



**AFRL-RX-WP-TP-2008-4314**

**AQUEOUS-BASED EXTRUSION FABRICATION OF  
CERAMICS ON DEMAND (PREPRINT)**

**Michael S. Mason, Tieshu Huang, Robert G. Landers, Ming C. Leu, Gregory E. Hilmas,  
and Michael W. Hayes**

**University of Missouri-Rolla**

**JULY 2007**

**Approved for public release; distribution unlimited.**

*See additional restrictions described on inside pages*

**STINFO COPY**

**AIR FORCE RESEARCH LABORATORY  
MATERIALS AND MANUFACTURING DIRECTORATE  
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750  
AIR FORCE MATERIEL COMMAND  
UNITED STATES AIR FORCE**

<b>REPORT DOCUMENTATION PAGE</b>				<i>Form Approved</i> OMB No. 0704-0188	
<p>The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. <b>PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.</b></p>					
<b>1. REPORT DATE (DD-MM-YY)</b> July 2007		<b>2. REPORT TYPE</b> Conference Proceedings Preprint		<b>3. DATES COVERED (From - To)</b>	
<b>4. TITLE AND SUBTITLE</b> AQUEOUS-BASED EXTRUSION FABRICATION OF CERAMICS ON DEMAND (PREPRINT)				<b>5a. CONTRACT NUMBER</b> FA8650-04-C-5704	
				<b>5b. GRANT NUMBER</b>	
				<b>5c. PROGRAM ELEMENT NUMBER</b> 78011F	
<b>6. AUTHOR(S)</b> Michael S. Mason, Tieshu Huang, Robert G. Landers, Ming C. Leu, and Gregory E. Hilmas (University of Missouri-Rolla) Michael W. Hayes (The Boeing Company)				<b>5d. PROJECT NUMBER</b> 2865	
				<b>5e. TASK NUMBER</b> 25	
				<b>5f. WORK UNIT NUMBER</b> 25100000	
<b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b> University of Missouri-Rolla 1870 Miner Circle Rolla, MO 65409-0050				The Boeing Company	
<b>9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)</b> Air Force Research Laboratory Materials and Manufacturing Directorate Wright-Patterson Air Force Base, OH 45433-7750 Air Force Materiel Command United States Air Force				<b>10. SPONSORING/MONITORING AGENCY ACRONYM(S)</b> AFRL/RXLMP	
				<b>11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S)</b> AFRL-RX-WP-TP-2008-4314	
<b>12. DISTRIBUTION/AVAILABILITY STATEMENT</b> Approved for public release; distribution unlimited.					
<b>13. SUPPLEMENTARY NOTES</b> Conference proceedings for the 2007 Solid Freeform Symposium, Austin, TX, July 16, 2007. PAO Case Number: MLLM-0035; Clearance Date: 19 Jul 2007. This work was funded in whole or in part by Department of the Air Force contract FA8650-04-C-5704. The U.S. Government has for itself and others acting on its behalf an unlimited, paid-up, nonexclusive, irrevocable worldwide license to use, modify, reproduce, release, perform, display, or disclose the work by or on behalf of the U.S. Government. Paper contains color.					
<b>14. ABSTRACT</b> Aqueous-Based Extrusion Fabrication is an additive manufacturing technique that extrudes ceramic slurries of high solids loading layer by layer for part fabrication. The material reservoir in a previously developed system has been modified to allow for starting and stopping of extrusion process on demand. Design pros and cons are examined and a comparison between two material reservoir designs is made. Tests were conducted to determine the optimal deposition parameters for starting and stopping of the extrudate on demand. The collected test data is used to create a process model that describes the relationship between ram velocity and material extrusion rate. This model allows for the development of a deposition strategy that improves material deposition consistency, including reduced material buildup at sharp corners. Example parts are fabricated using the deposition strategy and hardware design.					
<b>15. SUBJECT TERMS</b> ceramic, aqueous-based extrusion fabrication, ram velocity, material extrusion rate					
<b>16. SECURITY CLASSIFICATION OF:</b>			<b>17. LIMITATION OF ABSTRACT:</b> SAR	<b>18. NUMBER OF PAGES</b> 18	<b>19a. NAME OF RESPONSIBLE PERSON (Monitor)</b> Todd J. Turner <b>19b. TELEPHONE NUMBER (Include Area Code)</b> N/A
<b>a. REPORT</b> Unclassified	<b>b. ABSTRACT</b> Unclassified	<b>c. THIS PAGE</b> Unclassified			

