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Special Focus on Flexible Electronics Technology

Aerosol printing and photonic sintering of bioresorbable zinc nanoparticle ink for transient electronics manufacturing

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Abstract Bioresorbable electronics technology can potentially lead to revolutionary applications in healthcare, consumer electronics, and data security. This technology has been demonstrated by various functional devices. However, majority of these devices are realized by CMOS fabrication approaches involving complex and time-consuming processes that are high in cost and low in yield. Printing electronics technology represents a series of printing and post processing techniques that hold promise to make high performance bioresorbable electronics devices. But investigation of printing approaches for bioresorbable electronics is very limited. Here we demonstrate fabrication of conductive bioresorbable patterns using aerosol printing and photonic sintering approaches. Experimental results and simulation reveals that ink compositions, photonic energy, film thickness, and ventilation conditions may influence the effect of photonic sintering. A maximum conductivity of 22321.3 S/m can be achieved using 1 flash with energy of 25.88 J/cm² with duration of 2 ms. By combining two cascaded sintering procedures using flash light and laser further improve the conductivity to 34722.2 S/m. The results indicate that aerosol printing and photonic sintering can potentially yield mass fabrication of bioresorbable electronics, leading to prevalence of printable bioresorbable technology in consumer electronics and biomedical devices.

Keywords bioresorbable electronics, photonic sintering, aerosol printing, transient electronics, printed electronics, zinc nanoparticles

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1 Introduction

Bioresorbable electronics or transient electronics technology can be used to develop devices that dissolve in biological fluids and generate products that are safe for surrounding environment. This technology can potentially lead to revolutionary applications in healthcare by avoiding surgery processes for implantable devices [1] and environmental protections by reducing electronics waste by facilitating rapid recycling of consumer electronics [2, 3]. In addition, this technology can also prevent sensitive and valuable information from revealing by triggered device dissolution. Bioresorbable electronic devices have

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been demonstrated as functional components (e.g., sensors, diodes, transistors, antennas) [3] and circuits (e.g., logic circuit, signal generator, and power harvesters) [4,5] as well as systems [6,7]. In addition, research related to dissolution mechanisms and bioresorbable materials of such devices have also gained significant breakthrough [8–11]. The dominated approaches for fabricating these devices involve complex and time-consuming CMOS processes that have been modified to avoid water-based solvent and high temperature treatment due to sensitivity of the bioresorbable materials to humidity and extensive heating. Development of low cost and high yield fabrication approaches is urgently needed to allow bioresorbable electronics technology to generate more societal impact.

Alternative fabrication approaches for bioresorbable electronics can use printing technology, which features various printing methods such as screen printing [12], inkjet printing [13], and roll-to-roll printing [14] as well as series of post-processing approaches such as ink curing [15] and sintering [16] to improve mechanical and electrical properties of printed patterns. The printing electronic technology can lead to high performance flexible electronic devices with large surface area, and is notable for its low-cost, high-speed and low-temperature fabrication procedures [17]. It is capability has been demonstrated as thin-film transistors [18, 19], solar cells [20, 21], radio frequency identification (RFID) tags [22, 23], strain sensors [24, 25], and organic light emitting diodes (OLEDs) [26, 27]. Inspired by the printing electronics technology, bioresorbable electronic devices have been realized through laser, screen, stencil or 3D printing methods using nano or micro particles of zinc (Zn) [2,28], magnesium (Mg) [29], and iron (Fe) [30, 31]. However, printing bioresorbable patterns using more controllable mask-less manners such as inkjet printing and aerosol printing have not yet been achieved. In addition, transient metals such as Zn and Mg are highly reactive and tend to form spontaneous surface oxidation, thus sintering methods such as microwave and electrical sintering, which requires initial conductivity, are no longer feasible. Moreover, sintering approaches based on overall heating or intensive localized heating may cause irreversible bioresorbable substrate damage. Thus, fabrication of printable bioresorbable electronic devices demands improvement both in printing approaches and sintering methods. Recent developed photonic sintering approach used flash light with broad wavelength to promote photon absorption, causing localized heating of nanoparticles within short time periods. The transparent feature of the bioresorbable polymer substrates allows selective sintering of nanoparticles using photonic sintering, while keeping the substrate theoretically unaffected. Our previous works demonstrated some preliminary results of photonic sintering effect of ball-milled zinc nanoparticles (Zn NPs) [32]. However, some critical issues such as interaction of photonic energy with transient nanoparticles, surface chemistry and composition changes, and heat dissipation have not yet been fully investigated. In this paper, we demonstrate a technique to generate bioresorbable electronic patterns through aerosol printing and photonic sintering. The effect of photonic sintering to ZnNPs, including ink compositions, photonic energy levels, thermal effects, dendrite formation, and adhesion between printed patterns and substrates are studied by experiments and simulation. In addition, laser annealing approach has also been used to compare with photonic sintering in fabricating transient electronic devices. The high adaptability to a range of viscosity (0.5 to 2500 cP) and various solvents makes aerosol printing preferable for bioresorbable electronic inks. Both the experimental and simulation data have suggested that the combination of aerosol printing and photonic sintering can potentially yield mass fabrication of bioresorbable electronics, leading to prevalence of printable bioresorbable technology in consumer electronics and biomedical devices.

