Arrays of hollow out-of-plane microneedles made by metal electrodeposition onto solvent cast conductive polymer structures

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Abstract
Transdermal drug delivery using microneedles is a technique to potentially replace hypodermic needles for injection of many vaccines and drugs. Fabrication of hollow metallic microneedles so far has been associated with time-consuming steps that restrict batch production of these devices. Here, we are presenting a novel method for making metallic microneedles with any desired height, spacing, and lumen size. In our process, we use solvent casting to coat a mold, which contains an array of pillars, with a conductive polymer composite layer. The conductive layer is then used as a seed layer in a metal electrodeposition process. To characterize the process, the conductivity of the polymer composite with respect to different filler concentrations was investigated. In addition, plasma etching of the polymer was characterized. The electroplating process was also studied further to control the thickness of the microneedle array plate. The strength of the microneedle devices was evaluated through a series of compression tests, while their performance for transdermal drug delivery was tested by injection of 2.28 μm fluorescent microspheres into animal skin. The fabricated metallic microneedles seem appropriate for subcutaneous delivery of drugs and microspheres.

(Some figures may appear in colour only in the online journal)

1. Introduction
Microneedles are sharp microstructures less than 1 mm long, that have been used for transdermal drug delivery [1] or extraction of biofluids for sensing applications [2]. They can be solid or hollow, and they can be arranged in out-of-plane or in-plane arrays.

For drug delivery applications the drug is transferred to the epidermis or the dermal skin layer once the microneedle tips penetrate the skin’s first layer, the stratum corneum (SC). In the case of hollow microneedles, the drug is directly injected into the skin tissue, and then diffuses toward the blood vessels. Solid microneedles, however, are used to increase skin permeability by creating holes on the SC surface and then exposing the underlying layers to drugs that are embedded within the microneedle structure, coated on the microneedles surfaces, or later applied to the application site [3, 4]. Solid microneedles have been shown to improve skin permeability by four orders of magnitude compared with traditional adhesive patches that rely on drug absorption...
through the intact skin surface [4, 5]. Drug delivery using solid microneedles is, however, passive and limited by the volume of the drug to be injected as well as its delivery rate. In contrast, hollow microneedles can facilitate transdermal delivery of larger volumes of drugs at rapid and predetermined rates.

For biosensing applications, hollow microneedles are used to collect interstitial fluid (ISF) or small amounts of blood, which are then analyzed for a particular compound [2, 6–8]. One method for sampling the ISF or blood relies on capillary action, which requires the needle lumens to contain hydrophilic surfaces [2]. Another method for sampling ISF or blood is applying a low pressure through the microneedle lumens [8].

Microneedles can facilitate fluid injection and sampling painlessly or with minimal pain sensation (mostly from the applied pressure) if they do not penetrate deep enough to reach the nerve endings in the skin’s dermis layer. Previous studies have demonstrated painless injection of drugs into the skin using microneedles [9]. To ensure painless or minimally painful fluid transfer, microneedles should not penetrate much deeper than the total thickness of the epidermis. The average thickness of the human SC layer is 14.8 ± 4.8 μm while the average thickness of the viable epidermis is 68.9 ± 17.0 μm [10]. As the standard deviations suggest, these thickness values vary widely throughout the human body. Depending on the shape and aspect ratio of the microneedles as well as the elasticity and density of the application site on the human body, which itself is a function of age and gender [11–13], only a certain portion of the microneedles effectively pierce through the skin; to this date, no previous work has studied the effective penetration depth for different microneedle designs on various skin types. Therefore, having a flexible fabrication process that allows making microneedles with custom dimensions would be useful for situations where the optimized needle dimensions are studied.

Several fabrication concepts have been proposed for making hollow microneedles from single- or polycrystalline silicon [2, 14–20], glass [21], polymer [1, 22–25] and metal [6, 26–30]. The main barriers for wide commercial adoption of microneedles have been the costs and time-consuming steps associated with fabrication of these microneedles. Fabrication of silicon or glass microneedles, for instance, relies on deep reactive ion etching of silicon to create deep channels, which is not an ideal process for low cost batch fabrication. Many polymer microneedle fabrication processes require multiple UV exposure or mold transfer and assembly steps, which is time consuming and labor costly. Previous fabrication processes for metallic microneedles also require sequential formation of disposable polymer molds for electroplating; additionally, in most cases, metallic microneedles are opened through laser cutting of individual needle tips, which can be time costly for making arrays containing a large number of microneedles.

