Ceramics International xxx (xxxx) xxx–xxx

Contents lists available at ScienceDirect

Ceramics International

journal homepage: www.elsevier.com/locate/ceramint

Characterization of zirconia specimens fabricated by ceramic on-demand extrusion

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ARTICLE INFO

Keywords:
Additive manufacturing
ZrO_2
Mechanical property
Surface roughness

ABSTRACT

The Ceramic On-Demand Extrusion (CODE) process is a novel additive manufacturing method for fabricating dense (~99% of theoretical density) ceramic components from aqueous, high solids loading pastes (> 50 vol%). In this study, 3 mol% Y_2O_3 stabilized zirconia (3YSZ) specimens were fabricated using the CODE process. The specimens were then dried in a humidity-controlled environmental chamber and afterwards sintered under atmospheric conditions. Mechanical properties of the sintered specimens were examined using ASTM standard test techniques, including density, Young's modulus, flexural strength, Weibull modulus, fracture toughness, and Vickers hardness. The microstructure was analyzed and grain size measured using scanning electron microscopy. The results were compared with those from Direct Inkjet Printing, Selective Laser Sintering, Lithography-based Ceramic Manufacturing (LCM), and other extrusion-based processes, and indicated that zirconia specimens produced by CODE exhibit superior mechanical properties among the additive manufacturing processes. Several sample components were produced to demonstrate CODE's capability for fabricating geometrically complex ceramic components. The surface roughness of these components was also examined.

1. Introduction

Zirconia ceramics, especially 3Y-TZP (3 mol% Y_2O_3 stabilized tetragonal zirconia polycrystal), are important structural ceramic materials due to their superior mechanical properties resulting from the transformation toughening mechanism [1]. Additive manufacturing provides the capability of producing components with high geometrical complexity. However, most ceramic additive manufacturing processes exhibit less than satisfactory mechanical properties due to residual porosity in the final products, a result of additive manufacturing processes, and the flaw-sensitive nature of ceramic materials. Thus, pursuing mechanical properties equal to those of more traditional processing methods is a challenge for ceramic additive manufacturing.

The Ceramic On-Demand Extrusion (CODE) technique is a novel, extrusion-based, additive manufacturing (AM) process, which produces dense (~99% of theoretical density) ceramic components after sintering. It deposits high solids loading (> 50 vol%) aqueous ceramic pastes onto a substrate, layer-by-layer, at room temperature. Each deposited layer is solidified by uniform infrared radiation drying from the top surface. At the same time, undesirable water evaporation from the sides of the part is prohibited by surrounding the part with liquid [2,3]. This layered uniform radiation drying approach minimizes the water content gradient in the fabricated part and thus enables the CODE process to produce crack-free ceramic parts. The progressive cavity pump based extruder utilized in CODE guarantees a precise Extrusion On-Demand (EOD) control with a consistent deposition flowrate to avoid pores in the part [4], which further improves the density of the as-printed part.

In the work described in the present paper, the CODE process was used to fabricate 3YSZ specimens, and the mechanical properties and microstructure of these specimens were evaluated and compared with those fabricated by other processes. For demonstration, several geometrically complex parts made of 3Y-TZP were fabricated using CODE and their surface finish was examined.

2. Experimental procedure

2.1. Paste preparation

The aqueous zirconia paste was made of a commercially available

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https://doi.org/10.1016/j.ceramint.2018.04.008
Received 7 March 2018; Received in revised form 31 March 2018; Accepted 2 April 2018

3 mol% yttria-stabilized zirconia (3YSZ) powder (TZ-3Y-E, Tosoh USA, Inc., Grove City, OH, USA), distilled water, dispersant (Dolapix CE 64, Zschimmer & Schwarz GmbH, Lahnstein, Germany), and ammonium hydroxide solution (221228, Sigma Aldrich, St. Louis, MO, USA) for pH adjustment. The zirconia powder had an average particle size of 40 nm and a surface area of 16 m^2 /g, according to the manufacturer.

Batches of ceramic paste were produced at approximately 50 vol% solids loading by dispersing 3YSZ powder in distilled water using 5 wt% dispersant. Initially, the pH of the distilled water was adjusted using the ammonium hydroxide solution until an alkaline pH ≈ 9–10 was achieved, as measured by a pH meter (HI 2210, Hannah Instruments, Woonsocket, RI, USA). The 3YSZ powder was then added slowly and mixed. All mixing was performed using a whip mixer (Model F, Whip Mix, Louisville, KY, USA) while pulling a mild vacuum (~20 kPa) during discrete mixing steps to aid in deaeration, until all the powder was added. The paste was then stirred for an additional five minutes under vacuum to homogenize the paste.

2.2. Part building process

The zirconia paste was extruded at controlled flowrates through a circular nozzle. While the nozzle moved under the control of G&M codes, the extruded material was deposited on a substrate located in a tank designed to hold a fluid medium. Once the deposition of each layer was completed, oil was pumped into the tank surrounding the layer to prevent undesirable water evaporation from the sides of the deposited layers. A mineral oil (Florasense Lamp Oil, MVP Group International Inc., Charleston, SC, USA) was chosen as the fluid surrounding the part to preclude interaction between the fluid and the aqueous paste. The level of oil was controlled so that it was maintained at a level typically 0.4 mm (thickness of one layer) below the top surface of the part being fabricated. Infrared radiation was then applied to uniformly dry the deposited layer from its top so that the part being fabricated would maintain its shape while subsequent layers were deposited. By repeating the above steps, the component was fabricated layer-by-layer. A schematic of the process is displayed in Fig. 1. The layered uniform radiation drying, together with the prohibition of undesirable evaporation from the sides of the part, enabled rapid solidification of each layer without causing moisture gradients in the part, thus preventing part cracking and warpage. The remaining water content and oil on the part surface was eliminated through bulk drying during post-processing.

