3D Printing of Piezoelectric Element for Energy Focusing and Ultrasonic Sensing

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Abstract

Piezoelectric ceramics are currently of considerable interest for their capabilities of converting compressive/tensile stresses to an electric charge, or vice versa. Because ceramics cannot be cast and machined easily, additive manufacturing (AM) processes (3D printing technology) open an effective pathway in geometrical flexibility. However, the piezoelectric properties limit the application of printed ceramics. This work demonstrates that a piezoelectric-composite slurry with BaTiO₃ nanoparticles (100nm) can be 3D printed using Mask-Image-Projection-based Stereolithography (MIP-SL) technology. After a post-process, the density of 5.64g/cm³ was obtained, which corresponds to 93.7% of the density of bulk BaTiO₃ (6.02 g/cm³). The printed

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ceramic exhibits a piezoelectric constant and relative permittivity of 160 pC/N^{-1} and 1350 respectively. An ultrasonic transducer with printing focused piezoelectric element was fabricated to realize the energy focusing and ultrasonic sensing. A 6.28MHz ultrasonic scan was achieved by the transducer and successfully visualized the structure of a porcine eyeball.

Keywords: Additive manufacturing; Piezoelectric materials; Ultrasonic transducer; Ultrasonic imaging

1. Introduction

The applications of piezoelectric ceramics are far reaching, ranging from acoustic imaging to energy harvesting.^[1-6] However, conventional manufacturing processes such as etching and dicing for piezoelectric ceramics fabrication have limited capability of achieving complex geometry and high resolution(X-Y resolution<25 μm). Moreover, mechanical stress caused by the traditional machining processes can result in grain pullout, strength degradation and depolarization of the near surface region, which will led to significant degradation in piezoelectric device performance.^[7]

To enable the fabrication of various functional ceramics, novel additive manufacturing (AM) processes (3D printing technology) need to be developed. Some main AM processes include tape casting, soft molding, transfer/pad printing.^[8-12] However, the resulted geometry in tape casting method would be difficult to control for more complex shape. The molding techniques and pad printing are indirect processes with multiple steps and each steps requires significant

effort. During the past thirty years, direct inkjet based printing, extrusion-based direct-write technique, and light exposure based Stereolithography Apparatus (SLA) have been used in ceramic component fabrication.^[13-23] Many efforts have been made on applying SLA in ceramics component fabrication, due to its advantages in fabricating complex geometry and high resolution (X-Y resolution < 25 μm). Usually, the AM process of fabricating ceramic components involves two main steps: (1) green-part fabrication to define part geometry; and (2) debinding and sintering of green-parts to achieve dense components. However, since the SLA process has limit on the viscosity and photosensitivity of liquid resin, the solid loading of piezoelectric ceramic slurry cannot be high. Consequently, after sintering, the density of sintered part is low.^[24,25] This leads to unsatisfactory piezoelectric properties.

Since, for ceramic particles, the decrease in free energy due to reduction in surface area is the main driving force for the sintering process, nanoparticle-based fabrication to improve the property of sintered ceramic materials has been an active research area in recent years. For SLA, the challenge of using slurry with a high solid loading nanoparticles comes from the reduction of cure depth. According to Jacob's equation:^[26]

$$\ln\left(\frac{E}{E_c}\right) = \frac{D_p}{C_d} \quad (1)$$

Where C_d is the cure depth, E is the energy density of incident light, E_c is the critical energy density, and D_p is the resin sensitivity defined as:

$$\frac{1}{3Q} \quad (2)$$

Where d_{50} is the average particle size; Δn is the refractive index difference between the ceramic particle (n_p) and the liquid resin (n_0 , i.e. $\Delta n^2 = (n_p - n_0)^2$). When the average particle size d_{50} decreases from micro-scale to nanoscale, the resin sensitivity D_p and cure depth C_d decrease dramatically. This brings significant difficulty to the SLA fabrication process, in which each layer needs to be sufficiently over-cured such that it can firmly attach to the previously cured layers.