# **Aqueous-Based Extrusion Fabrication of Ceramics on Demand**

Michael S. Mason<sup>1</sup>, Tieshu Huang<sup>2</sup>, Robert G. Landers<sup>1</sup>,  
Ming C. Leu<sup>1</sup>, Gregory E. Hilmas<sup>2</sup>, and Michael W. Hayes<sup>3</sup>

1870 Miner Circle  
Department of Mechanical and Aerospace Engineering<sup>1</sup>  
Department of Materials Science and Engineering<sup>2</sup>  
University of Missouri-Rolla, Rolla, Missouri 65409-0050  
The Boeing Company<sup>3</sup>, St. Louis, Missouri  
{mmason,hts,landersr,mleu,ghilmas}@umr.edu  
michael.w.hayes2@boeing.com

## **ABSTRACT**

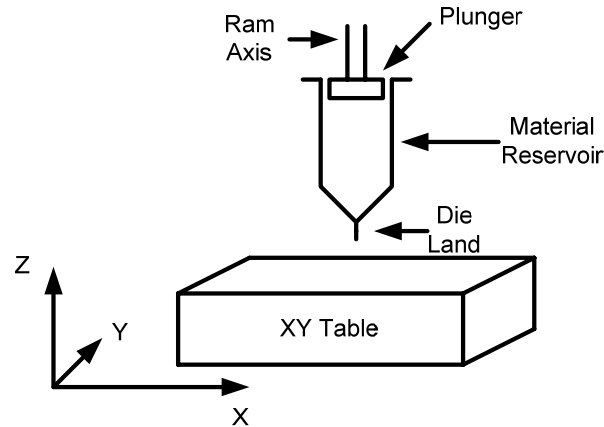
Aqueous-Based Extrusion Fabrication is an additive manufacturing technique that extrudes ceramic slurries of high solids loading layer by layer for part fabrication. The material reservoir in a previously developed system has been modified to allow for starting and stopping of the extrusion process on demand. Design pros and cons are examined and a comparison between two material reservoir designs is made. Tests were conducted to determine the optimal deposition parameters for starting and stopping of the extrudate on demand. The collected test data is used to create a process model that describes the relationship between ram velocity and material extrusion rate. This model allows for the development of a deposition strategy that improves material deposition consistency, including reduced material buildup at sharp corners. Example parts are fabricated using the deposition strategy and hardware design.

## **INTRODUCTION**

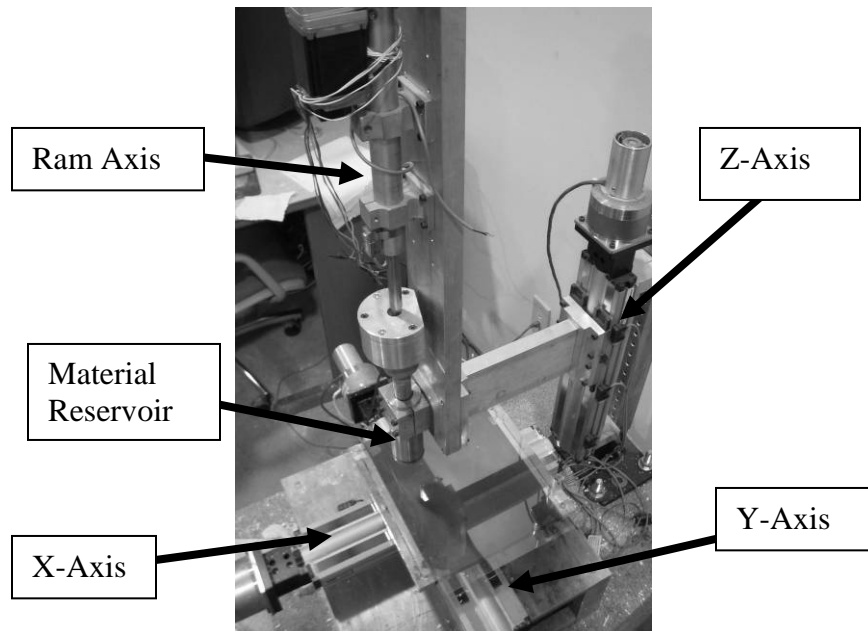
Aqueous based extrusion fabrication (ABEF) of ceramics is a Solid Freeform Fabrication (SFF) process that deposits ceramic paste layer by layer through extrusion. The process has been previously introduced, and details regarding the build material and equipment can be found in [1-3]. A process schematic and photograph are shown in Figures 1 and 2 respectively. The material deposition device previously developed gave good results for continuous material deposition. The ability to start and stop the material extrusion on demand was not yet developed in our previous work. With the need to be able to fabricate more complex geometries, different extrusion processes were surveyed for the process of starting/stopping material deposition on demand. The first system surveyed was the Fused Deposition of Ceramics (FDC) process, which modified a Fused Deposition of Materials (FDM) machine to deposit a ceramic/polymer fiber strand [4-5]. The FDM process uses two rollers with heaters to move the material fiber either forward for deposition or in reverse to stop the deposition. The second process uses a the Micro-Scale Robotic Deposition System [6]. This process uses air pressure to extrude material for deposition and uses one-way control valves for starting and stopping of the extrusion process. These “retraction” processes can be applied to the ABEF system by retreating

