Shrinkage and Deformation in Components Manufactured by Fused Deposition of Ceramics

Joseph J. McIntosh, Stephen C. Danforth
Center for Ceramic Research,
Department of Ceramic and Materials Engineering,
Rutgers - The State University of New Jersey
Piscataway, NJ 08854

Vikram R. Jamalabad
AlliedSignal Research and Technology,
Morristown, NJ 07962

Abstract

Fused Deposition of Ceramics (FDC) presents a new processing technique that may contribute to anisotropic shrinkage and deformation, which are critical issues in the manufacture of ceramic components. The aim of this study is to identify and quantify key FDC parameters and their influence on shrinkage and deformation. The study was divided into two focus areas. The first was the effect of the FDC build parameters on the shrinkage of ceramic parts. The second focused on the interaction of the FDC build process with the geometrical features of a part. A series of experimental design techniques have been implemented in order to gain a thorough understanding of said parameters, as well as any possible interactions between parameters Studies have been conducted across each processing step, from the green manufacture of the part, through binder removal, and sintering. The data and knowledge gained from these experiments will allow us to redesign the original CAD component files to compensate for the shrinkage and deformation encountered when using the FDC technique.

Introduction

*S*olid *F*reeform *F*abrication is used to make three dimensional bodies from a computerized design (CAD file). Most SFF techniques use a wax or polymer system to fabricate parts. There have, however, been advances in the process of making metal as well as ceramic parts using established SFF processes. *S*elective *L*aser *S*intering (SLS), *L*aminated *O*bject *M*anufacturing (LOM), 3D-Printing, and *F*used *D*eposition (FD), show promise for the manufacturing of metal or ceramic components. The common factor between all these techniques is the layerwise building process. The computer reads in a CAD file and generates a series of continuous layers called slices, which when superimposed upon one another, recreate the three dimensional CAD file. The computer then sends the generated data to the tool which then physically builds the part one layer on top of another. These are all additive processes which differ largely from the normal subtractive process, by which a bulk part is machined to reach the desired shape. This process of material addition potentially imparts a unique set of defects and stresses which can result in warpage and anisotropic shrinkage in ceramic as well as metal parts, and is the focus of this research.[1]

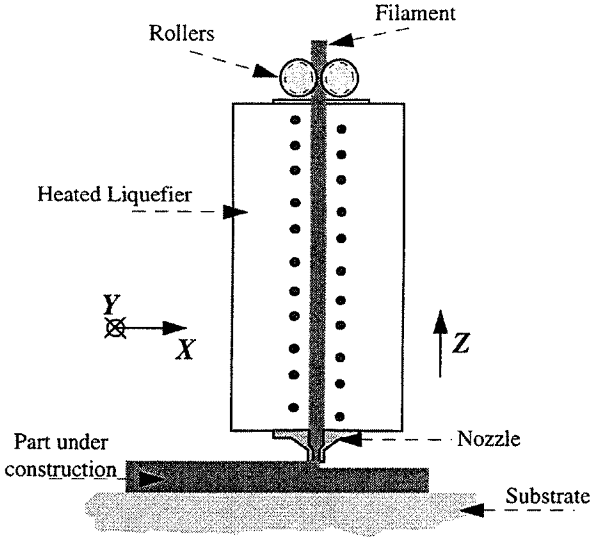

Figure 1: Schematic of Fused Deposition Liquefier

Ceramic feedstock for FDC is obtained by dispersing 55 vol% silicon nitride (GS-44) powder into an appropriate binder system.[2,3] Batch compounding occurs in a Haake torque rheometer where the surfactant coated powder is mixed with the molten binder.[4] The binder series used with GS-44 consists of approximately 20% elastomer, 15% tackifier, 30% wax, and 35% polymer.[2] The feedstock is extruded into a continuous filament 70 ± 1 mils in diameter.[3] Filament issues critical to successful FDC include flexibility, stiffness, viscosity, and adhesion or 'tack' behavior.[4] The filament is aged and spooled until ready for use. When ready, the filament is fed through two drive rollers into a liquefier where the binder material is melted, see Figure 1. The filament, which is continuously fed in, acts as a piston to drive the molten material through the liquefier. The liquefier moves in the X-Y direction and deposits material onto the substrate through a nozzle, generally 15 to 25 mils in diameter. In this manner, a singular layer is fabricated. The fill pattern of an individual layer can be manipulated during the setup of the computer file for the part. Once the layer is complete, the foam substrate is lowered and a new layer is built immediately over the previously built layer. The process continues until the entire part has been built. The binder is then removed and the part is sintered. The FDC process has been successfully demonstrated for silicon nitride, fused silica, aluminum oxide, lead-zirconate-titanate, stainless steel, and tungsten carbide cobalt.[2]

