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Introducing electric field fabrication: A method of additive manufacturing via liquid dielectrophoresis

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Abstract

Biomedical devices with millimeter and micron-scaled features have been a promising approach to single-cell analysis, diagnostics, and fundamental biological and chemical studies. These devices, however, have not been able to fully embrace the advantages of additive manufacturing (AM) that offers quick prototypes and complexities not achievable via traditional 2D fabrication techniques (e.g., soft lithography). This slow adoption of AM can be attributed in part to limited material selection, resolution, and inability to easily integrate components mid-print. Here, we present the feasibility of using liquid dielectrophoresis to manipulate and shape a droplet of build material, paired with subsequent curing and stacking, to generate 3D parts. This Electric Field Fabrication (EFF) technique is an additive manufacturing method that offers advantages such as new printable materials and mixed-media parts without post-assembly for biomedical applications.

Keywords

Microfluidics; Polymers; Dielectrophoresis; Field-assisted printing; Electrokinetics

1. Introduction

Microfluidic devices, including those used for mixing and separation, droplet generation for drug development, and organ-on-a-chip applications, have been a promising method for understanding basic sciences phenomena, *in vitro* diagnostics, and drug efficacy testing to name just a few applications [1–4]. These devices and the research surrounding them, like many other industries, have been advantageously affected by the potential of additive

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Declaration of Competing Interest

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Supplementary materials

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manufacturing (AM) [5]. AM is a process that develops 3D objects via material deposition layer-by-layer. The term itself encompasses a variety of methods to print 3D objects from computer-aided design (CAD) software or 3D scanner.

AM processes are already extensively used to develop new micro- and millimeter-scaled devices for a variety of applications [6–9]. For most researchers, AM provides an attractive manufacturing method and fast prototyping capabilities that could be far superior to traditional methods of manufacturing these devices [10]. These devices are traditionally made via soft lithography, that is the casting of polydimethylsiloxane (PDMS) from a silicon master. PDMS is a transparent, inexpensive, and biocompatible polymer that has become almost synonymous with biomedical devices. Soft lithography, however, though high in resolution, requires highly-trained personnel and access to and cost of a clean-room to develop the master via a time-intensive process [11]. Further, alignment of multiple layers of PDMS can be difficult and quite errant for more complex designs [12].

Not wanting to sacrifice the well-characterized materials and resolution that soft lithography or commercially available devices affords them, researchers in the microfluidic device industry have been slow to fully adopt AM processes as the primary method of fabricating these devices. Several AM processes, however, are common for direct device or mold fabrication for casting as an alternative method. Stereolithography (SLA) and digital light processing (DLP) are among the most common for microfluidic devices due to their high-resolution for small-scale features. These processes, however, rely on photosensitive materials; thus, limiting their material selection [13]. When used for making molds for soft lithography, the resins used in these processes result in leachates in the PDMS aqueous solution and could potentially be toxic for sensitive cells types or disrupt chemical reactions [14].

Along with uncertain materials, microfluidic devices that have embedded sensors, heaters, membranes, and valving are difficult to print with common methods such as SLA and DLP because they do not allow for the print to be paused to add a component [5]. In light of the advantages of AM but the challenges with current capabilities, we hypothesize that direct manipulation of the shape of the build material via electric fields can overcome some of the challenges of vat photopolymerization methods and provide rapid prototyping capabilities to microfluidic researchers in familiar, inexpensive and well-characterized materials.

Other studies have shown the use of field-assisted 3D-printing, referring to microstructure alignment due to electric, magnetic, or acoustic field exposure to closely control the microstructure of a part [15, 16]. Most relevant to the method presented here, electric field-assisted (EFA) AM was introduced to artificially fabricate biomimetic structures that give organisms their unique strength. EFA uniquely modifies the microstructure of polymer composites, or polymers encasing a reinforcing filler such as graphene nanoplatelets or carbon nanotubes, to optimize the strength of the build material [17, 18]. These methods however, are not suitable for all biomedical applications that do not require nor benefit from such fillers. Another method, electrohydrodynamic jetting, uses electric fields to overcome the surface tension of a liquid build material, essentially drawing the material out of the nozzle to decrease clogging. Paired with electrostatic deflection, Liashenko *et al.* showed

that electric fields can be used to improve feature resolution, print speed, and decrease nozzle clogging occurrences [17]. Capacitance edge effect, another EFA method, has also been used in biomedical applications such as printing hydrogels [19]. Non-uniform electric field lines along an asymmetric parallel capacitor, in junction with stacking, can produce hydrogel scaffolds [19]. This method specializes in fine line geometries along the capacitor edge rather than covering an entire area.