We have previously demonstrated a fast and cost effective method for making microneedles from polymer composite materials [1]. The needles were fabricated through a solvent casting process on a reusable mold that allowed fabrication of the microneedles with a variety of polymeric materials. Using that process, 250 μm long microneedles were fabricated from polyimide-clay composite and tested for robustness as well as drug delivery into animal skin.

In this work, we have used a similar solvent casting process to form a thin conductive polymeric layer on reusable molds that contain arrays of vertical pillar; the conductive polymer layer is then used as a seed layer in an electroplating process to fabricate metallic microneedles. Several polymers have been previously shown to be capable of conducting electricity [31, 32]. Although their conductivity is not as high as the conductivity of metals, they are used in many small-scale industrial applications because they are easy to process. In addition, nonconductive polymers can also be modified to conduct electricity by adding conducting particles to their matrix. Nickel-coated particles, copper powder, carbon black and graphite are some examples of fillers used to increase polymer conductivity [33–38].

In order to further characterize the fabrication process, some experiments were carried out to investigate the conductivity of the polymer layer, its plasma etching rate and the metal electroplating process. The fabricated metallic microneedles were also tested for robustness through a series of mechanical compression tests, and their failure loads were compared with forces required for skin penetration. The microneedles’ adequacy for skin penetration and drug delivery was then proven by using them to inject fluorescent beads into animal skin.

2. Fabrication of hollow metallic microneedles

2.1. Fabrication of the mold

The fabrication process of microneedles is summarized in figure 1. It starts with making a mold through photolithography for which the epoxy-type negative photoresist SU-8 (MicroChem, Newton, MA) was used. A 700 μm thick layer of SU-8 2150 was first spin coated onto a 300 μm thick Pyrex wafer. The wafer was then soft baked for 10 min at 65 °C and 2.5 h at 95 °C. The SU-8 was then exposed to 9200 mJ cm⁻² of UV light (performed in multiple 3 min intervals with 20 s cooling breaks in between). The exposure was performed through a dark field mask containing arrays of transparent circles with 40 μm diameter. These circular regions lead to the SU-8 pillars of the mold that are used as the basic structures for microneedle formation, and therefore, their spacing and size defines the final spacing and the approximate channel diameters of the microneedles in the array. In addition, the exposure was carried out from the back side of the Pyrex substrate. This method of exposure takes advantage of the gap between the mask and the photoresist and leads to cone shaped pillars, which eventually translates into microneedles with wide channel openings and sharp tips [1, 39]. The sharpness of the pillars can also be further increased by performing additional dry etching steps on the pillars to isotropically reduce the pillars’ width to achieve a desired sharpness. Characterization of such dry etching steps was not pursued for the purpose of this work. After exposure, the sample was
Figure 1. Fabrication process for making hollow metallic microneedles, (a) fabrication of the mold containing cone shaped pillars using backside exposure of SU-8 photoresist, (b) deposition of the PMMA/CB + NMP solution, (c) evaporation of NMP at 80 °C, (d) O₂-CF₄ plasma etching of dried PMMA/CB, (e) electrodeposition of the metal layer, (f) removing the microneedle array by dissolving the PMMA/CB in NMP.

baked for 5 min at 65 °C and 35 min at 95 °C. Finally, the sample was placed in a developer bath for 50 min to remove the unexposed photoresist. A final hard-baking step was carried out in an oven set to 175 °C for 1 h. For convenience, the Pyrex wafer was cut into smaller 1 × 1 cm² pieces, with each piece containing an array of pillars corresponding to a single microneedle array.

In order to further increase the strength of the pillars and their bonding with the Pyrex substrate an additional SU-8 layer was cast on the mold pieces. For this step, SU-8 3025 was first diluted with cyclopentanone to make a 6.7 wt% solution and then cast on the mold at 95 °C for 20 min, leading to a 30 μm thick coating on the base substrate. This layer was then cured with 900 mJ cm⁻² of UV light and then baked at 95 °C for 5 min followed by 190 °C for 1 h. At this stage, the molds were complete and used multiple times for the fabrication of microneedles (figures 1(a) and 2(a)).