Procedure for Designed Experiments

Samples were fabricated using a modified Stratasys Inc. 3D Modeler™. The perimeter, contour, and rasters were made using a road width of 20 mils, and a slice thickness of 10 mils. A single contour was placed adjacent to the perimeter. The remainder of the layer was filled with rasters with the desired orientation. Negative offsets were used between adjacent roads, see Figure 2. The X-Y build speed was 500 mils per second. All other software values were held constant with the Quickslice™ default settings for ICW05, an investment casting wax developed by Stratasys for fused deposition modeling. Flow rate for the Modeler was set to 188%, and a nozzle with an inside diameter of 25 mils was used. The filament used was RU955, which is the RU9 binder system, at 55 volume % GS-44 silicon nitride powder.

The first series of experiments examined the effect of the FDC build parameters on the shrinkage of a rectangular bar 1"x ½"x ¼", see Figure 3. Liquefier temperature, modeling envelope temperature, and an aligned raster fill pattern of either 0° or 90°, relative to the long axis of the bar, were varied, see Table I. Parts were built directly on the foam substrate. Several samples were made with the conventional deposition method where alternating layers have roads aligned at 45°/-45° ("cross hatched" samples).

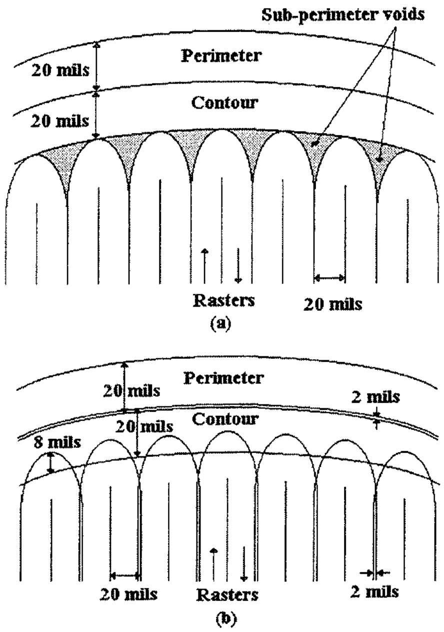

Figure 2: a) No offset b) Negative offset

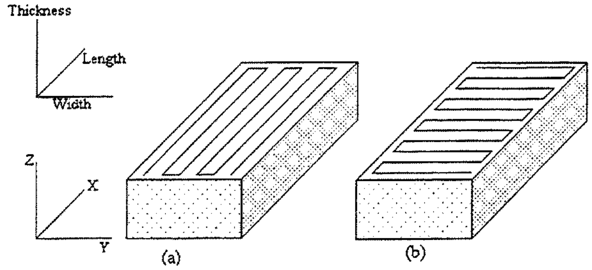

Figure 3: Fill patterns used a) 0° fill and b) 90° fill

Table I: FDC Build Parameters

	low	high
liquefier temperature	180° C	190° C
envelope temperature	40° C	50° C
road fill orientation	0°	90°

The second experiment investigated the effect of the FDC build process on geometrical features. The standard part geometry was a 1"x 1" plate, square on one side and hemispherical on the other, see Figure 4. To this standard plate was added in its center either a hole or a protrusion of diameter ¼" or ½". Build parameters were now held constant for all samples. The fill pattern used was a 45°/-45° cross hatch. Samples were built on a base five layers thick, which was removed prior to evaluation.

(a) (b) (c)

Figure 4: Various parts of the feature-based experimentation a) 1/2" or 1/4" hole, b) standard, c) 1/2" or 1/4" protrusion

Upon completion, all parts were stored in a low humidity chamber until ready for binder removal. Binder removal was accomplished in a tube furnace in a two stage process. In the first stage, parts were embedded in activated carbon and heated to 350° C in a nitrogen atmosphere. The second stage involved heating the samples to 450° C in an air environment.[5] The parts were then gas-pressure sintered under a schedule proprietary to AlliedSignal, Inc.

