Application of Design of Experiments to Extrusion Freeform Fabrication (EFF) of Functional Ceramic Prototypes

J. Walish, M. Sutaria, M. Dougherty, R. Vaidyanathan.
Advanced Ceramics Research, Tucson, AZ 85706

S. Kasichainula, P. Calvert. University of Arizona
Arizona Material Laboratories, Tucson, AZ 85712

K. Cooper. NASA Marshall Space Flight Center, Huntsville, AL 35812

Extrusion Freeform Fabrication (EFF) is an adaptation of the Stratasys Fused Deposition Modeling (FDM™) process for the Solid Freeform Fabrication (SFF) of functional ceramic prototypes. It is a complex process involving many process variables, including parameters that are operation, machine, materials, and geometry specific. A Taguchi factorial Design of Experiments (DOE) technique was utilized to study the effects of machine specific process parameters as well as their interactions based on the mechanical and physical properties of sintered ceramic specimens. Post-processing software was developed to control and modify these parameters. This software interface was designed to mimic the Quickslice™ interface for setting motion parameters based upon the material and the operation. The results of this investigation provided useful information for the experimental analysis of the machine specific process parameters. Suitable parameters were selected for the EFF process for fabricating representative ceramic prototypes. With the optimized parameters, complicated parts were successfully fabricated using both Kyocera SN282 and Starck M-11 silicon nitride powders.

I. Introduction

Extrusion Freeform Fabrication (EFF) is an SFF technique based on the Stratasys FDM™ approach[a] for the fabrication of functional ceramic prototypes [1]. More details are given in reference 2. While possessing the benefits of Fused Deposition of Ceramics (FDC™) [1995 Volume from reference 1] and FDM™, EFF has the added advantages that it can handle higher viscosity feed stock materials and higher extrusion temperatures compared to FDC™ and FDM™. Similar to other SFF techniques, EFF also allows the sequential deposition of multiple layers to form a complex ceramic shape. This has been achieved by retrofitting a high-pressure extruder head to a Stratasys FDM™ modeler (figures 1 and 2). The CAD file is processed by the Quickslice™ software and used to control the EFF high-pressure extrusion head [1]. However, the operation parameters that are automatically set by Quickslice, are optimized for polymer filament type of feedstock. These parameters are not necessarily the optimum parameters for the SFF of ceramic parts. To further control process parameters for SFF of ceramic parts, SML Post™, a Visual Basic based post-processing software was developed to modify selected process parameters. SML Post™ could modify the start-delay, preflow, start-flow, start-distance, main-flow, shutoff-distance, roll-back, speed, and acceleration that were originally set by Quickslice™. Presently, the road-width, slice-thickness, and fill-patterns are still set by

[a] Stratasys Inc., Eden Prairie, MN

Quickslice. However, SML Post™ could modify these parameters also, if needed.

An examination of the initial ceramic parts that were built using EFF suggested that further optimization was needed to improve the part quality and surface finish of the parts. This was the motivation for the work presented here. The optimization could be performed on both machine and material parameters. Material parameters include the type of ceramic powder used, the particle size distribution of the powder, the type of sintering aides used, and the type of binder system. Machine parameters are items such as extrusion temperature, nozzle diameter, modeling envelope temperature, and percent flow (flow rate). For the work presented here, only machine specific parameters were optimized using the Taguchi method of Design of Experiments (DOE) [3, 4].

