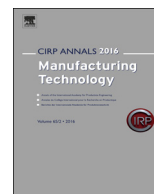




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## CIRP Annals - Manufacturing Technology

journal homepage: <http://ees.elsevier.com/cirp/default.asp>

# Fabricating ceramic components with water dissolvable support structures by the Ceramic On-Demand Extrusion process

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## ARTICLE INFO

## Keywords:

Ceramic  
Extrusion  
Additive manufacturing

## ABSTRACT

This paper describes a further development of the novel Ceramic On-Demand Extrusion (CODE) process, with focus on fabricating ceramic components that have external/internal features and cannot be fabricated without the use of support structures. The minimum angle of a wedge-shaped part that can be fabricated using Al<sub>2</sub>O<sub>3</sub> (alumina) paste without a support structure is first determined. An inorganic sacrificial material, CaCO<sub>3</sub> (calcium carbonate), is then identified for building support structures. After fabrication the green part is dried and then sintered. During sintering, the main material densifies, while the sacrificial material decomposes and is then dissolved in water or acid. Sample parts are fabricated and evaluated.

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## 1. Introduction

Fabricating three-dimensional components from ceramic materials is often expensive and time-consuming, and part geometrical complexity is limited when using conventional processing techniques such as machining. Additive manufacturing (AM) can be applied to reduce the fabrication time and cost, especially for small runs and for components with complex geometries.

Several AM techniques have been developed for ceramics and glasses, including binder jetting [1], material extrusion [2–6], vat photopolymerization [7], powder bed fusion [8,9], directed energy deposition [10], etc. Ceramic On-Demand Extrusion (CODE) is a recently developed paste extrusion based AM process, which produces ceramic components with near theoretical (>98%) density after sintering. It deposits high solids loading (>50 vol%) ceramic pastes onto a substrate layer by layer at room temperature. The printed parts are dried in a controlled environment with appropriate humidity to produce crack-free green bodies, after which the green bodies are sintered to produce highly dense ceramic parts [11,12].

The present paper describes the development of a method for fabricating ceramic parts with complex geometries or features that require the use of sacrificial support structures in the CODE process. A novel CODE fabrication machine was developed and configured to work with two extruders capable of depositing two different materials. Concurrent deposition of the sacrificial material enables the CODE process to fabricate ceramic parts that have external/internal features such as overhangs, conformal channels, etc. Unlike the Freeze-form Extrusion Fabrication

process [9], which is another paste extrusion based ceramic AM process that solidifies the paste by freezing, the selection of a suitable sacrificial material in the CODE process is more challenging because the process operates at room temperature. After identifying calcium carbonate (CaCO<sub>3</sub>) as a workable sacrificial material, which decomposes during sintering and can be removed by dissolving in water or acid after sintering, aqueous CaCO<sub>3</sub> paste was prepared as the support material. A multi-step sintering technique was developed for alumina (Al<sub>2</sub>O<sub>3</sub>) parts due to the favorable phase equilibrium between Al<sub>2</sub>O<sub>3</sub> and CaCO<sub>3</sub> within the sintering temperature range. For demonstration purposes, cuboid parts with rectangular through-holes were fabricated to examine part dimensional accuracy. The fabrication of a geometrically complex part with overhangs and tube-shaped features by the CODE process was also successfully demonstrated.

## 2. Process overview and experimental setup

The CODE process extrudes and deposits aqueous pastes onto a substrate to print each layer sequentially. The substrate is placed inside a tank designed to hold a fluid medium (normally oil). Once one layer is deposited completely, the oil is pumped into the tank to surround the part, preventing undesirable water evaporation from the perimeters of the deposited layers. The oil level is maintained just below the topmost layer of the part being fabricated. Infrared radiation is then applied in a direction perpendicular to the top surface to uniformly dry the deposited layer, so that the part being fabricated will be partially dried and stiffened. This enables the printed body to sustain the weight of the subsequently deposited layers without deformation. By repeating the above steps, the part is printed in a layerwise fashion [11–13]. The layered uniform radiation drying, together with the

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prohibition of evaporation from the sides of the part, prevents moisture gradation in the part, thus avoiding part cracking and warping. Once a part is completely printed and removed from the oil tank, the remaining water content is eliminated by bulk drying to obtain a green body. The green body then goes through post processing, including sacrificial material removal, binder burnout and sintering, to obtain a dense ceramic part.

The CODE fabrication system consists of a motion subsystem (gantry); extrusion devices mounted on the gantry and capable of extruding viscous ceramic pastes at controlled flowrates; an oil feeding device to regulate the oil level in the tank; and an infrared radiation heating device capable of moving the infrared source and providing on/off control. Fig. 1 shows the CODE fabrication system and its environment. The details of this system and its subsystems were presented in our previous papers [11,14].

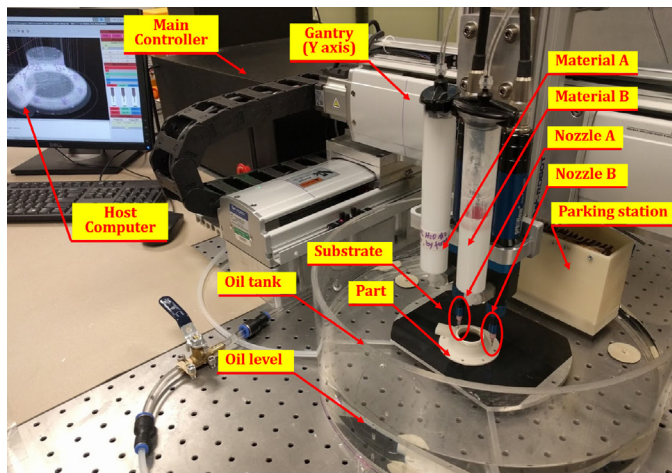


Fig. 1. The CODE fabrication system configured for dual-extruder printing.