In this study, 24 beams with dimensions of 6 mm × 25 mm × 4 mm (width × length × height, based on a CAD model), and 5 blocks with dimensions of 53.2 mm × 53.2mm × 6.4 mm (width × length × height, based on a CAD model) were printed for property evaluation.

2.3. Post processing

Once the parts were built and removed from the tank, the remaining water content in the parts, and the oil on the surface of the parts, were eliminated by bulk-drying to obtain “green” parts. The bulk-drying was performed in an environmental chamber where the relative humidity and temperature were controlled to 75% and 25 °C, respectively, for 20 h. The high humidity in the chamber slowed down the drying rate to avoid part warpage and crack formation. The green parts were then sintered in an electric furnace (DT-29-RSA, Deltech, Denver, CO) under atmospheric pressure to obtain the final parts.

In order to determine an appropriate sintering temperature and time, a sintering study was performed on the zirconia beams. The 24 “green” beams were divided into 8 groups and sintered under 8 different sintering conditions. The 8 groups of sintered specimens were then tested to compare their density, hardness, and fracture toughness. The best sintering condition among the 8 groups was determined through comparison of these properties. The 5 “green” zirconia block specimens were then sintered using these selected conditions.

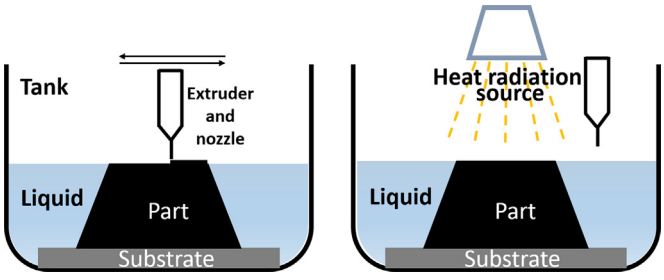

Fig. 1. Schematic of the part building process of CODE.

2.4. Characterization

The density of sintered specimens was determined by Archimedes’ method [5]. The dry mass of each specimen was measured first. Then, the specimens were saturated by submerging in distilled water and placing them under vacuum for 12 h. The saturated and suspended masses were then recorded to calculate the bulk density. This value was divided by the theoretical density (T.D.) of 3Y-TZP (6.056 g/cm^3 [6,7]) to obtain the average relative density of the specimen.

Vickers hardness was measured according to ASTM C1327 [8] using a microhardness tester (V-100-V2, LECO, Saint Joseph, MI, USA). The applied force was 98.07 N for 10 s. The test surfaces of specimens were polished using successively finer diamond abrasives down to 0.25 μm prior to indentation.

For the 24 sintered beams, fracture toughness was estimated from the indentation test using Anstis’ method [9]. For the blocks sintered at the selected final sintering condition, fracture toughness was determined by testing Chevron-Notched (CN) beams in four-point bending using a fully articulating test fixture for configuration A (L = 50 mm, B = 3 mm, W = 4 mm, and a_0 = 0.8 mm) according to ASTM C1421 [10]. Test bars were cut from the block specimens and ground to standard size using an automated surface grinder (Chevalier, FSG-3A818, Santa Fe Springs, CA, USA). A dicing saw (Accu-cut 5200, Aremco Products, Ossining, NY, USA) with a 0.15 mm-thick diamond wafering blade was then used to machine the chevron notches on each test bar. An instrumented load frame (Instron 5881, Instron Corporation, Norwood, MA, USA) was used to test the CN beams with a crosshead velocity of 0.2 mm/min. The dimensions of chevron notches were then measured using an optical microscope (KH-3000, Hirox, Hackensack, NJ, USA).

Flexural strength was measured by the four-point bending method according to ASTM C1161 [11] using an instrumented load frame (5881, Instron Corporation, Norwood, MA, USA) with a crosshead velocity of 0.2 mm/min. Both A-size (2mm × 1.5mm × 25mm) and B-size (4mm × 3mm × 45mm) beam specimens were prepared and tested. From the 5 sintered blocks, 30 A-size specimens and 30 B-size specimens were cut. All four surfaces of each specimen were ground using a 600-grit diamond wheel, and then manually chamfered using a 1200-grit diamond grinding disk. Young’s modulus was determined from Euler-Bernoulli beam theory [12] using the readings of the deflectometer (a linear variable differential transformer) on the load frame which measured the deflection at the center of the test beam during the bending test (see [13] for detailed calculation procedure).

Microstructural analysis was performed using scanning electron microscopy (SEM) (Helios Nanolab 600, FEI, OR, USA). Prior to SEM imaging, the specimen was first polished down to a 0.25-micron finish using successively finer diamond abrasive slurries, then thermally etched at 1350°C for 0.5 h to reveal the grain boundaries. The average grain size was measured by the linear intercept length method [14] in ImageJ [15], an open-source image processing software.