In order to overcome the problem, we presented a bottom-up projection-based Stereolithography method by integrating a tape casting process for nanoparticle fabrication.^[27] The X-Y resolution is 20 μ m and the Z resolution is 10 μ m.

The most popular piezoelectric materials include Pb-(Mg1/3Nb2/3)O3-PbTiO3 single crystal and Lead zirconate titanate (PZT),^[28-30] due to their high piezoelectric constant, processability, and affordability. However, all these materials contain lead, which has recently been expelled from many commercial applications, especially in clinic diagnoses, owing to concerns regarding its toxicity. Compared with lead-based piezoelectric ceramics, lead-free piezoelectric ceramics, such as Sodium potassium niobate (KNN) and Barium Titanate (BaTiO₃) are competitive for the same types of applications.^[31,32] In this study, we report that BaTiO₃, a widely used lead-free ceramic material, can be three-dimensional (3D) printed into complex 3D geometry with sufficient ferroelectric and piezoelectric properties that are suitable for the application of piezoelectric device such as ultrasonic transducer.

2. Experimental

Surface modification of BaTiO₃

An azeotropic mixture of methylethylketone (66 v/v%, 99%, MEK, Sigma-Aldrich, Saint Louis, MO) and ethanol (34v/v%, 99.5%, Sigma-Aldrich, Saint Louis, MO) with was prepared. The BaTiO₃ powders (25v/v% solid loading, 100nm, Sigma-Aldrich St. Louis, MO) were deagglomerated in the azeotropic mixture with dispersant (Triton x-100, 0.5-0.8wt% on a dry weight basis of ceramic powders) by planetary mill (pulverisette 5, FRITSCH Idar-Oberstein, Germany) using stainless steel balls for 12 hours. The rotation speed is 200 rpm. The mixture is then dried at 50°C for 12 h. After the evaporation of the solvent in the dispersion, dry BTO powders with dispersant adsorbed on to their surface can be obtained.

Green-Parts Fabrication

The 70wt% deagglomerated BaTiO₃ powder (100nm) and 30wt% photocurable resin SI500, (EnvisionTec Inc., Ferndale, MI) were mixed by ball milling for 1h. Figure. 1a shows the illustration of the MIP-SL system. The slurry (0.045ml) is dispensed onto the film collector and spread out to a thin layer (50µm) by a doctor blade when the film collector slides towards the left. After that, a Computer-Aided Design (CAD) model (Fig. 1c) is sliced into two-dimensional (2D) images, each of which is then projected onto the bottom of the film collector through a Digital Micromirror Device (DMD) (Fig. 1b). Figure 1.d shows the interface to control the motor and projector. The next procedure involves the photopolymerization of photocurable resin in the slurry that produces cross-linked matrix under the projected visible light, forming a strong

bond between piezoelectric particles and polymer network. Once a single layer is cured, the platform moves up to separate it from the film collector surface that is coated with PDMS. Extra slurry is then added by the dispenser and delivered by the film collector. The next layer is fabricated in the same manner.^[22] Figure. 1e shows the green-part fabricated by the MIP-SL process.

Debinding and Sintering Study of BaTiO₃ Green-Parts

To achieve dense piezoelectric ceramics, the post-processing steps including organic binder removal and high-temperature sintering (Fig. 2a). To avoid the deformation of samples, the fabricated green parts were debinded in a muffle furnace with Argon under a temperature of 600°C for 3 hours (supplementary Fig. S1). After a debinding process, the polymer in the composite is removed. After the furnace cooling, the debinded part was put in a regular muffle furnace with air at 1330°C for 4 to 6 hours. At a higher temperature, the BaTiO₃ particles undergo the initial-stage sintering, which leads to neck formation at particle-particle connection. As the temperature increases, more particles connect with each other through diffusion process and the growth of grain size as well. This process is associated with considerable shrinkage and consolidation of the powdery structure; at the same time, the porosity of the material is significantly reduced. Fig. 2c and 2b show the Scanning Electron Microscope (SEM) images of the debinded and sintered sample. For the debinded sample, as observed, the grain size is in nanoscale. Pores are irregular and interconnected at various dimensional planes. The sintered

samples show obvious grain growth and much less pores, which indicates a high density of the sintered part. It is caused by the nanoparticles (100nm) with higher free energy and larger driving force during sintering.