the ram extruder from touching the material reservoir plunger. This would in theory reduce the applied pressure, thereby stopping material extrusion.

In this paper material deposition tests are conducted, varying the ram extruder retreating velocity in order to develop a technique that can be used for starting/stopping the extrusion process on demand. A new extrusion mechanism is designed for improvements in process capabilities. Parts are then fabricated with the use of starting/stopping the extrusion on demand with emphasis being placed on deposition of sharp corners and edges.



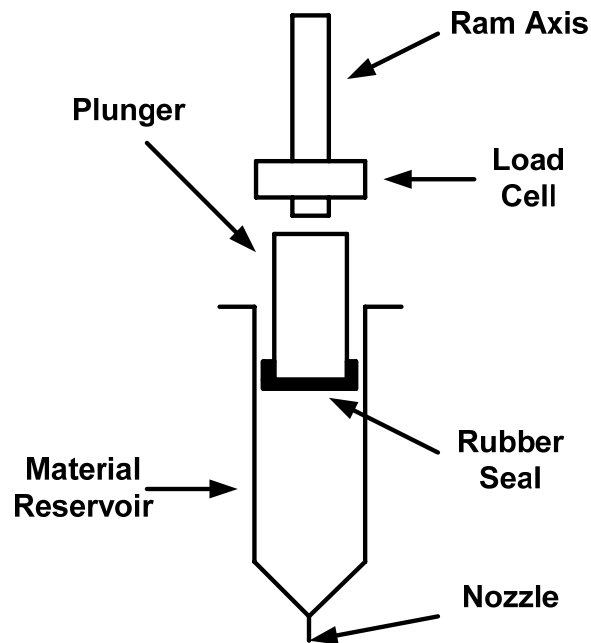
**Figure 1: Schematic showing the location of the die land for an extrusion nozzle.**



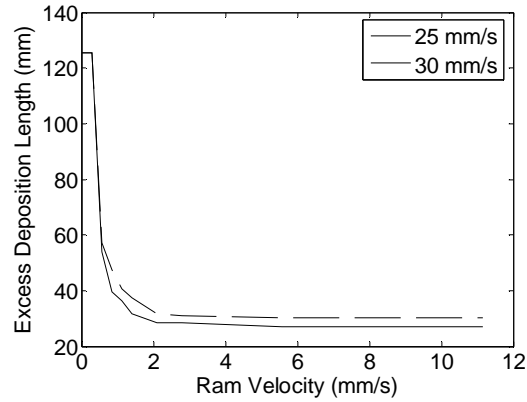
**Figure 2: Photograph of the ABEF system used in this study.**