2 Materials and experiments

2.1 Preparation of bioresorbable zinc nanoparticle inks and substrates

Bioresorbable zinc nanoparticle inks were prepared by mixing different concentrations (0.1 and 1 wt%) of Polyvinylpyrrolidone (PVP) (Sigma Aldrich, Mw: 10000) with a solution containing 90 wt% methanol and 10 wt% butyl acetate. The solution is then mixed with Zn NPs (US Research Nanomaterials Inc., high purity, 99.9%) at 20% filling ratio, followed by sonicating for 1 h to obtain the nanoparticle inks. The 20% filling ratio is chosen as a higher ratio may lead to the clogging of the aerosol printer, while a



Figure 1 (Color online) Overview of fabricating bioresorbable electronic patterns through aerosol printing and photonic sintering approaches. (a) The schematics of the setup for aerosol printing and photonic sintering; (b) as deposited Zn nanoparticles with 0.1 wt% PVP; (c) sintered Zn nanoparticles with 1 flash at 20.7 J/cm² on a glass substrate; (d) changes of average diameter of the particles and conductivity of printed patterns with flash energy; (e) dissolution of bioresorbable patterns in water. The error bars indicate the range of the experimental measurements.

lower filling ratio may result in diluted inks that demand several repeated printing process in order to achieve ideal linewidth, thickness and conductivity of printed patterns. Bioresorbable substrates used in the experiments were prepared by adding 2 wt% Sodiumcaboxymethyl cellulose (Na-CMC) (Sigma Aldrich) into water and stirring continuously at 600 rpm for 3 h. The solution was then subject to filtration, and left to dry for 3–7 days to form a thick, clear, flexible and water-soluble film.

2.2 Bioresorbable patterns achieved by an aerosol printer and photonic sintering

Figure 1(a) shows a schematic to obtain a conductive pattern of Zn on a bioresorbable Na-CMC substrate through aerosol printing and photonic sintering approaches. The aerosol printing technique is a relative new printing method, which can tolerate large range of viscosity and support a wide choice of materials The aerosol printing of Zn patterns was achieved using a customized aerosol printer, which contains a staging system, a flow controller, a pneumatic nebulizer, a virtual impactor, and 3 printer heads (Figure S1). The nebulizer atomizes the prepared ink into droplets $1-5 \ \mu m$ in diameter through Argon (Ar) gas flow supplied by the flow controllers, resulting in aerosol mist that contains both ink droplets and Ar molecules. The flow controllers is then generate Ar gas to carry the atomized mist to the printer heads, allowing ejecting the mist through the printer heads onto a substrate 5 mm away from the heads on the staging system that move the substrate to achieve designed geometry. A virtual impactor connected with the nebulizer can separate droplets of different sizes to obtain uniform droplet output.

