. For ceramic parts made by the FDC process, internal voids due to build defects are crucial because they will act as critical flaws that lower the fracture strength (i.e. functionality) of the ceramic component.

Since our objective is to directly produce high quality functional ceramic parts, achieving full density in the FDC parts is the top priority in the building process. The production of fully dense ceramic parts using FDC requires a high quality and continuous feedstock filament of FDC'able material and a fully automated FDC process. Functional ceramic parts also require good FDC process control of deposition tool path, build parameters and build environment.

## **2. Build Strategy and Condition:**

Green ceramic parts can be built by FDC in different orientations. The decision of which build orientation to use depends on the part's geometry and application. Parts can be made horizontally or vertically, with support or without support. Parts also can be made with different selections of perimeter/contour/raster combinations, and with different angles of raster fill. Build conditions, such as liquefier and envelope temperatures are also important and need to be considered before part production.

Producing high strength ceramic parts is a high priority in our FDC research. The strength of a green FDC part will be affected by the build orientation and the build conditions used. Control of the part's environment during the build process is also critical for ensuring a high quality part. If the build envelope temperature is too low or constant environment temperature is not maintained, previously deposited material will cool too much and imperfect adhesion to the next layer will result. Incomplete inter-road bonding also occurs when tool path vector lengths are too long.

Normally, a part with the largest cross section built in horizontal directions (X,Y) requires less build time compared with building the largest cross section in the vertical direction (Z). This is because of the Z-stage wait time of the 3D Modeler. When one layer is finished, the Z platform has to move down to open the space for brushing the nozzle tip, then move upward to the build position. This action normally takes about 4 seconds. For example the tensile bar shown in Figure 1 required 0.6 hour when built horizontally and 1.3 hour when built vertically with the same head speed.

Three RU955 GS-44 silicon nitride tensile bars were made, all with 45, -45 degree raster fill shown in Figure 1. Two of them were made horizontally under different envelope temperatures, one with 45°C and one with 30°C. One of the bars was made vertically with a 30°C envelope temperature.

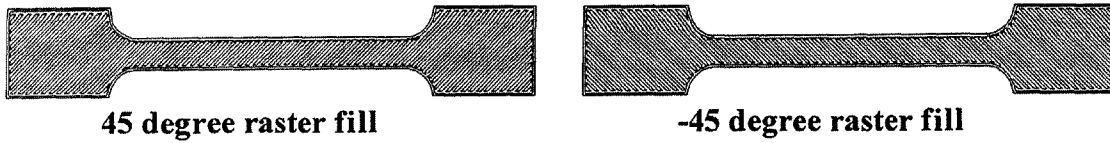


Figure 1: Green GS-44 silicon nitride tensile test sample with 45, -45 degrees raster fills.

According to the strength test results, Figure 2, the bar made in the horizontal direction with a 45°C envelope temperature had a higher strength (3.18 MPa) than the strength (2.84 MPa) of the identical bar made with a 30°C envelope temperature. However, the bar made in the vertical direction with a 30°C envelope temperature had the lowest strength (1.94 MPa) of the three. The test data and stress vs. strain curves are shown in Table I and Figure 2. The results indicate that green FDC parts have a lower strength in the Z direction than in X-Y directions because of the nature of layer bonding in Z direction. This result suggests that in order to obtain a high strength green part, the largest or critical section of the part should be built in the X-Y plane.