## STOPPING MATERIAL FLOW TESTING

Experiments were conducted to test the feasibility of stopping material deposition on demand by retreating the ram extruder. The schematic of original extrusion mechanism utilizing a plastic syringe and hypodermic needles is shown in Figure 3. Single lines were deposited at table velocities of 25 mm/s and 30 mm/s with a desired ram force of 308 N at a standoff distance of 150  $\mu\text{m}$ . These parameter values yielded good deposition from previously determined part fabrication parameters using the plastic syringe. Once the table velocity has reached a steady state and traveled a fixed distance (5 mm), the ram was retreated at a given velocity ranging from 0-10 mm/s. After the ram is retreated the amount of excess material deposited was measured from the desired stopping location. Figure 4 shows the deposition length, for table velocities of 25 mm/s and 30 mm/s. The difference in the excess deposition length between the 25 and 30 mm/s tests is due to the fact that a constant amount of material is extruded over the same time period, but it is deposited over a different length. Examining Figure 4 it can be seen that the extrusion is not able to stop on demand no matter what the ram retreating velocity is. This is due to the fact that the ram extruder is not directly connected to the plunger (see Figure 3). When the ram retreats it does not directly stop the pressure being applied to the fluid within the plastic syringe, but only detaches itself from the plunger. In order for the material to stop extruding, equilibrium must be reached between the applied extrusion force and the shear stress created by fluid motion. This occurs when enough material has been released, decreasing the amount of material present within the same volume. From these results the ram retreating method of stopping the extrusion process was not a viable solution with the plastic syringe extrusion mechanism design.



**Figure 3: Schematic of original extrusion mechanism utilizing a plastic syringe and hypodermic needles.**

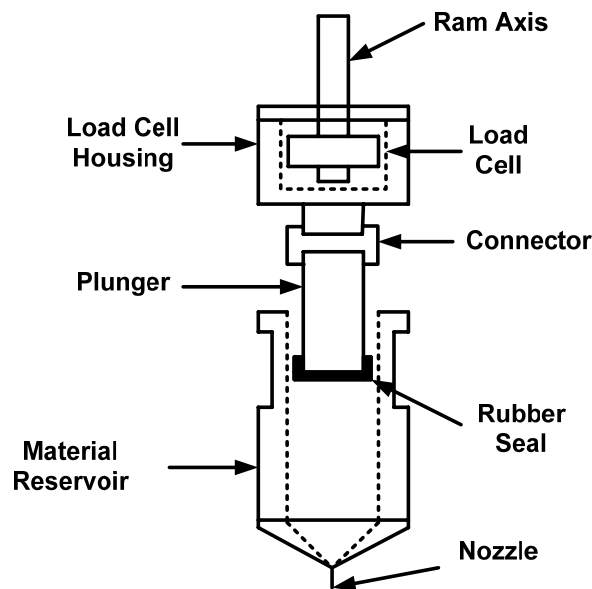


**Figure 4: Ram retreating test data for the original extrusion mechanism.**

### NEW EXTRUSION MECHANISM DESIGN

In order to improve the process and add the ability to stop the extrusion on demand a new extrusion mechanism was designed on the basis that to stop the extrusion, it would be necessary to retract not only the ram axis but the plunger also. To accomplish this a housing was placed around the load cell that would connect to the plunger directly. With the load cell enclosed as the ram axis is retreated the load cell touches the housing, thereby raising the plunger. A schematic of the new design is shown in Figure 5.

To further improve the system it was decided to replace the plastic syringe and hypodermic needles with a two-piece metal reservoir. The metal reservoir reduces variability in the internal pressure due to expansion of the plastic syringe. The mechanical design is such that different dies can be used for the desired material deposition height and width.



**Figure 5: Schematic of new extrusion mechanism utilizing an interlocking system made of 304 stainless steel.**

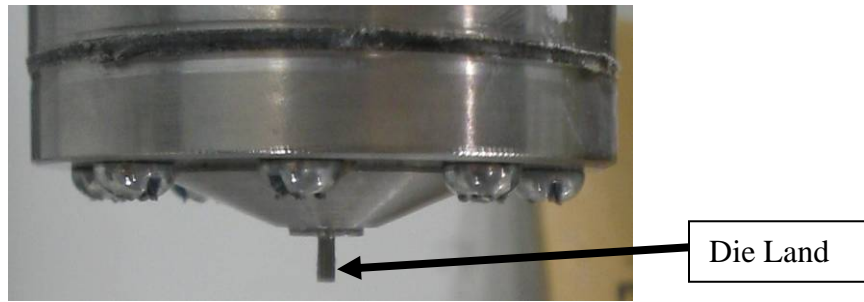
To determine a suitable design for a new material reservoir the resulting stress on the reservoir was calculated using