Figure 1. Stratasys FDM™ retrofitted With a high pressure head

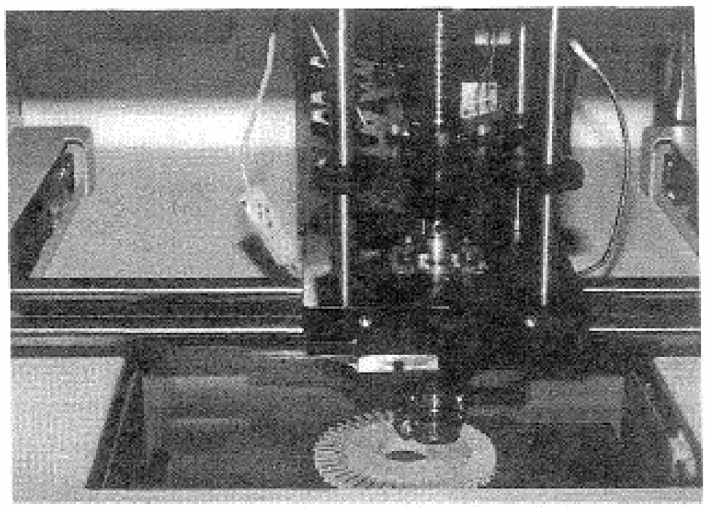

Figure 2. Close up view of the high pressure head creating a ceramic prototype

II. Experimental Procedure

An optimization of machine parameters was undertaken in order to improve part quality and surface finish. The part features used for optimization were dimensional accuracy, surface finish, and the time taken to build the sample. In order to reduce the number of experiments, the number of variables was reduced to seven. The same .sml file was used for all of the trials to reduce the number of variables. These variables could be changed using the post processing software (SML Post). The start distance and shutoff distance were held constant at values previously determined as optimal. These numbers were obtained by trial and error. Earlier work had produced a binder system that demonstrated favorable extrusion qualities when compounded with the ceramic powder [2]. The same ceramic feed stock was used for the optimization experiments. It is expected that minor modifications of the feedstock would result in similar optimized parameters for the EFF of those feedstocks.

A. Optimization of the Perimeter Operation

The Taguchi method of Design of Experiments (DOE) was used for the statistical experimental design. Within the four types of operations used to deposit the material (perimeter, contour, raster, and open), the parameters for perimeter were optimized first due to the relative ease and speed at which the experiments could be performed. A two level experiment with seven variables was performed to select and screen the most critical variables. The variables chosen for the screening experiment were start delay, pre-flow, start flow, main flow, rollback, speed, and acceleration. A description of the seven variables is given in table I.

A Taguchi L12 array with three repetitions was chosen. With three repetitions, there was a 95% confidence level that a term identified as being significant truly belongs within the standard deviation as well as a 90% chance of finding a significant term if one actually existed. At this point, the predetermined high and low values of the different variables were entered into a computer program which created and analyzed DOE experiments (see table II). The software is called "Keep it simple statistically or KISS" supplied by Digital Computation. A three-level set of experiments was conducted to optimize the variables.

Cylinders with a diameter of two inches and a wall thickness of one road per layer were fabricated. The cylinders were built to a height of 0.2 inches. An exit distance of 1.25 inches was used in all cases. Twelve samples were made for each repetition.

The parts were analyzed in a number of ways. Thickness measurements from three points on the cylinders were taken for all 36 parts. The thickness before and after the starting point, and exactly opposite the starting point were measured. The standard deviation in part dimensions between the three points on each cylinder was calculated. The average thickness, the average excess (an average of the measured thickness minus the thickness specified in the .sml file), time, and the end thickness minus the beginning thickness were also determined. By analyzing the statistics and graphs returned by the DOE software, the most important factors were found. The DOE software analyzed the measured responses and calculated a factor known as "P(2 tail)". According to the DOE rule of thumb, if the "P(2 tail)" number for a particular variable is below 0.05, then that variable should be considered to belong in the regression model. If the "P(2 tail)" value was between 0.05 and 0.10, then that variable was marginally important. However, this variable would also be included in the three-level set of experiments. A variable with a higher value of "P(2 tail)" would be considered not to belong in the regression model.

A Taguchi L9 matrix was created for the three-level experiments. The variables were chosen based on the screening experiments. The low and high values were retained from the screening experiment, while the middle value was an average of the low and high values. The parameters with a P(2 tail) number above 0.10 were set at values thought of as optimum, based on previous experimental results. The three-level Taguchi L9 matrix is shown in table III. The difference between the start and finish thickness of the parts created was measured and analyzed by the software. The optimized values of the machine parameters for obtaining the smallest difference in start and finish thickness were calculated by the DOE software. Several test parts were made with the optimized parameters.

B. Optimization of the Raster Operation

As with the perimeter optimization, raster optimization was carried out using the Taguchi DOE techniques. The two-level experiment for the raster was done with the same seven variables as the perimeter two-level experiment plus the road width. The test shape for this set of experiments was a 1"x2" rectangular plate with a rectangular hole cut out of the middle and a uniform wall thickness of 1/8". Thirty-six (36) experiments were conducted. However, some of the parts were irregular and had a poor surface finish. Therefore, a decision was made to judge these parts on a strictly qualitative scale of 0 to 10, 10 being the best. A road width of 0.0030" was used for all the experiments, since this closely matched the optimum road width obtained with the 0.0016" nozzle used.

