Characterization of ceramic components fabricated using binder jetting additive manufacturing technology

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Abstract

Binder jetting additive manufacturing is an emerging technology with capability of processing a wide range of commercial materials, including metals and ceramics (316 SS, 420 SS, Inconel 625, Iron, Silica). In this project, aluminum oxide (Al_2O_3) powder was used for part fabrication. Various build parameters (e.g. layer thickness, saturation, particle size) were modified and different sintering profiles were investigated to achieve nearly full-density parts (~96%). The material's microstructure and physical properties were characterized. Full XRD, compression testing, and dielectric testing were conducted on all parts. Sintered alumina parts were achieved with an average compressive strength of 131.86MPa (16 hours sintering profile) and a dielectric constant of 9.47 to 5.65 for a frequency range of 20Hz to 1MHz. The complexity offered by additive processing aluminum oxide can be extended to the manufacturing of high value energy and environmental components for environmental systems (e.g. filters and membranes) or biomedical implants with integrated reticulated structures for improved osseointegration.

1. Introduction

Fabrication of ceramics in the macro scale is difficult due to their brittle and hard nature, especially when producing complex geometries of fired ceramics. Ceramics, in general, have a wide range of applications ranging from biomedical implants to engine components in the aerospace and automotive industry (Doore and Hubner 1984). In this research, alumina was selected for fabrication because of its wide applicability in industries such as electronic, biomedical, and aerospace. Alumina is a high-temperature ceramic that provides stable electrical properties and mechanical robustness (Tan, et al. 2014), it is also a structural ceramic due to its wear resistant properties and its ability to withstand severe mechanical stresses in corrosive and thermal environments (Medvedovski, Alumina-mullite ceramics for structural applications 2006). In addition, alumina is catalogued as a versatile ceramic due to the wide variety of applications as a dielectric material, optical (when transparent and used in transparent envelopes in street lamps), biomedical (hip implants) and thermal (packages for integrated circuits). Some of its main properties are stability at high temperatures, permeability, high surface area and thermal shock resistance (Biasini, Parasporo and Bellosi 1997). Alumina can be present as α , γ and β phases, being α -alumina present in ambient atmospheres, which has a hexagonal closed packed structure, with a melting point of 2054°C and a hardness equal to 9 on the Mohs scale (Meethong, Sirirot and Srilomsak 2013). Advantages of this ceramic in traditional manufacturing are its low cost and ease of fabrication by slip casting, pressing, and injection molding (Medvedovski, Ballistic performance of armour ceramics: Influence of design and structure. Part I 2010). Machining of alumina ceramics can be expensive due to the level of experience required and material waste that can be generated during the process (Leu, Pattnaik and Hilmas 2010). It is important to note that alumina has a wide range of purity that depends on the amount of elements such as Silicon within the alloy. High purity alumina (>95%) is typically used for high voltage electrical substrates and insulation while low purity alumina (~60-85%) is primarily used for temperature resistance. In this research low purity alumina precursor powder was used to demonstrate the fabrication of alumina using binder jetting additive manufacturing technology.

Additive manufacturing (AM), or the method of creating a three-dimensional object through a layer-by-layer process, has been widely researched for its potential to process ceramics. One method that has been extensively investigated is Fused Deposition of Ceramics (FDC), developed by Rutgers University (Allahverdi, et al. 2001). This process is based on a material extrusion method used to fabricate

polymers. In order to process conventional ceramics, such as SiO_2 , Si_3N_4 , Al_2O_3 , lead zirconium titanate (PZT), the development of organic binders, binder removal techniques, and sintering conditions have been developed (Agarwala, et al. 1996). In a study conducted by Allahverdi *et al.*, alumina structures with photonic bandgap structures were fabricated with the use of wax as support material (Allahverdi, et al. 2001). Laser Engineered Net Shaping (LENS) has been used to fabricate $\alpha\text{-Al}_2\text{O}_3$ in different shapes such as cylinders, cubes and gears. Compressive testing results showed anisotropic properties that could not be altered by heat treatment (Balla, Bose and Bandyopadhyay 2008). By having a mixture of resin and ceramic where a slurry containing alumina powder and a UV curable monomer, among other things, provided the necessary means to fabricate three dimensional parts by stereolithography (Hinczewski, Corbel and Chartier 1998) (Kruth, Leu and Nakagawa 1998). In addition, by adding alumina particles to create colloidal ink, direct ink-jet printing of ceramics was achieved by Lewis in 2002 to provide tailored properties (Lewis 2002). In this research, binder-jetting technology was used as a means for fabrication. Binder jetting technology is a powder bed based technology that selectively deposits a binder layer-by-layer onto a powder bed to create a three-dimensional object. Upon fabrication, a green body is obtained which undergoes a heat treatment cycle that burns out the binder and sinters the powder particles. The goal of this research was to study the feasibility of fabricating ceramic structures that can be used as packages for sensors or electronics exposed to high temperature environments.