To demonstrate the developed piezoelectric ceramics fabrication process, a 3D printed segment annular array (3D-SAA) (Fig. 3b) and a printing focused concaved-shape piezoelectric element (PF-CPE) (Fig. 3e) were fabricated. Fig. 3a and 3c shows the optical image of a 3D-SAA with 64 pillars (1mm height) in a fan shape that are built on a 1mm base. In the traditional machining methods, the shape of pillars are usually designed in square or rectangular shape, the 3D-SAA demonstrate that our method can offer more flexibility to the design of complex geometry for a novel composite structure and piezoelectric array. Fig. 3d and Fig. 3f shows the detailed structure of PF-CPE under microscope. The aperture of working area (the concaved area in the inner ring) is 5mm. The arc length and thickness are 5.2mm and 390 μ m, respectively. The ultrasonic wave can be generated and focused by this structure for the application of ultrasonic sensing, which will be discussed later (refer to Fig. 6).

3. Results and Discussion:

Characterization of Sintered-Parts

In order to characterize the properties of the printed piezoelectric ceramics, a set of cylindrical piezoelectric samples (diameter 10mm, thickness 390 μ m) were fabricated using the same

fabrication process. The poling field is 30kV/cm at 100°C for 30 min. When alternating current is applied on the piezoelectric ceramics, the impedance and phase will change with the frequency. When the impedance drops to a minimum, the phase changes from -90° to 0°. When the impedance rises to a maximum, the phase changes from 0° to -90°. The first thickness resonance is shown in Fig. 4a. The electromechanical coupling factor K_t can be formulated as follows: ^[33]

$$\sqrt{\frac{f_a - f_r}{f_a}} \quad (3)$$

Where f_r is resonant frequency, and f_a is anti-resonant frequency. For our printed ceramic, the f_r and f_a are 6.00MHz and 6.67MHz, respectively (Fig. 4a), and the corresponding K_t is 47.4%.

Moreover, dielectric constant and clamped dielectric constant measured by an impedance analyzer (Agilent 4294A) are 1350 and 1030 respectively. Table 1 compares the density, dielectric constant, piezoelectric constant and loss tangent of our 3D-printed BaTiO₃ with the typical BaTiO₃ and the BaTiO₃ that is fabricated by other additive manufacturing fabrication methods.^[34-37] Density measured by ASTM B962-14 standard of 3D-printed BaTiO₃ is 5.64 g/cm³, which corresponds to 93.7% of the density of bulk BaTiO₃ (6.02 g/cm³). For typical BaTiO₃, the dielectric loss (tanδ) is usually less than 0.1 and the remnant polarizations P_r is larger than 2μC/cm². The low dielectric loss of the printed ceramics (tanδ=0.018) indicates that when working in a piezoelectric device, the energy loss of our sample is low. The ferroelectric hysteresis loop of the sintered samples is shown in Fig. 4b, which exhibits good symmetry. The remnant polarizations P_r of the samples are 2.2μC/cm², 2.4μC/cm² and 7.0μC/cm², respectively, when the electric fields are 10kV/cm, 20kV/cm and 30kV/cm. When the temperature increases

to 122°C, no hysteresis loop remain. Fig. 4c shows X-ray powder diffraction (XRD) measurement of sintered sample. The results show that the BaTiO₃ sintered samples keep perovskite structure after a high temperature heat-treatment. Therefore, they are suitable for piezoelectric applications. This performance suggests that the printed piezoelectric components have great potential to be used in piezoelectric devices.