III. Results and Discussion

For optimizing the parameters for building the perimeter, the time to build the part and the difference in part thickness at the start and finish were used as responses and analyzed by the DOE software. Since the build time yielded only one definite important factor (speed) and one possibly important factor (main flow), the difference in thickness at start and finish was used in constructing the three-level experiment. The effect of the different parameters on the difference in part thickness at the start and finish is shown in figure 3. The slope of the marginal means plot in figure 3 is indicative of the significance of the different build parameters. From figure 3, it was seen that the start-delay, main-flow, rollback, and speed were significant factors for the optimization of the perimeter. The Taguchi L9 matrix created for the three-level experiments based on these results was shown in table III. The difference between the start and finish thickness of the parts created was measured and analyzed by the software. The program returned values for the P(2 tail) which confirmed that all of the variables that were used at this experimental stage were significant in minimizing the difference between the start and finish thickness. The optimized values of the machine parameters for obtaining the smallest difference in start and finish thickness are shown in table IV. These values are for a particular powder/binder system and tip size. Further optimizations will have to be performed in order to obtain the same kind of results for different tip sizes and different feedstock material. In the case of raster, the start-delay, main-flow, roll back, speed and road width parameters were observed to be the most significant.

It was found that when using the optimized parameters, the surface finish of the parts was greatly improved with respect to parts that were built previously without optimization. Four test cylinders were made using the optimized parameters for perimeter. The most dramatic change occurred with the build speed. The build speed suggested by the optimization program was 0.3" per second, compared to 0.8" per second before optimization. The surface finish and the seam where the layer was started and finished showed no pooling of excess material. Figures 4a and 4b shows cylinders that were made with minor changes in the parameters, while figure 4c shows a cylinder that was built with the optimized parameters. Sample parts with good surface finish and part quality was built using the optimized raster parameters. As indicated above, optimum part quality was obtained with a road width of 0.030" when using a tip size of 0.016". Further optimization experiments are in progress with tip sizes of 0.012" and 0.025".

Mechanical testing was completed for Starck silicon nitride samples fabricated with the optimized conditions. Prior tests had shown that post-polishing provided increases in strength. Samples were made with both 0 degree and alternating 0/90 raster directions. The strength values for all test bars can be seen in table V. For samples with a 0 degree raster build, the strength data was similar for polished and unpolished specimens. The test bars that were made with 0 and 90 degree alternating raster angles showed poor surface finish, compared to 0 degree only, and thus the data shows a difference in strength values for polished and unpolished samples. Because of the geometry of the test bar, a 90 degree raster angle is a poor choice since the road would only be about 0.1 inches long. The smaller the road, the more chances of introducing a defect into the part.

Other silicon nitride powders have been used for EFF with the same binder system and extrusion conditions with favorable results. Two sample parts fabricated using the optimized conditions are shown in figures 5 and 6. In figure 5, green parts made with Kyocera SN282 silicon nitride and Starck M-11 silicon nitride powder are shown. Figure 6 shows a turbine blade that was made with the optimized parameters.

IV. Conclusion

This investigation has established the feasibility of using DOE techniques for the optimization of the EFF process when creating functional ceramic prototypes. For building the perimeter, the parameters that control the EFF process quality are start-delay, main flow, speed and rollback. For the raster, the start-delay, main-flow, roll back, speed and road width parameters were observed to be the most significant. It has been demonstrated here that with the optimized parameters, the overall part quality was improved. Complicated ceramic shapes were built by the EFF process using the optimized parameters. Currently more optimizations are planned for other tip sizes.

V. Acknowledgements

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Table I: A description of the software controlled (process) parameters.

Parameter	Description
Start delay	The lag time between layers
Pre-flow	Extrusion rate during stops
Start flow	Extrusion rate at the start of the exit distance
Start distance	Distance from start point for main flow to start
Main flow	Extrusion rate over the bulk of the layer
Shutoff distance	The distance from the stop point for main flow to stop
Rollback	The rate the material is pulled back to reduce the flow
Speed	The rate at which the head moves along a set path
Acceleration	The rate at which the extrusion head changes speed

Table II: The Taguchi L12 matrix used in the screening experiment for perimeter optimization.