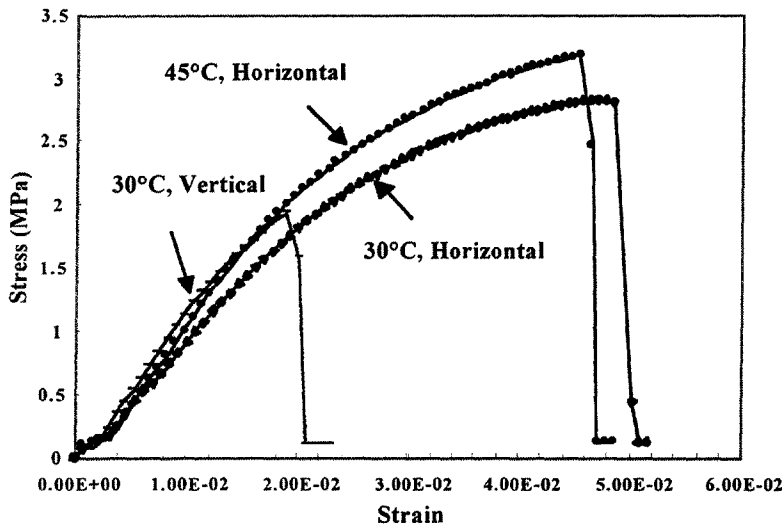


Figure 2: Tensile stress vs. strain curves of the green RU955 silicon nitride tensile bars made by FDC under different conditions.

The Fused Deposition build envelope temperature must be maintained at a sufficiently high temperature to provide for better material bonding [7]. The envelope temperature has an effect on material bonding between layers, and residual stresses in the green part. The temperature should be sufficiently high to ensure good bonding between layers. If the layers are cooled too rapidly (due to the surrounding temperature), then residual stress may develop and lead to cracking during BBO (Binder Burn Out). In addition, if the temperature is too high, the part may deform under its own weight.

Table I: Tensile Test Results for Green RU955 Silicon Nitride Bars.

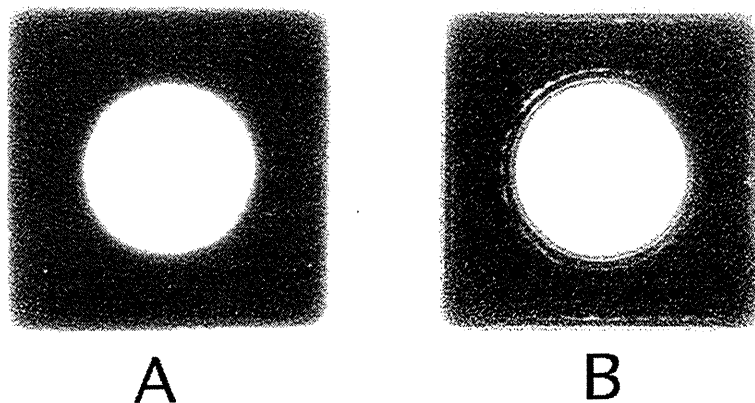
EnvTemp	Raster Angle	Road Width	STRENGTH	
			Horizontal	Vertical
30°C	45, -45	0.015"	2.84 MPa	1.94 MPa
45°C	45, -45	0.015"	3.18 MPa	

### 3. Sub-Perimeter Void and Software:

The sub-perimeter void is a common defect for both FDM and FDC processes. The sub-perimeter void occurs in the conjunction areas of perimeter and rasters. Because of the linear approximation of tool path, sharp turns for rasters occur. As a result, empty spaces (voids) are left without being filled due to the curvature of the perimeter and sharp corners (angles) of rasters. These sub-perimeter voids in FDC parts are too large to be overcome in the sintering process, hence the strength of FDC ceramic parts is affected significantly.

Recently, we have been using a simple method to eliminate sub-perimeter voids in the FDC process. This method might not be the best solution for solving this problem, but it is an easy and effective way to get ride of the sub-perimeter voids.

Normally, when creating the tool path on each slice, the build parameters need to be specified in the software QuickSlice. The air gap between roads can be set with negative, zero, or positive offsets depending on the application. For example, if the offset is set to a positive offset, then there is a real gap between fill vectors. If the air gap is equal to zero, then there is no air gap, which in principle means perfect filling and that the roads are perfectly in contact with each other. If the air gap has a negative offset, there will be overlapped fill. In the past, we have used small negative offsetting of the air gap to ensure internally fully dense parts. In fact, the function of the offsetting technique can be used effectively for the elimination of sub-perimeter voids. This simple method works well in most cases especially for our RU955 silicon nitride parts.