Here, we present an EFA technique we termed, Electric Field Fabrication (EFF), that uses liquid dielectrophoresis, or the polarization of dielectric liquids via non-uniform electric fields, to manipulate and shape an entire layer at a time. This technique mobilizes a droplet of liquid into the desired shape of that layer, is cured, and stacked on the inverted build plate until the 3D structure is complete. Using a droplet rather than a vat of liquid, this platform can be used to embed components such as sensors or membranes mid-print. Contrary to other EFA methods, the electric field in this context is used to manipulate a low-conductivity polymer without added reinforcements or clogged nozzles. Further, unlike SLA or DLP, the resin used to create a part via EFF is not restricted by a single curing method (i.e. photosensitivity), but the platform is amenable to a variety of curing sources, expanding its material selection to those that can be thermally-cured. This proof-of-concept study reviews the mechanism of droplet manipulation and shaping, introduces the platform and printing protocol, and discusses the advantages, drawbacks, and potential applications of EFF. In addition to the platform description and evaluation, preliminary parts to validate the feasibility of EFF include a microfluidic channel, a PDMS membrane, and a millimeter-scaled gear.

2. Theory

Liquid dielectrophoresis (LDEP) is the polarization of a dielectric liquid in response to a non-uniform electric field. When in the presence of an electric field gradient, a droplet of dielectric liquid will be pulled into the areas of highest field gradient magnitude. An interdigitated electrode array (IDE) of a conductive material will draw dielectric liquid from a droplet into a finger that travels along the length of the electrode when energized. This manipulation of the droplet and formation of a liquid finger along the electrodes can be modeled as a force balance of viscous and capillary forces counteracting the dielectrophoretic force. Other researchers have previously used dielectrophoresis (DEP) to manipulate fluids on chip, particularly to modify liquid-liquid interfaces [20–25]. The theory presented here for LDEP was first proposed by Jones and is summarized as follows [26, 27]:

When the field is off, the droplet remains sessile (Fig. 1A). Once the electrodes are energized, however, the force balance acting on the droplet can be described as:

$$\frac{\pi\rho r^2}{2} \frac{d}{dt} \left[Z \frac{dZ}{dt} \right] = F_{DEP} - F_{\mu} - F_{\gamma} \quad (1)$$

where ρ is the density of the liquid, r is the radius of the droplet, Z is the distance traveled by the finger as it is pulled along the electrodes, F_{DEP} is the DEP force acting on the liquid, F_{μ} is the viscous force acting on the liquid, and F_{γ} is the capillary force of the droplet. When

the field is turned on, the sessile droplet (Fig. 1A) is mobilized to form a liquid finger along the electrode (Fig. 1B). When F_{DEP} overcomes F_{μ} and F_{γ} on an interdigitated electrode array, the deposited droplet can be shaped into the precise design of that array (Fig. 1C). The distance that each finger travels is a function of time (Fig. 1D) and the contributing parameters to the remaining theory are labeled in Fig. 1E. The DEP force is a function of the applied voltage and the capacitance as the liquid moves as a finger:

$$F_{DEP} = \frac{V^2}{2} \frac{dC}{dZ}$$

where V is the voltage and the capacitance, C , is described as

$$C(Z) = \frac{Z}{\frac{1}{\kappa_w c_{air}} + 2/c_d} + \frac{L-Z}{\frac{1}{c_{air}} + 2/c_d} + C_{drop} \quad (2)$$

where, κ_w is the relative permittivity of the liquid, c_{air} is the capacitance of the surrounding medium (in this case air), c_d is the capacitance of the dielectric layer, and C_{drop} is the capacitance of the electrodes covered by the droplet.

The viscous force, F_{μ} , defined as:

$$F_{\mu} = 2rZ\tau_{\mu} \quad (3)$$

where the shear stress, τ_{μ} is defined as:

$$\tau_{\mu} = \frac{D\mu}{r} \frac{dZ}{dt} \quad (4)$$

where τ_{μ} is the shear stress on the droplet as it is pulled along the electrode, μ is the viscosity of the liquid and D is a coefficient to be determined from the velocity profile.