2.2. Deposition of the polymer-based conductive seed layer

Poly(methyl methacrylate) (PMMA, Polysciences, Warrington, PA), with molecular weight of 25 kDa, seeded with carbon black (CB, VULCAN XC72R, Cabot, Boston, MA), with a primary particle size of about 150 nm, was used as the conductive polymer composite [33, 34]. Although other particles such as copper particles or graphite can be used as conducting fillers, CB was used in this work mainly due to availability of the material and its low cost. Using any other filler would suffice as long as the conductivity of the deposited film is high enough to facilitate the electrodeposition of the metal layer. However, particles of higher density than CB have been observed to settle quickly during the evaporation process leading to a depleted region near the top of the pillars.

To prepare the PMMA/CB polymer solution/particle suspension, first 0.3 g of PMMA was dissolved in 4.55 g of N-methyl-2-pyrrolidone (NMP). Next, 0.135 g of CB was mixed with the solution. Subsequently, 0.015 g of sodium dodecyl sulfate (SDS, Sigma-Aldrich, Oakville, ON) was added as surfactant. The solution was then placed in an ultrasonic bath for 30 min. The SDS surfactant prevents the formation of CB particle clusters and therefore leads to a uniform suspension of the particles in the solution, which consequently results in a uniform distribution of the particles within the polymer matrix once the material is cast on the mold; this is necessary for getting a uniform conductivity and therefore metal coating on its surface. The resulting fluid had a solid concentration of 9 wt% with the CB accounting for 30% of the total solid content.

After fabrication of the mold, 20 μl of hexamethyldisilazane (HMDS, Sigma-Aldrich, Oakville, ON) was applied to the mold in room temperature to improve
Figure 2. Image of a mold used for microneedle fabrication. (a) before coating with PMMA/CB, (b) coated with PMMA/CB.

its surface adhesion. Next, 40 μl of the 9 wt% PMMA/CB mixture was deposited into the mold and then heated to 80 °C for 3 h to evaporate the NMP and fully dry the PMMA/CB composite (figures 1(b), (c), and 2(b)). The thickness of this layer was 100 μm on the base plate which gradually decreased toward the tip of the pillars. The PMMA/CB composite layer was then used as a seed layer in a metal electroplating process. This layer also served as a sacrificial layer, which was later removed to separate the metallic microneedle structures from the mold. It has previously been shown that the thickness of a polymer layer on the pillars that was deposited using solvent casting can be adjusted by controlling the solvent casting process parameters such as the temperature and the dew point [40]. The thickness of the PMMA/CB seed layer on the pillars can therefore be adjusted in order to tune the microneedle shapes. After the PMMA/CB casting step, a thin PMMA/CB layer covered the top surfaces of the pillars; this layer was removed using an O2/CF4 plasma etching step (O2: 80 sccm, CF4: 20 sccm, pressure: 500 mTorr, temperature: 25 °C, power: 200 W, and duration: 200 s) in order to expose the SU-8 pillar tips (figure 1(d)). This etching step ensures that the nonconducting needle tips would be open once the needles are formed through the electroplating process.

2.3. Metal deposition

The next fabrication step involved deposition of metallic layers that constitute the microneedle array structure. This process was performed in two steps: first a thick nickel layer was electroplated onto the conductive polymer layer (figure 1(e)), which made up the main structural material; then, the nickel layer was coated with a thin layer of gold to cover the outer surface of the microneedles. Nickel is inexpensive and has a high compressive strength and Young’s modulus compared with polymers, silicon, and many other metals [41, 42]. Electrodeposition of nickel has been used in numerous applications and is a well-established process. Since nickel has been found to cause allergic reaction upon skin contact in some people [43], it is coated with a gold layer to improve its biocompatibility [44].

For nickel deposition, after the microneedle mold piece was coated with the conductive polymer, it was positioned parallel to a pure nickel anode at a distance of 2.5 cm inside an electroplating solution consisting of nickel chloride (25 g l\(^{-1}\)), nickel sulfate (170 g l\(^{-1}\)), and boric acid (15 g l\(^{-1}\)). Figure 3 shows the schematic of the electroplating setup. Since the conductive layer had a lower conductivity than the contact wire connected to the power supply, the top portion of the coated mold piece was kept outside of the electroplating solution in order to prevent a point of high field strength resulting in an accumulation of nickel on the contact wire. The power source was set to provide a constant current of 2 mA and the process was set to run for 150 min, which resulted in a 70 μm thick backing layer. After deposition of nickel, a 20 nm layer of gold was sputtered onto the top surface of the microneedle array.