Factor	A	B	C	D	E	F	G				
Row #	Start Delay	Preflow	Start Flow	Main Flow	Rollback	Speed	Acceleration		Y1	Y2	Y3
1	0	49	51	50	141	200	1				
2	0	49	51	50	141	1000	9				
3	0	49	127	176	255	200	1				
4	0	127	51	176	255	200	9				
5	0	127	127	50	255	1000	1				
6	0	127	127	176	141	1000	9				
7	1	49	127	176	141	200	9				
8	1	49	127	50	255	1000	9				
9	1	49	51	176	255	1000	1				
10	1	127	127	50	141	200	1				
11	1	127	51	176	141	1000	1				
12	1	127	51	50	255	200	9				

Table III: The Taguchi L9 matrix used for the three-level experiments for optimizing perimeter.

Factor	A	B	C	D
Row #	Start Delay	Main Flow	Rollback	Speed
1	0	120	141	200
2	0	148	199	600
3	0	176	255	1000
4	0.5	120	199	1000
5	0.5	148	255	200
6	0.5	176	141	600
7	1	120	255	600
8	1	148	141	1000
9	1	176	199	200

Table IV. The optimized parameters obtained with DOE for EFF

Optimized parameters	
Start delay	0.82 sec
Preflow	79
Start flow	89
Start distance	0.06"
Main flow	128%
Shutoff distance	0.073"
Rollback	229
Speed	0.293"/sec
Acceleration	5

Table V. Flexural strength data for EFF test bars as a function of raster-build direction

Raster-build direction	Surface condition	Flexural strength (MPa)
0°	Unpolished	594 ± 80
0°	Polished	613 ± 12
0°/90° alternating	Unpolished	227 ± 39
0°/90° alternating	Polished	312 ± 71