The capillary force is a function of droplet radius, r , and surface tension, γ :

$$F_{\gamma} = \pi r \gamma \quad (5)$$

3. Materials and methods

3.1. Interdigitated electrode array fabrication

The desired IDE pattern is etched via laser ablation from a glass slide coated in indium tin oxide (ITO), a transparent electrical conductor. The electrode spacing is 100 μm . A 25 μm layer of fluorinated ethylene propylene (FEP), an omniphobic dielectric, is treated on one side to promote successful bonding of the dielectric to the IDE via Henkel LOCTITE 3104 UV curable resin (Westlake, OH). While other conductors may certainly be used for the IDE, a transparent conductor offers unique advantages such as ease of fabrication and

visualization of the printing process via microscopy and is also cost effective compared to precious metals such as gold or platinum. Alternative to ITO, a copper IDE for thermally-cured build materials, such as PDMS, is fabricated using standard printed circuit board (PCB) etching of a PCB substrate.

3.2. Platform description

The platform can be described according to its components and their functions. The IDE sits on the stage face-up. Beneath the stage and the IDE is a UV light source for curing the photosensitive resin, Henkel LOCTITE 3104 UV curable resin (Westlake, OH). The IDE is energized by a Teledyne T3AFG40 function generator (Thousand Oaks, CA), an Advanced Energy Ultravolt 2C24-P250-I5 power supply (Ronkonkoma, NY), a CREE CGD15FB45P1 Six Channel SiC MOSFET Driver (Durham, NC) and a CREE CCS050M12CM2 SiC MOSFET Power Module (Durham, NC). This setup supplies a square waveform at a frequency of 17 kHz and maximum voltage of 2 kV. An inverted build plate is positioned above the stage and is lowered onto the IDE while the layer cures.

3.3. Printing procedure

The printing process for EFF from material deposition to 3D structure is depicted in Fig. 2. First, a droplet of liquid build material is dispensed directly onto the electrode array via a pipette. The electrodes are energized at a voltage magnitude of 2 kV. In response, the DEP force overcomes the viscous and capillary forces of the droplet and the liquid moves along the electrode array and takes the shape of the IDE. The inverted build plate, previously suspended above the electrode array, is lowered onto the shaped, uncured layer. The build plate and the IDE substrate sandwich the manipulated liquid layer while it is cured. Because this platform manipulates the build material based on its electrical properties rather than its curing properties, the curing method is adapt-able to the build material (heat or light) without requiring an entirely new platform. Once the layer is cured, the inverted build plate is raised, removing the cured layer from the electrode array. The release of the cured layer from the IDE is greatly aided by the omniphobic layer on the IDE but absent from the build plate. This process can then be repeated with a new droplet to create a 3D structure by stacking. Because a layer is not fully cured in the initial exposure and the UV penetration depth is greater than the layer thickness, the interfacial adhesion is high between layers. The final part is then removed from the inverted build plate using a sharp, thin edge such as a razor blade or a chisel. Parts made of PDMS can be peeled off the build plate. The full process can be seen in a video included in the supplementary information.

4. Results

Here, we show proof-of-concept devices to illustrate the feasibility of the EFF platform.

As seen in Table 1, the maximum build area of the EFF platform is 625mm² with 25 mm x 25 mm x 75 mm (LxWxH). The build area (LxW) is primarily limited by the allowable area in the IDE manufacturing process. As the size of the substrate containing the IDE changes, the build area will fluctuate proportionally. The maximum build height for this EFF platform is 75 mm and is set for easy observation of each layer as the build plate moves

away from the IDE. The platform has never been used to print a design that meets or exceeds its maximum build height, but the height available for parts is 75 mm. This build height, while not yet optimized for target applications, not only allows for the fabrication of large parts, but provides the potential to print directly onto another part, object, or component. The build speed reported in Table 1 is for Henkel LOCTITE 3104 UV that has a curing time of ~1 second. This build speed is inversely proportional to viscosity and required curing time of chosen build material. The layer thickness was measured via image analysis software (ImageJ) and is primarily a function of the initial droplet volume dispensed on the IDE before the field is energized. The layer thickness is subject to change following optimization steps for future studies.