Characterization occurred in the green, brown (post binder removal), and sintered states. Measurements of length, width, thickness, and weight were taken. Shrinkage and weight loss percentage were calculated using the equation:

$$(X - X') / X * 100 = Y$$

where X is the original value, X′ is the new value, and Y is the percent change in the value. The experimental design program DOE KISS was used to analyze the results.

Results and Discussions

Experiment #1, CAD-Green

The Quickslice™ software generates an .sml file which the liquefier uses to direct its movement during the FDC build. In nearly all cases, it was found that the part was built larger than that specified by the .sml file, see Table II. Build direction was found to be the only factor tested which affected the dimensions in the build plane. The data shows the average shrinkage values calculated for both the 0° and 90° samples in the length and width direction. There are several phenomena which may account for the part being built larger than specified.

Table II: Cad-Green*

Build	0°		90°	
length	-0.26% ± 0.58		-1.65% ± 0.33	
width	-1.46% ± 0.61		-2.45% ± 1.48	
Liq Temp	180°	190°	180°	190°
thickness	-4.71% ± 1.15	-5.87% ± 2.22	-8.96% ± 1.93	-11.61% ± 2.97

* Note that a negative shrinkage indicates a final part dimensional larger than the .sml file

First, the part may be slumping due to insufficient stiffness while still in the modeling envelope. Too high a modeling envelope temperature may result in slumping. Within the temperature range tested here, 40° and 50° C, the modeling envelope temperature was not found to affect the green part size. Further testing at a broader temperature range may be required. The oversize in the samples may also have been due to the method used to eliminate sub perimeter voids. The mechanism of void filling employed was a single contour deposited just inside the perimeter of the sample which acts as a buffer layer into which rasters would plow, see Figure 2. There was a negative offset of 8 mils between the raster and contour fill which would provide excess material at their junction in order to prevent the formation of sub-perimeter voids. The large overlap would cause the rasters to plow into the semi-cool contour which would in turn push out into the perimeter and result in the perimeter being displaced outward from its original location. The perimeter will then cool in the new location resulting in a part with oversize X-Y dimensions.

The excess in size in X, Y, and Z, shown by the 90° parts, above that shown by the 0° parts, may have been due to the larger amount of excess material generated. The 90° samples required a larger number of roads to fill in the layer, as they had shorter path lengths. The higher number of roads contributed to more contour/raster interactions and resulted in even more excess material being deposited in the sub-perimeter region. With layer upon layer, this amount of extra material grew and would account for the 90° parts being oversized by a larger amount than the 0° parts. This was reinforced by the average original weights of the samples, 4.59g ± 0.09 for the 0° samples and 4.72g ± 0.09 for the 90° samples.

The data also shows that the length, or the 1" dimension, for both the 0° and 90° fill pattern was more accurate, or closer to the intended size, than the width, or ½" dimension. This was due to the greater effect of surface roughness on error when measuring the smaller dimension, and is reinforced by the data gathered for the cross hatched samples. They showed a similar effect, whereas the length had a shrinkage of -1.86% ± 0.15 and width a shrinkage of -3.15% ± 0.50.

The liquefier temperature was found to affect the part thickness, see Table II. At a liquefier temperature of 190°C, the deviation from theoretical was greater than at 180°C, and the cause for this is uncertain at present. From these results, we have determined that the present method of sub-perimeter void elimination is inadequate for the desired tolerances and contributes to a large variation in green part size dependent on the path length of the road, and the build pattern. Dog-earing, which alters the liquefier's path in the sub-perimeter region, will be revisited as an alternative to overfilling.[6]

Recall that all parts fabricated in this first experiment have an aligned raster pattern whereby all the roads in the part are deposited in the same direction. Normal FDM or FDC does not employ this type of fill, but instead uses a cross hatch pattern where the road direction is at 90° to the road direction of the previous layer. The rasters have been aligned in this study in order to gain an understanding of the effects of individual layers on shrinkage and warpage. This knowledge is vital when designing a part with very thin sections, high aspect ratios, or tight dimensional tolerances. One must be able to compensate in the CAD file for any differential shrinkage due to differences in the vector lengths of the fill.