### 3. Part fabrication

#### 3.1. Paste preparation

A 55 vol% solids loading  $\text{Al}_2\text{O}_3$  paste was prepared by following a previously developed recipe described in Ref. [13]. The calcium carbonate paste was prepared from  $\text{CaCO}_3$  powder (Sigma-Aldrich, St. Louis, MO, USA). Acetone (Sigma-Aldrich, St. Louis, MO, USA) was the solvent chosen for ball milling to reduce the average particle size. An ammonium polyacrylate, Dolapix CE 64 (Zschimmer & Schwarz, Germany), was used as the dispersant; PEG 400 (poly (ethylene glycol); Sigma-Aldrich, St. Louis, MO, USA) as the lubricant and humectant; and cold water dispersible methocel (methocellulose; Dow Chemical Company, Pevely, MO, USA) as the binder. Both the alumina and calcium carbonate pastes have a viscosity around 200 Pa s.

#### 3.2. Determination of maximum overhang wedge angle

An experiment was conducted to find out the maximum angle of overhanging features that the CODE process is capable of printing without using support structures. In this experiment, a 55 vol% solids loading  $\text{Al}_2\text{O}_3$  paste was used. Several wedge-shaped parts with different overhang angles were identified as test parts. The process parameters in this experiment are listed in Table 1, whose values were determined and successfully implemented in previous studies [13]. As shown in Fig. 2, the overhang did not start to collapse until the test parts with a  $60^\circ$  overhang were attempted. Hence, for the 55 vol% solids loading alumina paste, the maximum overhang angle for the CODE process was identified to be between  $55^\circ$  and  $60^\circ$ . Note that the limit of overhang angle may vary for different part shape and process parameters. The wedge-shaped structure, compared to thin-wall and cylindrical structures, better represents the typical overhanging structure in AM processes. Hence, it was selected as a benchmark in this test.

Table 1

Process parameters for wedge-shaped test parts.

Parameter	Value
Nozzle diameter	610 $\mu\text{m}$
Layer thickness	400 $\mu\text{m}$
Line spacing	600 $\mu\text{m}$
Overall printing speed	20 mm/s
Number of perimeters	2
Drying time	10 s per layer

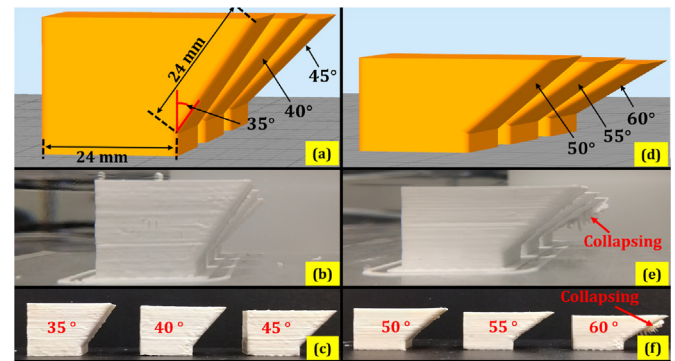


Fig. 2. Wedge-shaped test parts: (a) and (d) CAD models, (b) and (e) as-printed parts surrounded by oil, (c) and (f) as-sintered parts.

#### 3.3. Printing of sample parts

In this experiment, some sample parts which require support structures were fabricated to demonstrate the CODE system's capability of fabricating 3D components, evaluate the dimensional accuracy of fabricated parts, and validate the feasibility of using  $\text{CaCO}_3$  paste as a support material.

##### • Simple-geometry sample part

A cuboid with rectangular through-holes was chosen as a sample geometry to evaluate the dimensional accuracy of parts fabricated by the CODE process. The CAD model and the printing process of the cuboid are shown in Fig. 3. Five cuboid sample parts were fabricated using the process parameters in Table 1. The main structure infill density was 100% (i.e. solid) and the support structure was printed with 50% density.

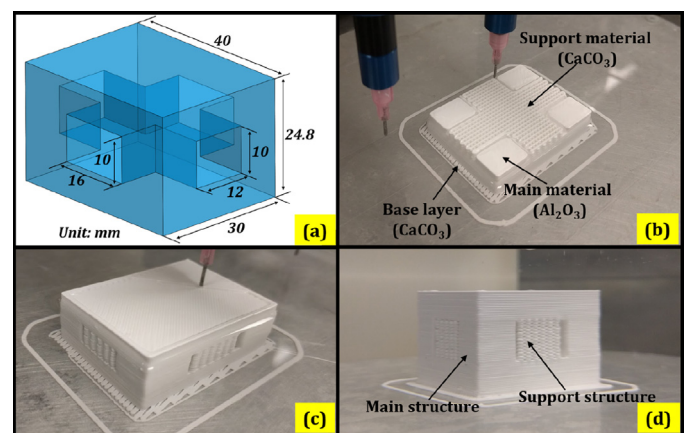


Fig. 3. Cuboid part with rectangular holes: (a) CAD model, (b) and (c) part being printed, (d) part printed completely and surrounded by oil.

##### • Complex-geometry sample part

In addition to the sample cuboids, a turbine-blower housing with a relatively complex geometry (Fig. 4) was printed to further validate the capability of the CODE process and the feasibility of the  $\text{CaCO}_3$  support material. The same process parameters used for the cuboid samples were used in this experiment.

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