The printed pattern cured at a temperature below 100°C to remove the solvent before being photonic sintered. The photonic sintering system (Sinteron 2000-L, Xenon Corporation) offers a broad spectrum (200–1000 nm) of pulsed xenon light with an output energy density from 9.88 J/cm² to 33.33 J/cm² and

a pulse duration of 2 ms. The photonic sintering was conducted in a custom-made aluminum enclosure filled with Argon. The pulsed light can penetrate through a transparent double-side polished sapphire wafer (University Wafer Inc.) on the top of the aluminum enclosure. To achieve better uniformity, the patterns printed on Na-CMC substrates can be hot rolled using a commercial hot roller (MTI Corporation, MSK-2150-DC) [33] at 60°C before sintering to compress the patterns and reduce the void space within the patterns due to solvent evaporation.

2.3 Sample characterization and simulation

The conductivity of the printed patterns was measured using a C4S 44/5S four-point probe measurement system from Cascade Microtech, Inc with a Keithley 220 programmable current source, before and after sintering. Morphology of the sintered patterns were characterized by a scanning electron microscope (Helios NanoLab 600), which also equipped with an Oxford Energy Dispersive Spectrometer (EDS) with resolution < 30 nm to conduct simultaneous surface analysis. X-ray photoelectron spectroscopy (XPS) was conducted using a Kratos Axis 165 spectrometer with a 150 W aluminum X-ray source. Some undesired effects such as sample charging were compensated by fixing the C1s carbon peak as suggested by previously published papers [34–36].

The simulation was performed based on numerical solution of mass, energy and momentum conservation equations. The volume of fluid method was used to track the interfaces between nanoparticle and ambient gas. The conservation equation was discretized and solved using a finite difference method in Fluent. The transient simulation was performed in a 2D 5 μ m×5 μ m computation domain, which comprises a layer of Zn NPs (2–3 μ m in thickness) on a glass substrate. The Zn NPs layer was initialized using results from separated Discrete Element Method simulation in which Zn NPs depositions and aggregations were simulated. An optical penetration depth of 3 μ m and an incident optical intensity of 9×10⁸ W/m² were used. The optical attenuation and absorption was calculated following the Beer's law. The pulse duration was assumed to be 1 μ s, which is shorter than actual pulse (2 ms) due mainly to the limited computational capability. Accordingly, higher optical intensity was used in simulation (actual intensity in experiments ~ 4×10⁷ to 2×10⁸ W/m²).

3 Results and discussion

The effect of photonic sintering is first demonstrated by comparing the morphology and conductivity changes of printed patterns before and after sintering. The as-printed Zn NPs isolated by PVP encapsulation is shown in Figure 1(b). These Zn NPs are coalesced to form conductive matrix after photonic sintering (Figure 1(c)). The degree of coalescence can be characterized by measuring the sizes of particles with respect to the increased sintering energy. Similar trends both in the degree of coalescence and conductivity are observed (Figure 1(d)). The best conductivity obtained was 22321.3 S/m for the ink with 0.1 wt% PVP concentration and spherical Zn NPs (50 nm in size) at an sintering energy of 25.88 J/cm². This conductivity is about 5 times better than the previous work on Zn powders [2], but twice smaller than our previous reported data using ball milled Zn NPs with irregular shapes [32]. The resulting patterns demonstrated as coils and meander lines can dissolve in water completely within 12 h, leaving almost not trace in the water (Figure 1(e)). Further increase of photonic energy cause large temperature increase in the printed patterns, resulting in substrate deformation and delamination of the printed patterns from the substrate (Figure S2), thus higher photonic energy (> 25.88 J/cm²) was not used throughout the experiments.