*Figure 3 : X-ray radiographs of green GS-44 silicon nitride part made with a negative offsetting (A), and without negative offsetting (B).*

Figure 3A shows an X-ray radiograph of a FDC green GS-44 silicon nitride part containing no sub-perimeter voids. This was accomplished by specifying an air gap with a negative offsetting in the build parameters. The negative offsetting allowed the raster fill to overlap with the contour fill, eliminating the possibility of voids. However, when

an air gap with no offsetting was specified, sub-perimeter voids were created. Figure 3B shows an X-ray radiograph of a part, built without a negative offset, containing voids.

Overflow (too much material deposited on the part surface) created by using the negative offsetting is the disadvantage of this method. It sometimes causes dimensional changes in parts.

However, by carefully selecting the value of negative offsetting, the overflow problem can be minimized. We have been using negative offsetting from  $-1/4$  to  $-1/3$  of the raster width in most cases. The dimensional expansion of the part sometimes can be as large as  $0.008''$ , depending on the build condition and part geometry.

This negative offset technique works well for rasters with small road widths. This is because of the void created at the sharp turning corner for small road width rasters is smaller than the one created by large road width rasters. For parts which have an interior small boundary, e.g., a small hole, it is difficult to eliminate the sub-perimeter voids especially when the size of a small boundary or hole is not much greater than the road width of the raster fill.

#### **4. Feed Material Standardization:**

In the past, we had frequently experienced clogging of the nozzle in the FDC process especially for small size nozzles ( $0.020''$ D or less). The causes of nozzle clogging were foreign objects and agglomerates of ceramic particles. Recently, the FDC filament fabrication laboratory at Rutgers University has gone through a series of steps for improving the quality of the FDC filament [8, 9]. The main focus was to eliminate any foreign contamination, and to produce better mixing/deagglomeration and more uniform filament. A fine screen is used in filament extrusion to remove large particles or foreign objects. The Standard Operating Procedure (SOP) of filament fabrication has been implemented to ensure reproducibility and to minimize the chance of contamination and process variation.

Filament buckling is another critical problem in the FDC process. Buckling is caused by poor quality of filament and insufficient filament aging. Poor quality filament refers to filaments which either are not stiff enough, or have high viscosity, or is not uniform in diameter. Most buckling due to poor filament quality has been eliminated via improved ceramic processing. In most cases, we believe that the filament buckling is due to a material aging problem. In the recent past, we experienced some difficulties due to the inconsistency of FDC feedstock material. A large part of the problem was due to the effect of filament storage and due to the lack of understanding of the RU955 binder's aging. In order to produce the same condition of feed material which is feasible to FDC on a daily base, we performed a series of aging tests for the RU955 binder [8, 9].

According to the test results, moisture does affect the quality of RU955 filament. Too high a moisture content of the FDC filament results in a high drive motor current and bubbles on the extruded surface. However, the aging studies suggest that the moisture effect can be eliminated by a vacuum heat treatment as part of filament aging. A fixed and standardized filament aging process and storage procedure has been employed to ensure consistent FDC conditions [8, 9].

#### **5. Hardware Improvements:**

Since the filament used in the FDC process also acts as the piston for extrusion, the filament should have enough stiffness when entering the liquefier. It has been shown that the  $G'$  (storage modulus of the filament) is a strong function of temperature between  $0^{\circ}\text{C}$  and  $30^{\circ}\text{C}$ . Therefore, cooling the filament near the top of the liquefier becomes critical. If the heat brought up from the