2. Materials and methods

2.1 Fabrication

Fabrication was achieved by the binder jetting process using the ExOne M-Lab system (Irwin, PA serial number: 0600H2). The process consists of fabricating three-dimensional parts (previously designed by computer-aided design (CAD) and uploaded in the system as a '.stl' file) from powder precursor material. Powders are bonded together in a layer-by-layer fashion. The ExOne M-Lab printer consists of a two-bed system (as shown in Figure 1); one bed is for the base powder material and the other for part fabrication. Powder layers are spread with a roller while the bed is mechanically moving in the X-direction. Once the powder layer is uniformly spread, the powder bed returns to its original position where binder droplets are selectively deposited through piezoelectric printhead for part fabrication. Once the binder is deposited, the powder bed moves below the heater, where heat is provided for a set amount of time to achieve powder binding. This is considered as the fabrication of one layer (one full cycle), and the process continues until part fabrication is complete.

For this project, a 1.75:1 ratio of powder and binder deposition was used, which means that the equivalence of one layer and three fourths of a layer of powder from the powder bed (feed) are used to fabricate one layer in the build bed to achieve better powder packing. Based on successful results described elsewhere (Gaytan, et al. 2013) fabrication of Al_2O_3 parts was performed using binder saturation of 60% and various layer thicknesses. The layer thickness was determined based on the particle size of the powder that was being used. Three different powders were used for fabrication and three layer thickness parameters were used for each powder. The first layer thickness that was used corresponded to the particle size. Then half the particle size and double the particle size were used as the layer thickness to determine its influence on final sample property. After the select layer thicknesses were used for all the powders, the powders were combined and mixed evenly to achieve a large distribution of particle size, in order to determine if the density could be improved. For the mixed powders, the range of layer thickness depended on using the largest, median, and the smallest used on the previous runs. Based on the amount of empty space that is present due to the packing of the powder during each layer, the binder will replace such space in order to remove any empty space. After fabrication is finished, the building envelope is placed in the oven at 195°C for 2 hours. This step is performed to cure the binder selectively placed in the fabricated part, allowing the separation of the fabricated geometry from the unbound powder. After the binder is cured, the parts are brushed to remove unbound powder and prepared for sintering.

2.2 Powder

Aluminum oxide grinding powder purchased from Ted Pella, Inc. was used for the fabrication process. Three different grits were used in order to determine the effect of particle size on build quality (e.g. density). The three different grits used were 240, 320, and 400. Note that the grit size is related to particle size where 240 grit is 53 microns, 320 grit is 45 microns, and 400 grit to 30 microns.

2.3 Sintering Profile

Two different schemes of sintering time and temperature were used in order to determine how the shrinkage and density would change. The furnace used for sintering was a Rapid Temp Model 1710 FL Laboratory Box Furnace by CM Furnace Inc. The highest temperature that was reached for both sintering profiles was kept consistent at 1600°C. The heating rate that was used was 10°C per minute, and parts were sintered in atmospheric pressure with no other gas being introduced during the process. The part was sintered at two different dwell durations: 2 hours and 16 hours.

2.4 Characterization

Characterization of shrinkage was defined by the change in part dimensions caused by the sintering. That is, by sintering voided space will be filled by surrounding particles that diffusion and bond with one another, which will reduce the overall geometry of an object. The two different sintering profiles were reviewed experimentally to compare the shrinkage percentage in X, Y and Z dimensions. The original dimensions of the samples were set by the CAD file to be 20mm, 25mm, 10mm as seen in Figure 2. Green bodies were measured using caliper, and re-measured after sintering (Figure 3).