Using the method described above, we were able to use the EFF platform to print parts with millimeter-scaled features. Fig. 3 shows the proof-of-concept parts fabricated using this platform, illustrating its feasibility and advantages. To fabricate a part on this platform, the process begins with an IDE of each layer. For the part shown in Fig. 3 A, this copper IDE (i) was used to shape the middle layer of the lidded, dogbone-shaped channel (ii) out of resin. A single copper IDE, as opposed to ITO, was used to prevent electrode degradation at high fields; thus, preserving the IDE for future or repetitive uses. The channel is unobstructed by support material or uncured resin (Fig. 3 Aiii)— a challenge not easily overcome using other AM techniques. This channel was fabricated by reusing a single substrate with three separate IDEs. For the first layer of the device, a droplet in contact with all three IDEs was placed and all IDEs were energized allowing the liquid to form the square bottom layer across both arrays. For the second layer, the same substrate was used, but a droplet was placed on either side of the dogbone-shaped array and only the two outer arrays were energized, leaving the center channel area clear of liquid. For the ceiling layer, another droplet was placed covering all three IDEs and after energizing all three arrays, the lid to the channel as formed.

For feasibility, the EFF platform was also used to print a gear, showing a more complex geometry and stacking of larger parts. This gear was formed using an ITO IDE but is shown next to its representative IDE for clarity in Fig. 3 B. For this part, the same IDE was reused for each layer to build the 2D design in the vertical dimension.

PDMS is essentially ubiquitous to microfluidic and lab-on-a-chip devices; some of these devices employ PDMS membranes or coatings for their desired application. As shown in Fig. 3 C, the EFF platform was used to fabricate a PDMS membrane on only a strategic portion of a glass slide, ready to be integrated or bonded to another device.

5. Discussion

5.1. Advantages of EFF

Electric Field Fabrication is a unique approach to creating 3D structures and differs from other techniques most notably by manipulation of the build material directly. While comparable to EFA, SLA, and DLP, EFF excels in areas that have yet to be attained by other techniques.

EFF allows for the printing of common materials such as epoxies, resins, and more novel materials to printing such as polymers, particularly PDMS. Contrary to other EFA methods that require a material with high electrical conductivity, EFF has been used to manipulate epoxy resin and even PDMS. The potential build materials could surpass the ones presented here following more investigation, but EFF simply requires that a material be a liquid, have dielectric properties, and the ability to be cured into its final state.

Unlike other methods relying on photosensitive materials, any method of curing appropriate for the chosen build material could be used; these include UV curing and thermal curing. Other studies seem to suggest that a similar mechanism, electrowetting on a dielectric (EWOD), could be used for manipulating conductive liquids, but this technique is not thoroughly discussed here [28]. Further, direct manipulation of PDMS for 3D printing reduces the cross-contamination of leachates from a resin mold [14].

Parts fabricated via EFF have notable layer adhesion fidelity and, consequently, low porosity- a common problem with other AM methods. Several commercially available AM techniques, such as material jetting, do not produce watertight parts [29–31]. With EFF, the bond between layers is formed when uncured build material is bonded to cured build material. For some applications, PDMS devices for example, using uncured build material is an accepted and widely used method to bond cured layers. This method, alternative to using an additional adhesive, produces water-resistant bonds between the layers comparable to multi-layer soft lithography devices.

Several single-cell analysis devices have embedded electrodes for impedance measurement, microfluidic systems often have valving and interfacing hardware, and organ-on-a-chip devices might include track-etched membranes, but these are difficult to include when a device is 3D printed. The most common methods of 3D printing these devices are, as mentioned, SLA and DLP. These prints take place in a reservoir of resin, making it difficult to integrate components mid-print. The integration of components while the part is being made reduces post-processing methods such as machining and assembly. EFF, however, because each layer is formed directly on the substrate after direct material deposition, the user can insert desired components hassle-free.

State-of-the-art high-resolution printing methods such as SLA or material jetting require extensive post-processing techniques from washing to baking to support material removal, or even machining to achieve the desired finish. These post-processing procedures often require entire machines dedicated to finishing the part such as high-pressure water jets, harsh chemicals with required containment, or machinery. Electric Field Fabrication, on the other hand, has virtually no post-processing requirements because each layer is fully cured as it is formed. Once the part is complete, the part can be removed from the inverted build platform and is immediately ready for use.