liquefier is not removed, then the heat will soften (lower the  $G'$  - stiffness) the filament and in turn cause buckling. Because the cool air (at R.T.) from the FDM cooling system at the top of the head is partially blocked by the wheels and motor block, the FDM cooling system is insufficient to maintain enough stiffness in the FDC filament. The cooling path shown in Figure 4 for FDC has been altered from the top to the right side to provide cooling directly in the region where the filament can buckle. In the new cooling system, the cooling path and the filament feeding path have been separated above the work platform. A temperature controlled cooler is attached to the air blower on the back of the machine, so that cool air ( $\sim 15^{\circ}\text{C}$ - $18^{\circ}\text{C}$ ) is blown by the cooling fan into the liquefier head. The air then goes back to the cooler's thermostat chamber to make a closed-loop so that the temperature can be controlled. This modification has dramatically reduced the filament buckling problem, allowing better process control, and maintenance of more uniform FDC conditions.

Temperatures under three different conditions were measured for one FDC build condition (liquefier temperature= $186^{\circ}\text{C}$ ; envelope temperature= $44^{\circ}\text{C}$ ; envelope humidity= $25\%$  RH), they are with A/C ( $\sim 18^{\circ}\text{C}$ ) cooling, without A/C but with room air ( $\sim 25^{\circ}\text{C}$ ) cooling, and no cooling. Temperatures for each case were measured at two locations, at the location between filament rollers and at top of the liquefier (see Figure 4). The measured temperatures are listed in Table II.

Table II: Temperature Measurements With and Without Cooling.

	No Cooling	$\sim 25^{\circ}\text{C}$ RT Cooling	$\sim 18^{\circ}\text{C}$ A/C
Temp. Between Rollers ( $^{\circ}\text{C}$ )	43-45	28-29	20-21
Temp. at Top of Liquefier ( $^{\circ}\text{C}$ )	61-64	32-35	24-25

The thermostat on the A/C unit was set at  $18^{\circ}\text{C}$ , and it continuously pumped  $18^{\circ}\text{C}$  cool air into the liquefier head. The cool air then leaves the head from the electric wire conduit to the back of the 3D Modeler and back to the air conditioner. Since it is a closed-loop and the cooling system does not affect the build envelope environment, the envelope temperature can still be well controlled. The FDC build envelope temperature recently has been raised to  $45^{\circ}\text{C}$  from previous values used of  $32^{\circ}\text{C}$ - $38^{\circ}\text{C}$ . We believe that the relatively high FDC envelope temperature results in better material bonding in the FDC build process for RU955 binder, and therefore improved the green part quality.