$$\sigma = \frac{F}{A} = \frac{F}{\pi(R_o^2 - R_i^2)} \quad (1)$$

where  $\sigma$  is the stress on the system,  $F$  is the extrusion force,  $A$  is the equivalent area, and  $R_o$  and  $R_i$  are the outer and inner radii of the reservoir respectively. Substituting in the values of  $F = 4.4 \text{ kN}$  (maximum extrusion force),  $R_o = 15.2 \text{ mm}$ , and  $R_i = 12.7 \text{ mm}$  we arrive at the maximum stress of  $\sigma = 63 \text{ MPa}$ . In order to guarantee the reservoir would not fail under this stress the wall thickness was increased to  $12.5 \text{ mm}$  and 304 stainless steel was chosen for its yield stress of  $500 \text{ MPa}$ . With these changes made, a factor of safety of 7.9 is present in the design. Figure 6 shows the newly designed material reservoir as implemented into the ABEF system. For the design of the nozzle, a diameter of  $0.5 \text{ mm}$  was chosen for a similar diameter ( $580 \mu\text{m}$ ) to that previously used with the plastic syringe and a die land length of  $5 \text{ mm}$  was chosen, which was a 50% reduction in length from the previously used hypodermic needles. Figure 7 shows a close-up view of the die land of the new reservoir. Tests proved that the new design worked without failure.

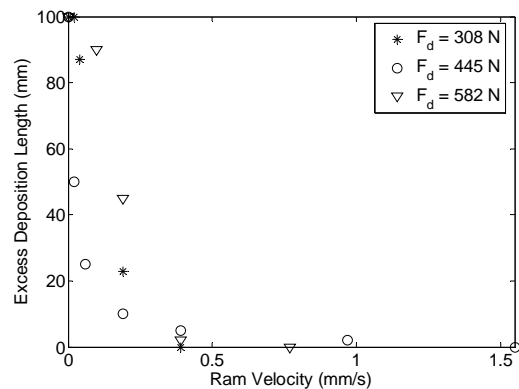


**Figure 6: 304 stainless steel extrusion mechanism design implemented in the ABEF system.**



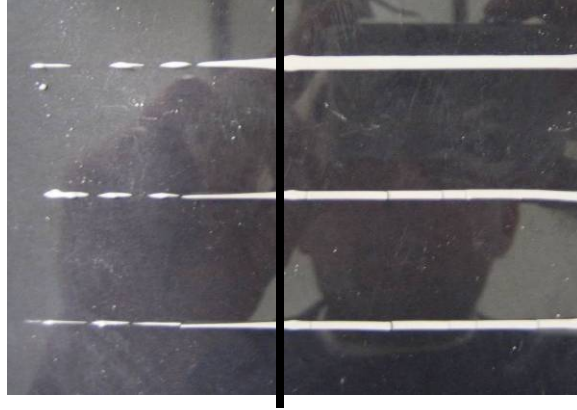
**Figure 7: Picture of 304 stainless steel die.**

The ram retreating experiments were repeated using the new material reservoir. Three different extrusion forces were used: 308, 445, and 582 *N*. These extrusion forces were chosen due to good deposition results during part fabrication using the original extrusion mechanism. Figure 8 shows the results from the deposition tests using the new material reservoir. The extrusion was able to be stopped on demand using the retreating method at all three extrusion forces. Figures 9-12 show pictures of the line deposition tests at ram velocities of 0.5, 1, 1.5, and 2 *mm/s*, respectively.