All of the parts built, including the 0°, 90°, and cross hatched samples, were warped in the green state. Prior to removal from the foam substrate, it was evident that the parts were warped about an axis normal to that of the raster direction as indicated by the arrows in Figure 5. This may be inherent to the process by which a hotter layer is deposited onto a colder layer. On cooling, the hot layer is put in tension and the cold in compression due to both the higher thermal expansion of the hotter material, and its viscoelastic behavior. It is also possible that stresses are induced in the part by the repeated shearing of the deposited material. The liquefier increases its position by 10 mils for each new layer. Any excess material, above the 10 mil thickness will be sheared by the nozzle movement while depositing the next layer. With this material in its semi cool state, the shearing stresses may become significant. Warpage for the 90° samples was found to be 0.06%/in ± 0.28, while the 0° samples showed 0.53%/in ± 0.29. Extra samples were fabricated and green machined in order to eliminate the warpage found in the green state. They were then burnt out and sintered. There was no further warpage in the brown state, but after sintering it was apparent that some subsequent warpage had occurred. Future work will take a detailed

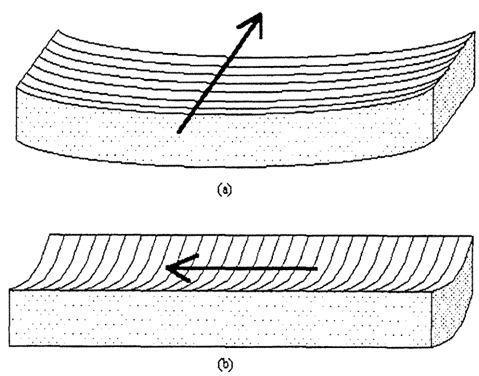

Figure 5: Warpage in a) 0° samples and b) 90° samples

look through each subprocess using sample geometries specifically designed to facilitate the study of warpage.

Green to Brown

Shrinkage in the X-Y plane was found to occur after binder removal, see Table III. It was found that more shrinkage occurred in the road direction than normal to it (within a given layer). This was contradictory to previous notion that the most shrinkage should occur in the direction normal to the roads. This notion stemmed from the hypothesis that there may be small density gradients found across a layer. The road center was thought to be slightly more dense than its edges, resulting in a less dense area between roads, which would shrink more upon binder removal and sintering. The fact that this is not the case suggests that reevaluation of the density gradients is required, or that there is yet unexplained phenomena occurring. A possible explanation is the relief of the viscoelastic tensions imparted to the roads during deposition, as described previously. DMA and thermal expansion tests will be carried out to understand the relaxation behavior of the RU955.

Table III: Green-Brown Shrinkage and Weight Loss

thickness	1.62% ± 0.83	
weight	19.39% ± 0.15	
Build	0°	90°
length	1.34% ± 0.31	0.97% ± 0.23
width	0.93 ± 0.40	1.31% ± 0.61

The cross hatched samples exhibited comparable brown shrinkage's of 1.32% ± 0.13 in the length and 1.22% ± 0.29 in the width. The anisotropy found in the aligned rasters was not visible here because the road directions were not isolated as before. The thickness showed a 1.62% ± 0.29 shrinkage.

Neither the liquefier temperature, environment temperature, or the build pattern was found to effect the shrinkage (from the green to brown state) in the build, or Z direction. The Z shrinkage was larger than either X or Y, however. This is common to parts made through layering processes and may stem from somewhat poorer bonding or lower densities between subsequent layers. At the conclusion of these experiments, it will be possible to compensate for this anisotropy in the software. The weight loss on burnout was also found to be independent of the three tested factors, as expected. Weight loss averaged 19.39% ± 0.15 for all parts tested.

Brown to Sinter

None of the FDC build factors tested had any effect on the shrinkage from the brown to the sintered state, see Table IV. The density of the samples was calculated by Archimedes' method to be 3.23 ± 0.06 g/cm^3, consistent with isopressed samples of GS-44 silicon nitride. There was some weight loss during sintering, which is typical for GS-44.

Table IV: Brown-Sintered Shrinkage and Weight Loss

length	16.10% ± 0.36
width	16.45% ± 0.53
thickness	18.06% ± 0.51
weight	0.85% ± 0.11

Table V: Bulk sample shrinkages through processing stages

	length	width	thickness
Green-Brown	0.95% ± 0.26	0.89% ± 0.48	0.97% ± 1.83
Brown-Sintered	16.14% ± 0.47	16.12% ± 0.40	17.65% ± 1.77
Green-Sintered	16.94% ± 0.50	16.85% ± 0.52	18.44 ± 1.12

Experiment #2

The second experiment examined the effect of various features on the bulk shape. This would provide information about the ability to hold tolerances on the critical features of complex parts. Samples were built with various features as shown in Figure 4. The goal of this experiment was first to determine whether or not a particular feature, such as a hole or a protrusion, would affect the shrinkage behavior of a known sample made by the FDC process. Second, the size dependence of the features was to be examined. The final objective was to observe the effect of the FDC processing parameters on the feature itself. Each subprocess was examined in a similar manner to the previous experiment.