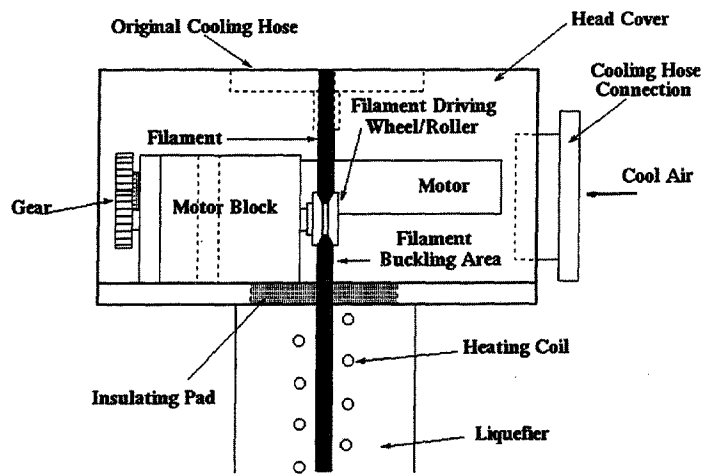


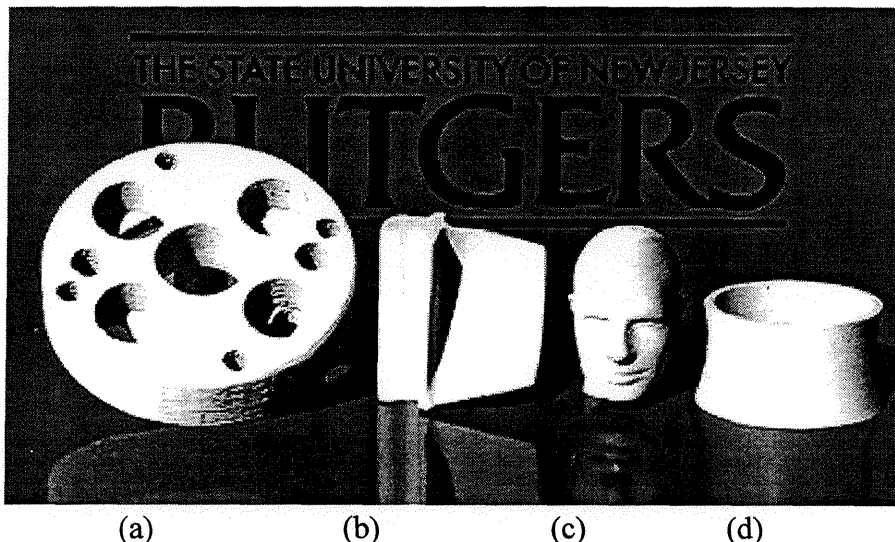
Figure 4: Schematic of FDC liquefier head with new cooling path.

## 6. FDC Issues and Components:

The selection of road width when creating build files is very important, especially for complex shaped parts. For example, the port plate shown in Figure 5(a) has several cavities. Improper selection of road width will cause internal fill air gaps most likely appearing in two regions. One is the area where the raster fill is parallel or near parallel to a curved boundary (sometimes, called high angles). The second is the area where two boundaries are separated from each other by a small distance. Improper selection of road width will cause voids in these two regions.

As an example, because of the complex geometry of the port plate shown in Figure 5(a), it is necessary to use more than one “set” of build parameters for creating internal roads. The advantage of using multiple sets for creating internal roads is that each set will match well with assigned slices which represent a particular geometry. The multiple assignments of sets will overcome the difficulty for handling a complex part by a single set, and will eliminate internal defects. However, complex objects normally require several iterations of internal road design. It is therefore important to investigate all different slices of internal roads created by QuickSlice before saving it into a build file or download it to the 3D Modeler.

The port plate shown in Figure 5(a) designed by AlliedSignal has a complicated geometry with different size holes and under cuts which is very difficult and expensive to make by normal manufacturing processes. A total of 3 port plates have been made by FDC recently. The turbine blade for an auxiliary power unit (APU) shown in Figure 5(b) is also a complex part because of the twisting curvature of the blade. Recently, we made several 1.5” tall turbine blades using the FDC process. The overall dimension, FDC build speed and time, and length of filament used for each port plate and turbine blade are listed in Table III.



*Figure 5: Demonstration of recent green FDC parts made out of silicon nitride.*

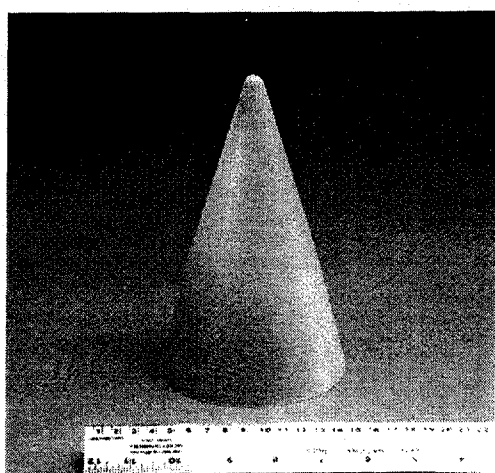
The human head model and the curved cylindrical shell shown in Figure 5(c) and 5(d) respectively, were made by using a regular FDM 0.015”D nozzle. It took 4.6 hours build time to

make one human head model with 0.007" slice thickness at 0.5"/sec speed. During the FDC process of these parts, there was no human intervention and no process interruption. It has been shown that the current FDC process has great potential and the flexibility to use small size nozzles (0.015"D) and therefore improve the part surface quality. The surface quality of the parts made out of the 0.015"D nozzle are much better than using 0.025"D nozzle, and in some applications, may not require further machining.