The effect of PVP in the bioresorbable inks has been studied by varying the PVP concentrations in the inks as shown in Figure 2. PVP polymers function as stabilizers to wrap around the nanoparticles to prevent the nanoparticles from direct contact with atmosphere that may cause surface oxidation of Zn NPs. In addition, the amphiphilic nature of PVP can stabilize neighboring particles through electrostatic or/and electrosteric effect, and substantially reduce the van der Waals attraction between the particles. Inks with three different PVP ratios (0, 0.1 and 1 wt%) were prepared and aerosol printed. When no



(b

0.1% PVP



Figure 2 (Color online) Effects of PVP in bioresorbable inks made of Zn NPs. Aerosol printed patterns with (a) 0 wt%, (b) 0.1 wt%, and (c) 1 wt% of PVP when sintered using an increased energy density at 10.18, 14.15 and 20.7 J/cm² from left to right. The scale bar is 5 μ m. (d) Changes of conductivity and (e) changes of average particle diameter of the samples with different PVP concentrations with flash energy. The error bars indicate the range of the experimental measurements.

PVP is involved, the ink tends to agglomerate, causing frequent clogging in the printing heads and the nebulizer. Only high sintering energies (> 20.7 J/cm^2) can cause the coalescence of the nanoparticles after photonic sintering. The best conductivity that can be achieved without any damage to the Na-CMC substrate is 5605.3 S/m. A large PVP level (1 wt%) results in thick surface coating, which is difficult to be completely removed during the sintering process, resulting in lower conductivity (Av: 2224.2 S/m). The ink containing 0.1 wt% PVP can achieve a high degree of coalescence of nanoparticles and increased conductivity as shown in Figure 1(d) and 2(c), suggesting that 0.1 wt% of PVP may be an optimum amount that offers both good dispersion and proper surface coating. This finding is also consistent with our previously reported results using ball milled samples. It is also noticed that both the conductivity and the diameters of the nanoparticles distribute over large ranges with notable error bars after different sintering conditions in Figure 2(d) and (e). In contrast, large distribution of the conductivity and the diameters only occur at low sintering energy for samples coated with 0.1% PVP (Figure 1(d)), indicating that the repeatability of sintering effectiveness may be related to uniformity of the sintered samples and their light absorption capability. The agglomeration of nanoparticles with PVP and thick surface coating of nanoparticles with 1% PVP may cause large deviations over different measurement areas in terms of surface qualities and light absorption efficiency.

To further study the role of PVP, XPS was performed on the samples sintered at 2.3 kV. The XPS spectra only shows the presence elements of Zn, C, N and O, which are expected within the samples.



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Figure 3 (Color online) Study of sintering mechanism in printed Zn patterns. (a) A model containing Zn NPs on a glass substrate in Argon environment is used to conducted simulation in Fluent. Simulated geometry changes as a function of time in (b) thin and (c) thick layers of Zn NPs. (d) Simulated temperature changes during the photonic sintering process for thick and thin layer of Zn NPs. (e) SEM images of samples sintered in air using a photonic energy of 20.7 J/cm² after 1–4 flashes. The images indicate dendrite formation due to rapid cooling rates.

The XPS spectra of samples treating with 0, 0.1 and 1 wt% PVP are significantly different from each other (Figure S3). Using sample containing no PVP as the baseline, changes of surface composition of the sintered samples with varied PVP levels can be comparably analyzed. The C1s peak at 284.8 eV has been deconvoluted to three peaks at 284.8, 285.5 and 288.6 eV for the sample without PVP (Figure S3(a)), corresponding to C-C (adventitious carbon), C-O and C=O respectively. The peaks representing C-C are present in all samples, while the C-O peak reduces with increased PVP ratios and eventually disappear at 1% PVP ratio. The C=O peaks show reduced magnitude with increased PVP ratios, indicating that increased PVP can effectively reduce surface contamination associated with C-O and C=O groups. The oxygen peak, which has been deconvoluted into 529.7 and 531.15 eV for the sample containing no PVP, corresponds to Zn-O [37] and C-O groups [38] respectively. While Zn-O peak is consistent suggesting some degree of oxidation in all samples, the ratio between metal oxides and carbonates becomes smaller as higher concentration of PVP is used, indication surface protection by carbon. It observed that the $Zn2p_{3/2}$ peaks for the sample containing 0 wt% PVP is around 1018.8 eV and samples containing 0.1 and 1 wt% PVP are around 1021 eV. This $\sim 2 \text{ eV}$ shift in binding energy indicates that the samples with 0.1 and 1 wt% PVP are much less oxidized than the sample without any PVP [39]. The reduced oxidation effect correlates well with the surface coating function of PVP. As the sintering process may causes melting, evaporation and rearrangement of the zinc nanoparticles on the surface, which influence the magnitude of the XPS detection, thus, the weight percentages of different elements are not quantified using the XPS results.