2.4. Microneedle array lift-off

Removing the microneedle array was finally facilitated by dissolving the PMMA/CB layer in NMP and in an ultrasonic
bath for 60 min (figure 1(f)). Once the microneedle array was separated, the mold could be further cleaned with acetone or NMP to remove any leftover CB particles and used again for fabrication. In addition, the dissolved PMMA/CB layer can be potentially reused for fabrication after evaporating part of the NMP to reduce its concentration to an amount applicable for solvent casting. Figure 4 shows images of microneedles fabricated using this process.

3. Experimental procedures

3.1. Conductivity measurements of PMMA/CB composites

A series of experiments were carried out to investigate the effect of CB content on conductivity of the PMMA/CB composite. For these experiments, PMMA/CB suspensions in NMP were prepared with a CB concentration in the final solid varying between 0 and 50 wt%. All suspensions had a total solid concentration (CB, SDS, and PMMA) of 9 wt%. After the solutions were prepared, they were deposited into cylindrical cavities with diameters of 4 mm to create disk-shaped composite films for conductivity measurements. The film thicknesses were then obtained based on deposition volume and the solids volume fraction in the solution, and were also verified using a digital micrometer. A copper layer was evaporated on the top and bottom surfaces of the films. The electrical resistance values $R$ of the films were then measured using a conventional multimeter, by placing the meter probes on the top and bottom copper surfaces. The resistivity values $\rho = \frac{R A}{l}$ were calculated from the thickness $l$ of the cast film, the measured resistance $R$, and the area $A$ of the circular cross section of the cylinders. The conductivity values were obtained by inverting the resistivity values.

3.2. Dry etching of the PMMA/CB layer

The dry etching step in the fabrication process impacts the height of the microneedles as well as their tip diameter; it is therefore necessary to precisely control this process to ensure there will be enough polymer removed from the pillar tips so that the microneedle tips will be open, while maintaining the sharpness and aspect ratio of the microneedles.

Through a series of experiments, the plasma etch rate of the PMMA/CB composite films were measured for different gas flow settings. It had previously been shown that O$_2$ and CF$_4$ plasma etching is an effective dry etching technique for removal of many organic polymers including PMMA [45, 46]. Here, a Trion RIE/PECVD (Trion Technology, Clearwater, FL) was used to investigate the etch rate of the
PMMA/CB composite in plasma. First, 100 μm thick layers of PMMA/CB composite, with 30 wt% CB in solid, were cast on 1 × 1 cm² glass chips. The surfaces were then partially covered with an adhesive polyimide tape to partially protect them from the plasma. The glass chips were then placed in the plasma etcher and were etched with different combinations of O₂ and CF₄ volumetric flow rates. The total flow rate was adjusted to 100 sccm. The power was set to 200 W, and the chamber pressure and temperature to 500 mTorr and 25 °C, respectively. The etching process was set to run for 300 s for all the trials.

For determining the etch rates, first, the amount of polymer that was removed by plasma was measured. For this purpose, once the process was complete, the polyimide tape was removed from the polymer surface, and then a 40 nm layer of gold was uniformly evaporated onto the surface. A Wyko NT1100 optical profilometer (Veeco, Plainview, NY) was then used to measure the height difference between the areas exposed to the plasma and the areas covered with the tape, which corresponded to the etch depth. The etch rate was then calculated by dividing this height difference by the duration of the etching process.

3.3. Characterization of the nickel electroplating process

The electroplated nickel constitutes the main structural material of the microneedles and its deposition thickness controls the rigidity and wall thickness of the microneedles, as well as the back plate of the array.

Through a series of tests, the effect of the process duration and the electric current on the nickel thickness was investigated, in order to measure the deposition rate of the nickel with respect to these parameters. For this purpose, 100 μm layers of PMMA/CB, with 30% CB in solid, were cast on glass chips, which were then immersed in the electroplating solution and used as seed layers similar to the microneedle electroplating fabrication steps. Once the electroplating process was complete for each trial, an Alpha Step 200 profilometer (1% accuracy, KLA-Tencor, Milpitas, CA) was used to measure the deposited metal thickness.

3.4. Mechanical compression tests on the fabricated microneedles

The strength of the microneedles was measured through a series of mechanical compression tests and compared with literature data. The schematic of the test setup is shown in figure 5. A Physica MCR 301 Rheometer (Anton Paar, Ashland, VA) was used to apply vertical compressive loads to 500 μm tall microneedles. The rheometer tool was set to move at a constant velocity of 5 μm s⁻¹ throughout the tests, and force versus displacement data were obtained for analysis of the failure loads. After each experiment, the needles were visually observed to make sure they did not buckle. These tests were carried out on five individual microneedle samples made through the process shown in figure 1.