Parts selected for characterization with the use of scanning electron microscope were sputter-coated with gold and palladium (Gatan Precision Etching Coating System Model 682) for 1 minute, at a current of 300 mA, and vacuum of 10^{-6} Torr. The morphology and sintering of particles in the specimens were examined using a scanning electron microscope (Hitachi S-4800: Field Emission SEM, Hitachi High Technologies America Inc., Dallas, Texas). A Bruker X-ray diffractometer was used to confirm the phases present in sintered parts and un-sintered powder. X-ray diffraction is based on elastic scattering, which generates intensity peaks characteristic of orientation and phases present in the analyzed sample (Cullity 1976). The slit size on the controller side was one of 8 mm, and of 0.6 mm on the tube side. The scan range (2 θ values) started at 20° and ended at 90°. The scan was performed in increment of 0.05° at a speed of 0.05°/min to obtain very distinct intensity peaks and simplify phase and orientation detection.

3. Results

3.1 Density

The apparent density was calculated using the Archimedes' method. Here, the product of the average air and the density of water is computed and divided by the difference of the average air and average water measurements. The theoretical density of the alumina used to compare with the fabricated parts was 3.95 g/cm³. Relative density was determined to quantify the amount of sintering that was done by each sintering profile. Note that sintering and density are related to the phenomena during the sintering process that causes powder particles to diffuse together, which causes voids to be filled by bulk material, thus ideally decreasing porosity and increasing density. The graphs that are represented in Figure 4 demonstrate how the density was affected with the change of layer thickness between the two sintering profiles. A typical trend shows that the highest density was achieved by the smallest layer thickness using the mixed powders where the relative density was 96.51%. The lowest density calculated was using the 240 grit powder size, which only averaged 64%.

3.2 Shrinkage

Shrinkage was experienced on all directions. On average, samples shrunk the most on the Z direction, that being 10.27% from green body dimensions. The X direction had 7.92% shrinkage and 8.34% in the Y direction. Standard deviation was calculated for all values, where 4.99 for the X direction, 5.49 in the Y direction, and 6.39 in the Z direction (Table 1). The minimum amount of shrinkage occurred on the samples with the lowest density values. Samples fabricated with 240 grit powder size experienced the lowest amount of shrinkage with the part only shrinking 1.07% in the X direction, 1.73% in the Y direction, and 0.62% in the Z direction. The maximum amount of shrinkage was on samples fabricated with the mixed powder at 45 μ m layer thickness. The shrinkage experience on these samples was 8.75% in the X direction, 10.92% in the Y directions, and 15.37% in the Z direction.

3.3 SEM Analysis

Virgin powder, a green body obtained after the binder jetting process, a part exposed to a 2-hour sintering cycle, and a part exposed to a 16-hour sintering cycle was analyzed under a scanning electron microscope (SEM). According to the images in Figure 5 (a) and (b), there is a minimal difference between virgin powder and a green body after fabrication was completed. It appears that the alumina particles have a random morphology between them, where the packing of particles is completed by the fabrication process. After sintering is completed, the fusion of particle is visible; however pores or voids are still visible. Figure 5 (C) shows how the surface of the 2-hour sintering appears, where (D) shows less visible pores that are consistent with higher density measurements. Higher magnification was conducted on the part exposed to a 16-hour sintering cycle, where neck formation between two particles is visible (Figure 6), which is ideal when powdered materials are fully sintered.

3.4 XRD

The XRD intensity peaks for virgin alumina powder were compared to its sintered counterparts. Figure 7 shows alumina powder peaks obtained for both as-fabricated and sintered parts. The XRD analysis concludes that the powder has a monoclinic crystal structure. This structure is retained when the pure alumina was sintered. Figure 7 identifies the crystallographic orientations of the matched intensity peaks. According to Chang, *et al*, the peaks that were identified for the purchased powder aligns with the alumina peaks that indicate that the peaks appear to be α -Al₂O₃. (Chang, et al. 2009)

3.5 Compression Testing

Compression testing was performed on the mixed powder parts for parts exposed to both sintering profiles. A comparable strength value was obtained from traditionally fabricated alumina. An MTS Landmark servohydraulic test system (Eden Prairie, MN) was used to perform the compression testing. ASTM standard C773-88 was used for the design of the compression test, however the strain rate was altered from 45kN/per to 0.5mm/min. The average area of the test samples was 32.23mm², and length was 11.84mm. Figure 8 are the stress-strain curves for the compression test that were conducted samples both sintered at 2 hours and 16 hours. The samples that were sintered for 16 hours began to break at an average stress of 131.86 MPa, with the highest stress reached being 146.60 MPa before fracture. Samples sintered for 2 hours were averaging 71.79 MPa before the samples began to crack, and the highest stress reached was 77.86 MPa. By sintering the parts for 16 hours, an increase in 82.98% stress was achieved when compared to parts sintered for only 2 hours.