The thermal process during the photonic sintering can be analyzed using finite element analysis by Fluent. The simulated results offer further insight to the coalescence process and the time-dependent temperature within the sintered film, when experimental measurement of temperature variation is difficult to achieve given the short sintering time period. Two films with thickness at 1.8 and 3.2 μ m were selected for the simulation. The results indicate that Zn NPs rapidly transform into liquid phase in 1.75 μ s, and gradually turn into a large spherical droplet before solidified (Figure 3(b) and (c)). Additionally, temperature profile shows that thicker film reaches higher temperature and remains in liquid phase (above the melting point of Zn) for a longer period (Figure 3(d)). Higher temperature generated within



Figure 4 (Color online) Adhesion of printed Zn NPs patterns with Na-CMC substrates. Cross section view of images of (a) an as-printed sample and (b) a sample after 1 flash at 25.88 J/cm^2 . (c) EDS images of color maps of Zinc (Zn) and Carbon (C). Sample with different line widths (d) before and after peeling by Kapton tapes for (e) 5 times and (f) 25 times, respectively.

the thicker film may be due to increase photonic energy absorption and temperature buildup in larger volume which in turn produces more melting and hence high conductivity. SEM images in Figure S4 is in cooperation with the simulation findings. When two films with different thickness exposed to same amount of photonic energy, the thick film shows larger solidified particles as compared with a thin film. The simulation suggests that flash light with a time duration of 2 ms may be sufficient to generate enough heat to melt Zn NPs and proper thickness should be chosen to achieve better sintering effect.

The ventilation conditions during the sintering process can significantly change the surface morphology of the sintered samples. Dendrite formation is observed when samples were sintered directly in an atmosphere condition with forced air flow to promote heat dissipation. By sintering a Zn nanoparticle film with various numbers of flashes (from 1 to 4) using a photonic energy density of 20.7 J/cm^2 , different levels of Zn dendrite formation are observed. The EDS results indicate that the dendrites majorly contain Zn element (82.42 wt%), while carbon and oxygen only take up 4.75 wt% and 12.83 wt% (Figure S5), respectively. The formation of dendrite during the metal solidification processes may be attributed to the rapid cooling rate of the melted Zn NPs, as dendrite formation is not observed when the alumina enclosure filled with static Argon gas is used due to minimized heat exchange. Other ventilation parameters were not explored in current research due to limitation in experimental setup. Further investigation of different ventilation conditions such as gas flow rate and heat dissipation speed will be pursued in our future research to gain better understanding of the dendrite formation mechanism and photonic sintering effect.

The adhesion between the bioresorbable patterns and substrates were investigated. It is observed that localized heat generated during the photonic sintering process enhances the adhesion between the bioresorbable patterns and substrates by melting the top surface ($\sim 5-10 \ \mu m$) of the substrate, allowing embedding the bottom portion of the conductive matrix into the substrate (Figure 4(b)). The melt thickness of the top surface is subject to change due to different sintering parameters and ventilation conditions. The EDS images (Figure 4(c)) indicate that the interface of conductive Zn matrix and underlying Na-CMC substrate changes from a smooth surface (Figure 4(a)) into a rough surface, which is desirable for achieving good adhesion. Adhesion tests performed (Figure 4(d)–(f)) by peeling off Kapton tapes from the surface of the printed inks shows negligible delamination after 5 peeling cycles, suggesting