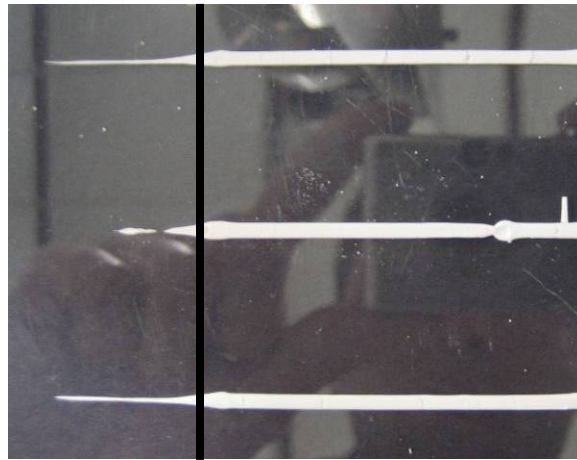


**Figure 8: Ram retreating results with use of the new extrusion mechanism.**

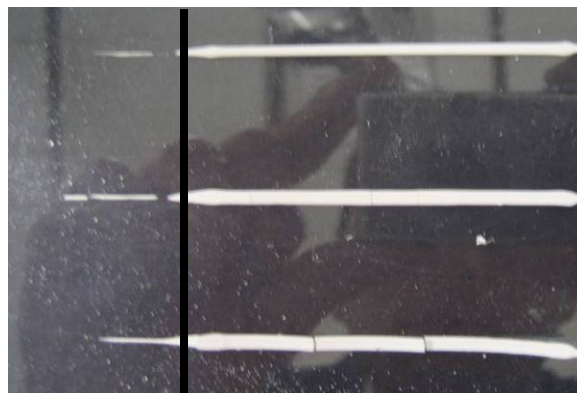




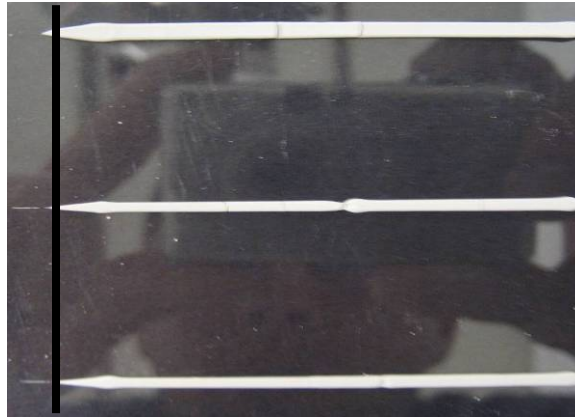
**Figure 9:** Three runs of line depositions with ram retreating velocity of  $0.5 \text{ mm/s}$  and  $F_d = 308 \text{ N}$ . Black line indicates desired stopping location.



**Figure 10:** Three runs of line depositions with ram retreating velocity of  $1 \text{ mm/s}$  and  $F_d = 308 \text{ N}$ . Black line indicates desired stopping location.

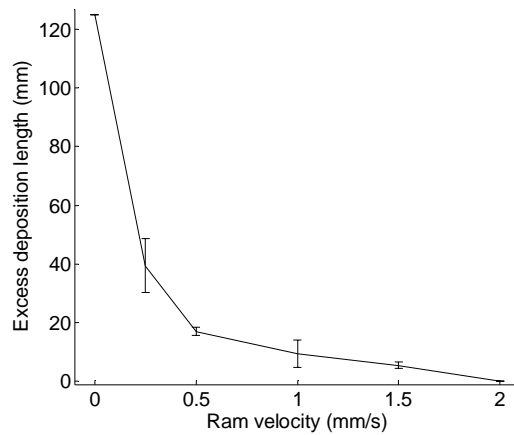


**Figure 11:** Three runs of line depositions with ram retreating velocity of  $1.5 \text{ mm/s}$  and  $F_d = 308 \text{ N}$ . Black line indicates desired stopping location.



**Figure 12: Three runs of line depositions with ram retreating velocity of 2 mm/s and  $F_d = 308\text{ N}$ . Black line indicates desired stopping location.**

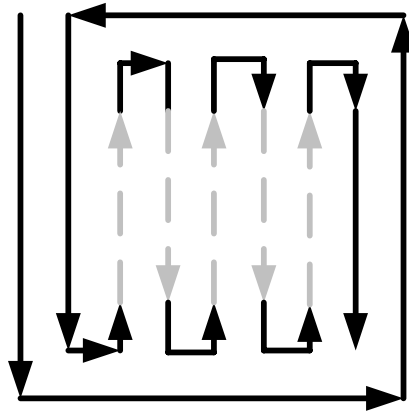
In order to determine the repeatability of stopping the extrusion process, the line deposition tests were conducted 10 times for each of several ram retreating velocities between 0-2 mm/s and an extrusion force of 308 N. The process was determined to be fairly repeatable, with smaller variations in excess deposition at higher ram velocities and larger variations at lower ram velocities. Figure 13 shows the results from the repeatability tests.