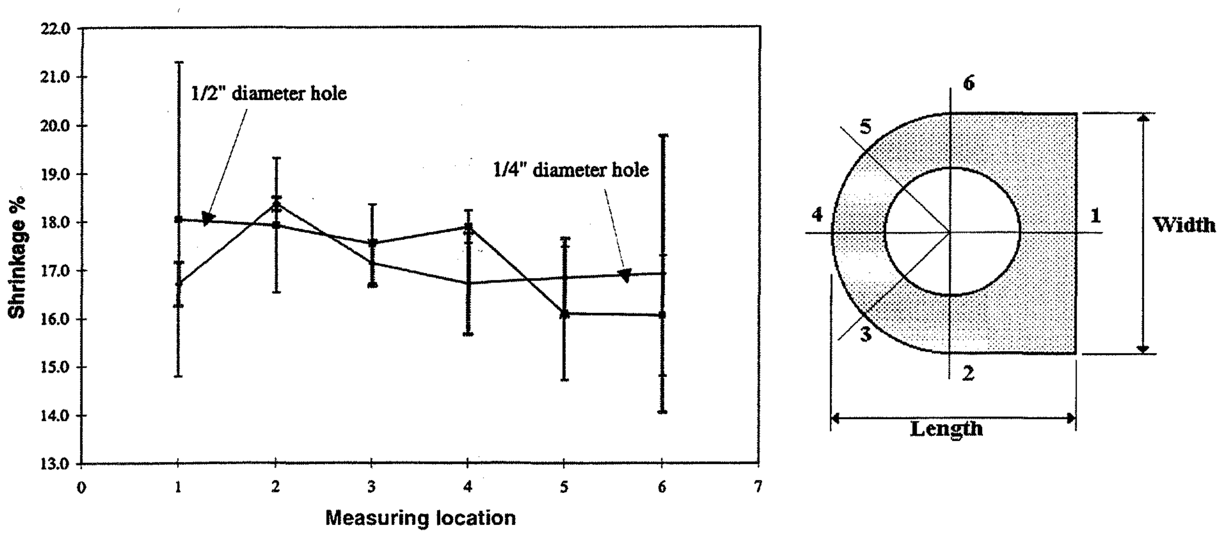

Figure 6: Dimensional variations from Green to Sintered stages for feature experiment samples, measurement location is shown to the right.

It was found that none of the geometric features employed significantly affected the shrinkage in the bulk part through any stage of processing. Data for the processes is shown in Table V. This is encouraging in that it suggests that if the part is fully dense in the green state, complex shapes are possible without the need for specific corrections according to feature shape. Figure 6 shows the shrinkage around the sample with the 1/4" and the 1/2" holes. Shrinkage's for both diameters were indistinguishable. The green holes themselves were smaller in diameter than designed, due once again to the overfilling as described previously. The 1/2" and the 1/4" diameter protrusion shrinkage's were also insignificantly different from one another. It was observed, however, that the base of the protrusion was slightly larger in diameter than regions above it. This enlargement affected only the first 3-5 layers of the protrusion. It is surmised that this may be another result of the subperimeter void fill method and further experimentation is ongoing.

Conclusions

Overfilling was determined to be a poor choice for use as a sub-perimeter void fill. It resulted in oversize parts and may have contributed to deformation at the base of some of the features. Road direction was found to influence shrinkage in the X-Y plane during binder removal. Shrinkage was larger in the road direction than normal to it. The FDC build parameters tested did not affect the shrinkage during the sintering stage. Warpage was found to occur in the green and sintered states and was about an axis normal to the direction of the roads. It was determined that geometrical features did not significantly affect the shrinkage of ceramic components manufactured by fused deposition. The samples tested do not, however, have features that approach the thickness of singular roads, nor the complexities possible with the 3D Modeler. Further studies will examine thinner cross section and more complex features.

Acknowledgments

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