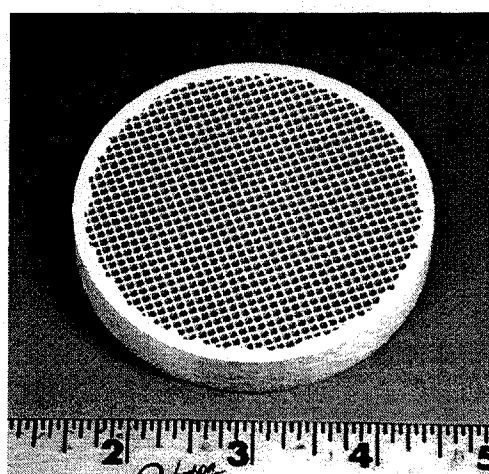
*Table III: Data Sheet for Each of the Port Plates, Turbine Blades, and the Radome and Cellular Filter Made Out of GS-44 Silicon Nitride.*

FDC Part	FDC Part Dimension	FDC Build Speed (in/sec)	RU955 Si <sub>3</sub> N <sub>4</sub> Filament (ft)	FDC Build Time (hour)
Port Plate	2.7"Dx0.6"tall	0.5	65	9.5
Turbine Blade	1.5" tall	0.5	24	4
Radome	4.5"Dx7.5"tall	0.5	117	16
Cellular Filter	3"Dx0.5"tall	0.5	41	4.3

Among all of the parts made recently, the largest part is the section of a missile radome shown in Figure 6 designed by Lockheed Martin. The radome section is 7.5" tall and the base section diameter is 4.5". This radome was made in 16 hours at 0.5"/sec speed and used approximately 117 ft of FDC filament. It was manufactured with total computer control in a fully automated process. There was not a single interruption during the 16 hour run. The RU955 GS-44 Si<sub>3</sub>N<sub>4</sub> filament was used just like the ICW-05 wax filament, i.e. it was on a spool and automatically and continuously fed from the back of the 3D Modeler. During the entire process the Modeler's door was kept closed and the build envelope temperature was controlled at 40°C. A 0.015"D nozzle was used and there was no nozzle clogging. The surface quality of the radome is excellent even without green finishing. So far, this is the longest uninterrupted FDC build since the system has been modified.



*Figure 6: Green silicon nitride Lockheed Martin's radome made by FDC.*



*Figure 7: Green silicon nitride cellular filter substrate made by FDC.*



Another complex part that has been made is the cellular filter substrate, which was designed and made for use as a sintering base as shown in Figure 7. This part is 3" in diameter and 0.5" thick. Because of its fine mesh structure, it is difficult and expensive to manufacture by the standard commercially available manufacturing processes. The filter was made in a 4.3 hour FDC build at 0.5"/sec speed with a 0.015"D nozzle, without interruption. The mesh structure was created by specifying a positive 0.040" offset between raster roads for the internal fill of the disk. The green cell wall thickness was 0.025", and the overall part quality was excellent.

## **7. Conclusion:**

The success of the FDC manufacturing process demonstrated in this paper is mainly due to the recent efforts on improved filament fabrication, feedstock material standardization, new FDC build strategies, hardware modifications, and improved process control. The degree of automation for high quality FDC process has been increased dramatically. Now, the FDC process is capable of making complex, fully dense, and high quality ceramic parts using a fully automated FDC process. Since January, 1997, we have made approximately 100 parts using FDC without human intervention or interruption. This success has demonstrated the capabilities of the improved FDC process.

Further improvements in the areas of elimination of material flow variation, elimination of contamination, and good tolerance (shrinkage) control need to be made. Increasing the build speed along with using a fine sized liquefier nozzles need to be investigated further. Better process control and improved surface finishing are needed for commercialization [10].

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