3.5. Fluid delivery into sample skin using the fabricated microneedles

The capability of the metallic microneedles for skin penetration was realized by applying a 500 μm tall microneedle to pig skin and then taking histology sections to investigate the penetration depth and skin surface damage. The capability of the microneedles for transdermal drug delivery was assessed through an injection of fluorescent beads into pig skin. For this purpose, a 500 μm tall microneedle was bonded to the tip of a conventional 1 ml syringe. After filling the syringe with a 0.01 wt% suspension of 2.28 μm fluorescent beads in water, the microneedle was pressed against the skin, and then a force of approximately 2 N was applied to the syringe plunger for 5 min. The skin surface near the injection site was then washed with water and dried with a wipe. A Nikon Eclipse C1 confocal microscope (Melville, NY) was then used to scan the distribution of the fluorescent beads inside the skin.

Pig skin was used for transdermal analysis due to its similarities to human skin in terms of skin layer thickness and elasticity, which makes it a suitable model for in vitro studies [47].

4. Results and discussion

4.1. Conductivity measurements

The resistivity of the polymer is shown in figure 6 as a function of CB content. This plot shows the change in the order of magnitude of the resistivity value as the CB% increases from 0 to 50%; this is consistent with previous works investigating resistivity for lower CB contents [33, 34, 48]. Similarly, the conductivity of the polymer, shown in figure 7, increases rapidly as the CB content passes 40 wt%. Although using the PMMA/CB composite with the higher conductivity could facilitate faster electroplating, using CB beyond 30% leads to cracks in the polymer film during the casting step. As the higher CB content makes the polymer more brittle, the cast films become more sensitive to internal stresses that develop during the evaporation process. Using CB above 30 wt% creates cracks mostly around the pillar bases and around the edges of the mold in areas of high thickness variations. The 30 wt%
CB concentration, therefore, was chosen in this process to prevent crack formation while providing a sufficiently high conductivity for electroplating.

4.2. Dry etching of the PMMA/CB layer

The plasma etching rates of the PMMA/CB composite are shown in figure 8 for different gas flow settings. It has been previously shown that the addition of fluorine-containing gases can lead to an increase in the oxygen atom density in the plasma and therefore enhance the polymer etching rates up to a certain fluorine concentration [45]. In figure 8, the etch rate peaks for a CF4 flow rate approaching 20%, which is consistent with the peak locations for some other polymers [45]. The 20% CF4 flow rate has then been used for the microneedle fabrication.

The plasma etching step could potentially be eliminated from the fabrication process if the PMMA/CB solution concentration was kept below a certain value for specific pillar heights. It was seen during the experiments that for low solution concentrations (less than 5 wt% total solid concentration) the polymer would not cover the pillar tips and the solution does not wet the pillars entirely. This could be due to a moving contact line of the polymer solution during the evaporation process, which occurs for lower polymer concentrations or taller pillars. The resulting electroplated layer would be expected to be uniform on the base plate and metal would not cover the pillars tips.

4.3. Nickel electroplating process

The thickness of the nickel layer over time is shown in figure 9, for a constant current source of 2 mA, and a cathode area of $1 \times 1 \text{ cm}^2$ positioned 2.5 cm from the nickel anode. In figure 10, the nickel thickness is shown for different current source settings and a deposition time of 90 min with the same electrode size and orientation as the previous experiment. The change in nickel thickness is linear in both cases within the range of the tested parameters. The nickel deposition rate of $0.49 \mu \text{m min}^{-1}$ at 2 mA was found from the slope of the linear fit in figure 9, and the thickness as a function of current of $21.9 \mu \text{m mA}^{-1}$ over 90 min was found from the linear fit in figure 10, yielding a current-dependent deposition rate of $0.24 \mu \text{m mA}^{-1} \text{min}^{-1}$.

The recorded thickness values correspond to the thickness of the microneedle array backing plates and not the microneedle structures themselves; as seen in figure 4(a), the thickness of the nickel at the microneedle tip is much
The thickness of electroplated nickel on a PMMA/CB layer over time, with 2 mA supply current. The slope of the linear trend line is 0.49 \( \mu \text{m min}^{-1} \).