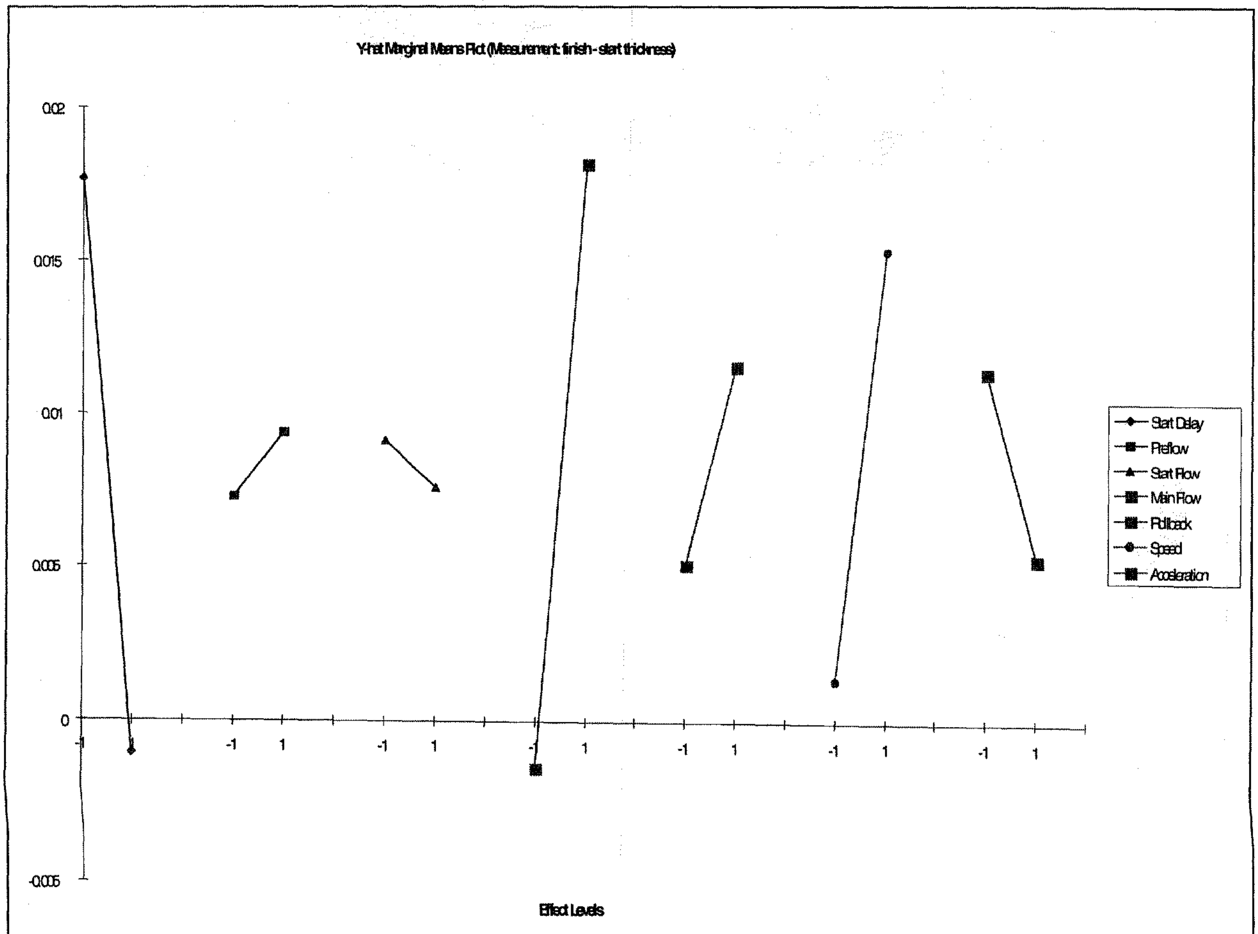

Figure 3: The marginal means plot based on the difference in start and finish thickness.

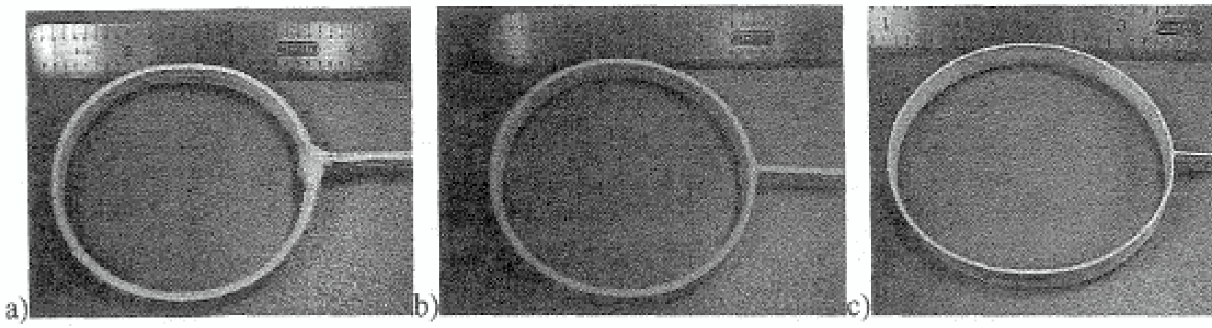

Figure 4: Cylinders a and b were fabricated prior to optimization. Cylinder c was constructed with the optimized parameters.

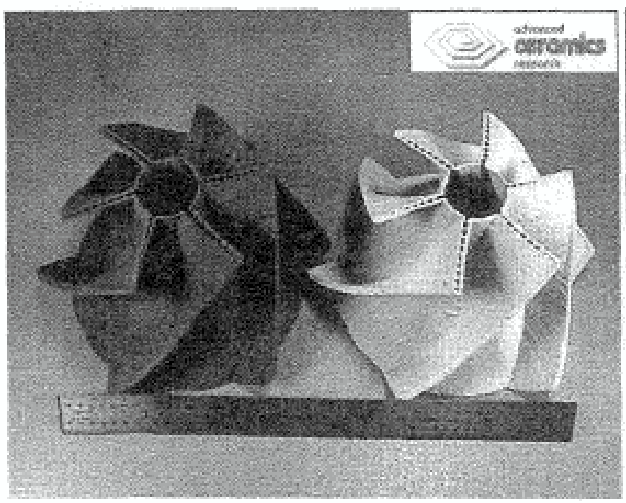

Figure 5: Impellers made with Kyocera and Starck silicon nitride powders.

Figure 6: A turbine blade made with the new parameters.