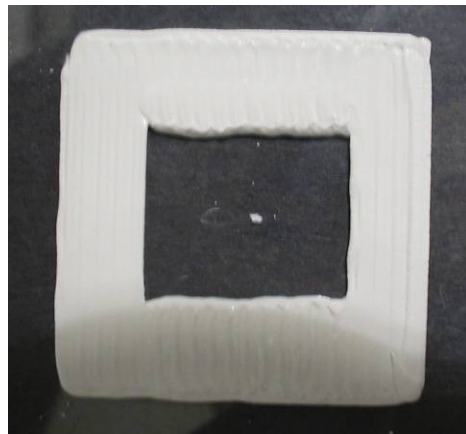
**Figure 13: Chart showing retreating test data with error bars representing one standard deviation.**

### Part Fabrication

With the ability to stop the extrusion on demand, the next objective was to test this technique for part fabrication. The first part used for testing was a single layer square with an interior rectangular hole. In order to fabricate this part a motion path as shown in Figure 14 was used. First the exterior contour was deposited for a smooth outer surface. Next the interior was deposited in a rastering motion. The black lines in Figure 14 indicate material deposition and the gray lines indicate no deposition. Figure 15 shows the fabricated part.



**Figure 14: 2D test part motion path. Dashed lines indicate no deposition.**

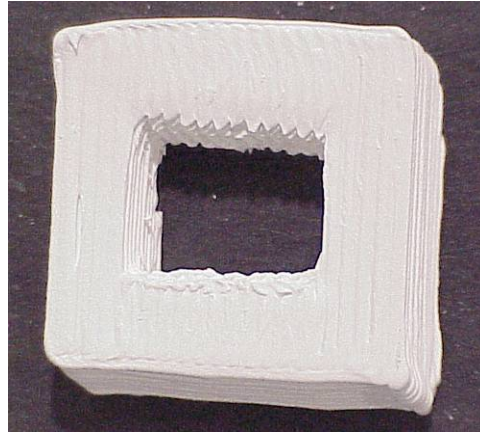


**Figure 15: Top view of deposited material from motion path in Figure 14.**

With the success of being able to fabricate 2-dimensional shapes by starting and stopping the extrusion on demand, the described technique was implemented towards the fabrication of 3-dimensional parts. The part fabricated in this test was a slanted block with an interior hole. The same motion path is followed as for the 2D part, but between layers a 200  $\mu\text{m}$  shift is made in both the X and Y directions. Figures 16 and 17 show the side and top views of the part respectively.



**Figure 16: Side view for slanted part fabricated from starting/stopping extrusion.**



**Figure 17: Top view of part in Figure 16.**

### **Summary**

A new extrusion mechanism for the process of aqueous-based extrusion fabrication of ceramics on demand has been designed and implemented, providing an extrusion capability that cannot be done with the use of a plastic syringe for extrusion. Deposition tests were conducted to examine the relationship between ram retreating velocity and excess material deposition. The implementation of the new extrusion mechanism allows for the extrusion to start and stop as programmed. The ability to start and stop the extrusion on demand allows for more complex 3D part fabrication including sharp corners and internal features. Several parts were fabricated using the new material reservoir to demonstrate the developed capability.

### **Acknowledgement**

This work was supported by the Air Force Research Laboratory under Contract FA8650-04-C-5704.

### **References**

1. Huang, T., Mason, M., Hilmas, G.E., and Leu, M.C., 2006, "Freeze-Form Extrusion Fabrication of Ceramic Parts," *International Journal of Virtual and Physical Prototyping*, Vol. 1, No. 2, pp. 93–100.

2. Huang, T., Mason, M., Hilmas, G.E., and Leu, M.C., 2006, "Freeze-Form Extrusion Fabrication of Ultra High Temperature Ceramics," *Materials Science and Technology Conference*, Cincinnati Ohio, October 15-19.
3. Mason, M., Huang, T., Landers, R., Leu, M.C., and Hilmas, G., 2006, "Freeze-Form Extrusion of High Solids Loading Ceramic Slurries, Part I: Extrusion Process Modeling," *Proceedings of Solid Freeform Fabrication Symposium*, Austin, Texas, August 14-16.
4. Jafari, M. A., Han, W., Mohammadi, F., Safari, A., Danforth, S. C., and Langrana, N., 2000, "A Novel System for Fused Deposition of Advanced Multiple Ceramics," *Rapid Prototyping Journal*, Vol. 6, No. 3, pp. 161-174.
5. Qui, D., Langrana, N.A., Danforth, S.C., Safari, A., Jafari, M., 2001, "Intelligent Toolpath Extrusion-Based LM Process," *Rapid Prototyping Journal*, Vol. 7, No. 1, pp. 18-23.
6. Mukhopadhyay, S., Bristow, D.A., and Ferreira, P.M., 2005, "Multi-Material Micro-scale Robotic Deposition," *Proceedings of the 2005 IERC*, Atlanta, Georgia, May 14-18.