Figure 10. Thickness of electroplated nickel as a function of power source current, for a process duration of 90 min. The slope of the linear trend line is 21.9 \( \mu \text{m mA}^{-1} \).

less than that of the 70 \( \mu \text{m} \) backing plate, which shows that the Ni coating is not isotropic. This could be due to the orientation of the mold pillars as well as the lower conductivity of the polymer at the pillar tips due to decreased thickness. Since the pillars’ orientation in the electrolyte is perpendicular to the anode electrode plane, there could be less Ni\(^{2+} \) accumulation on their tips due to the perpendicular movement of the charged particles to the microneedle plane. In addition, the lower conductivity at the pillar tips could lead to lower current density in those regions which results in a slower metal coating at the beginning of the electroplating process.

4.4. Mechanical compression tests

Figure 11 shows an example of a force versus displacement measurement used to determine the microneedle failure load.

In this figure, the first peak in the force graph corresponds to the tip failure under compressive loading. From the five compression tests, the average failure load was 4.2 \( \pm \) 0.61 N. A plot with all measurements is provided in the supplemental material. In a previous study by Davis et al. [49], the authors have studied the fracture force of hollow metallic microneedles with respect to the needle tip radius, wall thickness, and wall angle. For this purpose, the authors have compared the experimentally obtained fracture force data with an analytical solution estimating the force, as well as a finite element model. The analytical model assumes thin-shell needles, which requires the ratio of the tip radius to the wall thickness to be larger than ten. This model is therefore not applicable to the needles presented in this work, since this ratio is much smaller. The finite element analysis was also found to underestimate the fracture force due to a conservative failure criterion of the needle material. However, the experimental data in [49] for needles with a similar material, deposition method and height
as in this work can be compared with the experimental results obtained here.

It was found in [49] that the fracture force is insensitive to the tip radius for radii ranging from 40 μm to 65 μm; the fracture force increases significantly with wall thickness from 4 μm to 15 μm and slightly with wall angle from 60° to 70°. Extrapolating the data in [49] following these trends would lead to a fracture force above 5 N for the needle geometry in this work (tip radius: 28 μm, wall thickness: 15 μm, wall angle: 80°), which is slightly higher than the measured 4.2 ± 0.61 N. This slight difference in strength can be a result of a different nickel nanocrystalline structure formed during the electrodeposition [42].

In addition to the fracture force measurements in [49], the forces required for the penetration of microneedles into human skin have been investigated experimentally for microneedles with different tip diameters. According to this study, the forces required for tip diameters of less than 50 μm is below 1 N; the failure load here is well above this force indicating that the needle will most likely not break upon insertion into skin.

4.5. Transdermal fluid delivery into sample skin

The histology image in figure 12 shows the skin surface damage caused by the microneedle penetration. The confocal scan of the injection site, shown in figure 13, indicates delivery of the fluorescent beads to a depth of 250 μm into the pig skin, which shows the usefulness the microneedles for transdermal delivery of drugs including suspensions. To verify that the skin is not permeable to the fluorescent beads without the aid of the microneedles, a series of control tests were performed by applying the fluorescent beads solution to skin surface for 10 min and then scanning the skin after washing the surface. The control tests did not show any fluorescent microspheres penetration below the skin surface.

5. Conclusions

A new fabrication process was demonstrated for making metallic microneedles. The process uses a conductive polymer as the seed layer for the electrodeposition of metal. Open microneedle tips are achieved through a single plasma etching step prior to deposition of the metal layer. This process can potentially allow fabrication of microneedles with a wide range of dimensions and spacing. This process can also be used for batch fabrication of microneedles, since large arrays of hollow needles can be formed without the need of opening the individual needles’ tips, a commonly used process that is labor intensive and requires methods such as laser micromachining. In addition, the molds can be reused for multiple fabrication runs which would substantially reduce the materials and labor costs for mass production. The molds in this work, for instance, were used up to three times without seeing any degradation of the needle shape and structure.

PMMA filled with CB has been used as the conductive polymer layer during the microneedle preparation. The conductivity of this polymer composite was characterized to find the optimum CB content which was 30 wt%. Also, from a series of O₂–CF₄ plasma tests, a 20% CF₄ flow rate composition was found to result in the fastest etch rate for the PMMA/CB composite with 30% CB. The thickness of electroplated nickel was found to be linear with respect to both process time as well as supply current.

Through mechanical compression tests, the average strength of the fabricated microneedles was found to be above 4 N, showing that these needles can pierce through human skin without breakage. In addition, the usefulness of the microneedle devices was demonstrated by successful delivery of 2.28 μm fluorescent microspheres into pig skin to a depth of about 250 μm, using 500 μm long microneedles.

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