Printing Focused Ultrasonic Transducer

In ultrasonic defectoscope (Nondestructive testing), ultrasonic imaging and high intensity focused ultrasound (HIFU), the focused piezoelectric element offers high sensitivity and smaller lateral resolution.^[38-40] The focusing features are usually achieved by machining process. However, with such process, the cracking and ferroelastic domain reorientation can increase the loss tangent and decrease the working life of piezoelectric layer, compared with the materials without machining process. To realize the energy transform as well as the focusing without the defect caused by the machining process, PF-CPE (shown in Fig. 3 d, 3e and 3f) was fabricated by the presented printing method and post-process. The PF-CPE was assembled in an ultrasonic transducer (Fig. 5b) to show its potential applications in ultrasonic imaging. Fig. 5a shows the design of the ultrasonic transducer. A Cr/Au layer was sputtered on the two faces of PF-CPE as the electrodes. A conductive epoxy (E-Solder 3022, Von Roll Isola Inc., New Haven, CT) with an acoustic impedance of ~6 MRayl was applied to the printed ceramic as the backing layer. A

10 μ m-thick parylene (SCS, Specialty Coating System, Indiana, USA) was vapor-deposited on the front face of the transducer to serve as a protection layer.

The ultrasound beam originates from mechanical oscillations of the piezoelectric element in the transducer. Echoes are produced due to the interaction of ultrasound with a target, and some of these echoes travel back to the transducer. The returned echoes are converted back into electrical impulses by the transducer and are further processed in order to form the ultrasound image. Supplementary Fig. S2 reveals the sketch of a non-focused and focused transmit beam. The focused transmit beam has the highest energy (output voltage) at the focus point where the lateral resolution has the minimum value.

Transducer performance

The actual performance of the transducer was test by the ultrasonic system shown in Supplementary Fig. S3. Figure. 6a shows the initial pulse and the echo generated by the printed focused transducer when the depth from the transducer to the quartz target is 5mm. The pulse and echo shows that the 3D printed ceramics have the ability to convert compressive/tensile stresses to an electric charge, and vice versa.

Field II program^[41,42] was used to predict the azimuthal beam profile generated by the printing focused transducer. Fig. 6b shows the distribution of pressure while the color bar presents the

normalized pressure. Which indicates that, in depth direction, the focus point (the maximum pressure) is about 14~18mm away from the transducer.

In order to find the focus point, the impulse-responses at different depth were measured. When the depth from the transducer to the quartz target is 15.5mm, the received echo has the maximum output voltage (peak to peak voltage is 0.301V). Fig. 6c shows the receive-echo response in time domain (solid line) and frequency spectrum converted by a Fast Fourier Transform of the time domain signal (dash line), in the depth direction. The center frequency and bandwidth (magnitude >-6dB) are 6.28MHz and 41.28%, respectively.

A tungsten wire (50 μ m diameter) phantom (Fig.7a) was imaged by the transducer to measure the resolution of the transducer (Fig. 7b). The amplifier portion and dynamic range were set at 20dB and -30dB, respectively. Figure. 7c shows that the lateral resolution has the minimum value (770 μ m) while the received echo has the maximum value (0.301V) at the same depth. This indicates that the beam can be well focused at 15~16mm by the printed focused transducer.

Figure. 8a shows the 6.28-MHz ultrasonic scan with 240 μ m axial resolution and 770 μ m lateral resolution through a porcine eyeball. The ultrasonic imaging shown in (Fig. 8b) provides crucial biometric information regarding eyeball structures, including the cornea, its constituent layers, and the anterior and posterior chambers, which cannot be achieved by the unfocused

piezoelectric element. The test results verify that our printing focused transducer suitable for ultrasonic application.

4. Conclusion

In summary, we investigated a novel 3D printing process in the fabrication of piezoelectric components using high solid loading slurry with nanoparticle. Through a specifically designed debinding and sintering process, the printed piezoelectric components display piezoelectric properties that can be used in biomedical imaging and other applications. The printed element was integrated into an ultrasound transducer. The transducer morphology lends itself to a focused beam profile. Examples images from a wire phantom and an Ex vivo porcine eye demonstrate the imaging capability. The results indicate that the printed piezoelectric element using MIP-SL technology based on nanoparticle opens a facile and effective way toward the realization of functional ceramic assembly in three-dimensional geometry. More piezoelectric array and matrix of composite with complex geometry enabled by the AM process potentially increase the effectiveness with which a piezoelectric material converts electrical energy into mechanical energy and vice versa, which bring us tremendous possibilities in applications such as energy harvesting and high intensity focused ultrasound.