3.6 Hardness

A Hydritron TI 750H Ubi depth-sensing indenter was used to quantify mechanical properties in the nanoscale. Parts fabricated using the mixed powder for both sintering profiles were measured along with a green body. The green body alumina was too weak to provide any type of reading capacities. A range of data measurements was performed on all samples and averaged respectively. Parts exposed to a 2 hour sintering cycle resulted in a Young's modulus of 31.25 GPa (+/- 8.43 GPa) and a hardness of 240 MPa (+/- 66 MPa). Parts exposed to a 16hour sintering cycle resulted in a young's modulus of 54.14 GPa (+/- 14.54 GPa) and a hardness of 1.51 GPa (+/- 0.0967 GPa). Figiel, *et al*, calculated that a pore-free alumina young's modulus value is 379 GPa. When compared with 5% porosity the young's modulus drops to 279GPa (Figiel , Rozmus and Smuk 2011). Hardness may have been affected the rough surface that is created during fabrication. Due to this surface, the indenter may have tested on a valley of peak and have caused values to be lower.

3.6 Dielectric Constant

Dielectric constant measurements for the alumina parts ranged from 9.47 to 5.65 for a frequency range of 20Hz to 1MHz. Dielectric loss stayed constant with all parts averaging about 2.4×10^{-3} . These low dielectric constant (ϵ) and low dielectric loss characteristics can be beneficial for applications in radio-frequency (Alford and Penn 1996). Figure 9 is dielectric property spectra of a sample tested after fabrication.

4. Conclusions

With the use of binder jetting technology, the fabrication of alumina parts was achieved. By altering parameters such as layer thickness, particle size, and sintering profile, high density parts were achieved with 96% relative density. The increase in powder distribution and smaller layer thickness provided the best results. By increasing sintering time, a much desirable density and compression strength was achieved. Further characterization must be conducted to fully understand how alumina fabricated using binder-jetting technology may perform in certain applications. Due to the capacities of alumina and that of additive manufacturing, complex components are achieved for such applications of sensor housing. The ability to fabricate high dense parts could provide the needed protection to sensors in harsh environments. Other structure that may not be achievable with traditional fabrication processes, such as mesh and foam structures, could be achieved thus allowing the opportunity for medical applications.

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Figure 1. Schematic representing the ExOne components used for the fabrication process.

Figure 2. Original CAD file used in order to measure the shrinkage that occurs after sintering.

Figure 3. Green and sintered body after sintering at 1600°C for 16 hours.

Figure 4. Relative density vs. sintering profiles including the three layer thickness used for each powder. (A) 240 grit size powder or 53 μm particle size, (B) 320 powder size or 45 μm particle size, (C) 400 grit size powder or 30 μm particle size, (D) combination of all three powders.

Build Direction	Shrinkage (%)	Standard Deviation
X	7.92	4.99
Y	8.34	5.49
Z	10.27	6.39

Table 1.Average shrinkage occurs on samples after sintering process

Figure 5. SEM images for (A) As-purchased powder (B) Green body fabricated part (C) Part sintered for 2 hours and (D) Part sintered for 16 hours. All parts were mixed powder at 45μm layer thinness.

Figure 6. SEM image of 16 hour sintered part with a high-magnified image of creation of necks between two powder particles.

Figure 7. XRD spectra of pure alumina powder (bottom) and sintered pure alumina component (top). Matching peaks are referenced with the blue symbols

Figure 8. Stress-Strain curves of the alumina compression test. (A) Samples that were sintered for 2 hours and (B) Samples that were sintered at 16 hours.

Figure 9. Dielectric constant and dielectric loss for alumina fabricated by binder jetting technology