Figure 5 (Color online) Thermal effect between the photonic sintering and laser annealing. (a) A schematic of an experimental setup to conduct photonic sintering followed by laser annealing; (b) substrate damage when only laser annealing is used; (c) morphology changes of a sample that was only subjected to laser annealing; (d) conductivity changes of the samples annealed by laser only; (e) morphology changes of a sample that was only subjected to both photonic sintering and laser annealing; (f) conductivity changes of the samples that have been photonic sintered and laser annealed.

good adhesion of the Zn matrix to the substrate surface. The printed patterns can be further encapsulated with layers of MgO, SiO_2 or Si_3N_4 , which can also slowly dissolve in aquatic environment, to protect the underlying patterns from humidity in the environment and ensure their longevity.

As both photonic sintering and laser annealing are considered as promising methods to sinter nanoparticles in printing electronics, thus the thermal effect between the photonic sintering and laser annealing has been compared, and the possibility to combine these two processes to yield better device performance has also been studied. The samples were sealed in the Al enclosure filled with Ar. They were either purely annealed by laser or dually sintered by combination of photonic sintering and laser annealing (Figure 5(a)). However, directly annealing with laser (IPG, 1065 nm, CW, 100 W) can cause significant damage to the substrate due to high intensity localized heating (Figure 5(b)). For samples (containing 0.1 wt% PVP) that have been purely annealed by laser, exhibit randomly distributed dendrite structures with large pin holes on the underneath substrate (Figure 5(b) and (c)). The maximum conductivity can reach 10416.7 S/m in this case. However, the changes of conductivities do not vary monotonously with the scanning speed (from 20 to 60 mm/s) and the power of the laser beam (from 0.125 to 0.45 W) (Figure 5(d)). The randomly distributed conductivity for laser annealed samples may be due to combined effects of damaged substrates, different measurement areas, and pattern delamination. In contrast, negligible substrate damage has been observed when samples are processed by photonic sintering (Figure 5(e)). When combining photonic sintering with laser annealing, a Zn film with uniform distribution of sintered Zn microparticles and improved conductivity can be obtained (Figure 5(e)). The samples were initially subjected to photonic sintering with a flash of 25.88 J/cm^2 and then moved to the laser annealing setup without disturbing the Ar environment in the aluminum enclosure. An optimum laser power (0.45 W) and scanning speed (40 mm/s) are observed in Figure 4(e), leading to a highest conductivity of 34722.2 S/m (Figure 5(f)). The experimental data demonstrate that the photonic sintering generate significantly less damage to the underlying substrates. The combination of two sintering processes may potentially lead to further improvement in device performance.

4 Conclusion

This paper reports a process of mass manufacturing printable bioresorbable electronics devices by aerosol printing and photonic sintering methods. A bioresorbable ink containing PVP coated Zn NPs was printed on bioresorbable substrates and sintered in argon environment to achieve a conductive Zn matrix with the best conductivity of 22321.3 S/m. The conductivity of the Zn matrix can be further improved by combining photonic sintering and laser annealing or by reducing surface oxidation using formic acid or acetic acid as demonstrated by Lee et al. [40]. Systematic characterization reveals the role of PVP in controlling the oxidation and the effect of thickness of PVP coating on Zn NPs in the process of sintering. Simulation results indicate that sintering of thin and thick layers of nanoparticles can be completed within a few microseconds. The novel printing and sintering techniques proposed here feature low-cost and high efficiency, and potentially pave the way for mass manufacturing of bioresorbable printed electronics to facilitate the applications of the bioresorbable electronics technology to renovate healthcare and consumer electronics.

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Supporting information Figures S1–S5. The supporting information is available online at info.scichina. com and link.springer.com. The supporting materials are published as submitted, without typesetting or editing. The responsibility for scientific accuracy and content remains entirely with the authors.

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