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Figure. 1 (a) Illustration of the MIP-SL system. (b) Imaging pattern controlled by the projection. (c) 3D geometry designed by SolidWork. (d) Interface to control motor and projector. (e) Optical images of Green-part fabricated by MIP-SL system.

Figure. 2 (a) Schematics of green-part fabrication and post-processes. (b) SEM image of sintered sample after 6h sintering process at 1330°C . (c) SEM image of debinded sample.

Figure. 3 (a)(b)(c) Optical microscopy images of 3D-SAA with the 64 pillars annular segment array. (d)(e)(f) Optical microscopy images of the concaved-shaped piezoelectric element (PF-CPE).

Figure. 4 (a) The spectrum of impedance and phase. (b) Polarization–electric field hysteresis loops of the sintered ceramic. (c) XRD patterns of BaTiO_3 sintered ceramic.

Figure. 5(a) Profile of the transducer structure. (b) Optical image of the printed ceramic based transducer.

Figure. 6 (a) Initial pulse and echo generated by the printing focused transducer. (b) Beam profile simulation by Field II program. (c) Pulse-echo waveform (solid line) and normalized spectrum

Figure. 7 (a) Schematic of tungsten wire (50 μ m diameter) phantom (d) Phantom imaging using the 6.28-MHz transducer. (e) Voltage and lateral resolution as the function of depth between the transducer and the target.

Figure. 8 (a) The 6.28-MHz ultrasonic scan through porcine eyeball using the printing focused transducer. (b) Ultrasonic imaging of porcine eyeball.

Table 1 Comparison our printed BaTiO₃ to reported literature values.

Fabrication method	Density (g/cm ³)	ϵ (1kHz)	d33 (PC N ⁻¹)	$\tan\delta$ (1kHz)
Typical BaTiO ₃ ^[34]	6.02	1700	190	<0.1
MIP-SL (This study)	5.64	1350	160	0.012
Laser-sintering ^[35]	5.59	----	----	----
Binder Jetting ^[36]	3.93	640	74.1	0.03
Injection Moulding ^[37]	5.86	----	----	----

Figure. 1 (a) Illustration of the MIP-SL system. (b) Imaging pattern controlled by the projection. (c) 3D geometry designed by SolidWork. (d) Interface to control motor and projector. (e) Optical images of Green-part fabricated by MIP-SL system.

Figure. 2 (a) Schematics of green-part fabrication and post-processes. (b) SEM image of sintered sample after 6h sintering process at 1330°C . (c) SEM image of debinded sample.

Figure. 3 (a)(b)(c) Optical microscopy images of 3D-SAA with the 64 pillars annular segment array. (d)(e)(f) Optical microscopy images of the concaved-shaped piezoelectric element (PF-CPE).

Figure. 4 (a) The spectrum of impedance and phase. (b) Polarization–electric field hysteresis loops of the sintered ceramic. (c) XRD patterns of BatiO₃ sintered ceramic.

Figure. 5(a) Profile of the transducer structure. (b) Optical image of the printed ceramic based transducer.

Figure. 6 (a) Initial pulse and echo generated by the printing focused transducer. (b) Beam profile simulation by Field II program. (c) Pulse-echo waveform (solid line) and normalized spectrum

Figure. 7 (a) Schematic of tungsten wire (50 μ m diameter) phantom (d) Phantom imaging using the 6.28-MHz transducer. (e) Voltage and lateral resolution as the function of depth between the transducer and the target.

Figure. 8 (a) The 6.28-MHz ultrasonic scan through porcine eyeball using the printing focused transducer. (b) Ultrasonic imaging of porcine eyeball.

Graphical abstract