5.2. Drawbacks

While AM is typically known for its ability to fabricate rather complex structures not possible by traditional manufacturing methods, EFF caters to some geometries better than others. EFF excels in creating continuous layers rather than isolated structures within a

single layer. This limits the number of parts that can be made from a single droplet. To create multiple parts on a single bed with a single dispensed droplet, the parts must either be connected via the IDE or multiple droplets would have to be deposited on individual IDE patterns. The need for continuous structures also restricts the complexity of the design possible with EFF. For example, micropillar arrays would be difficult to fabricate on this new platform, while channel and micro-gear patterns would produce ideal parts.

Unlike other methods, EFF does not currently house the capability to generate structures via support material. Depending on the application, however, this might not be a limitation. The omission of support material in this process allows for the fabrication of enclosed channels not easily fabricated by other methods. These channels, because they aren't filled with support material that needs to be removed via post-processing, can be used as-is after curing. Also, EFF still has the potential to fabricate parts with large overhangs. Because each layer is cured on a flat substrate before it is removed, it is effectively supported by the layers before it on the build plate and the IDE below it. While the lack of support material decreases the possible design complexity possible on this platform, it introduces and allows for a new complexity not yet well met by other AM processes.

For full and transparent evaluation of this platform, the time and energy required to prepare the platform to print may be extensive. If the layer pattern is not a repetition of the layer printed before (as with the gear in Fig. 3B), an additional etched IDE will need to be made for that layer. For repetitive layers, this is not a cumbersome issue; to fabricate a part where each layer is different, an IDE for each layer is required. This greatly increases the processing time from CAD to part. While this might be unwieldy, the combination of integrated circuit fabrication (IDE layers) and AM actually offers a combination of each technique's benefits —complexity and resolution with new materials. Build material, defined by the user, might also lead to longer print times. PDMS, for example, takes at least 15 min to cure at 150°C, while photosensitive resins might take a few seconds. This, while not an inherent quality of the printer, is something to consider before use.

With electrical manipulation of liquids, Joule heating is a possible concern of this platform. The Joule heating generated with the manipulation of each layer leads to more rapid degradation of the IDE, particularly those made with ITO as opposed to metal electrodes. It can be observed that with fields that generate extensive temperature increases as a result of Joule heating, the ITO electrodes begin to flake off the substrate. While this does not immediately affect the performance of this platform, future build materials that are sensitive to high temperatures might cure prematurely or incorrectly. It will also increase the price per part by virtue of replacing the degraded IDE. In some instances, this might be the desired method of curing allowing one to forego the use of an external heating element for thermally-cured resins. In other instances, the material properties might be altered undesirably due to heat exposure. In the future, this problem can be abated by applying different coatings to stabilize and protect the electrodes from Joule heating.

5.3. Applications

Electric Field Fabrication brings the capabilities of AM to fields such as microelectromechanical systems (MEMs), micromachines and gears, and microfluidic

medical devices that are not readily well-served by current techniques. These devices often require high-resolution features, multiple components, and sometimes biocompatible materials. The devices are traditionally made using integrated circuit batch processing and/or soft lithography. Due to the nature of this fabrication method, most devices have been limited to 2D structures, halting innovation. Further, researchers within these fields have been hesitant to adopt 3D printing due to lack of familiar and/or comparable materials to PDMS [5]. Electric Field Fabrication offers these fields specializing in micro-fabrication and biocompatible applications the luxury of AM processes by directly printing PDMS.

6. Conclusion

Electric Field Fabrication is a method of AM that employs liquid dielectrophoresis, an electrokinetic technique, to manipulate bulk liquid build material to generate 3D structures. Here, we have introduced the feasibility of a platform that allows for AM with new materials for biomedical applications. Future work will be primarily focused on technique and platform characterization and, subsequently, optimization. Significantly, this method opens the door for new and relevant materials, such as PDMS, for 3D structures offering a new revolution of AM to researchers focused on medical applications. Electric Field Fabrication combines the resolution of traditional soft lithography techniques, already widely used in the field, with the innovation of AM not yet fully realized by other commercially available printers.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

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Data availability

Data will be made available on request.

Abbreviations:

EFF	Electric Field Fabrication
DEP	Dielectrophoresis
LDEP	Liquid Dielectrophoresis
SLA	Stereolithography
PDMS	Polydimethylsiloxane

AM	Additive Manufacturing
DLP	Digital Light Processing
CAD	Computer-aided Design
EFA	Electric Field Assisted
IDE	Interdigitated Electrode Array

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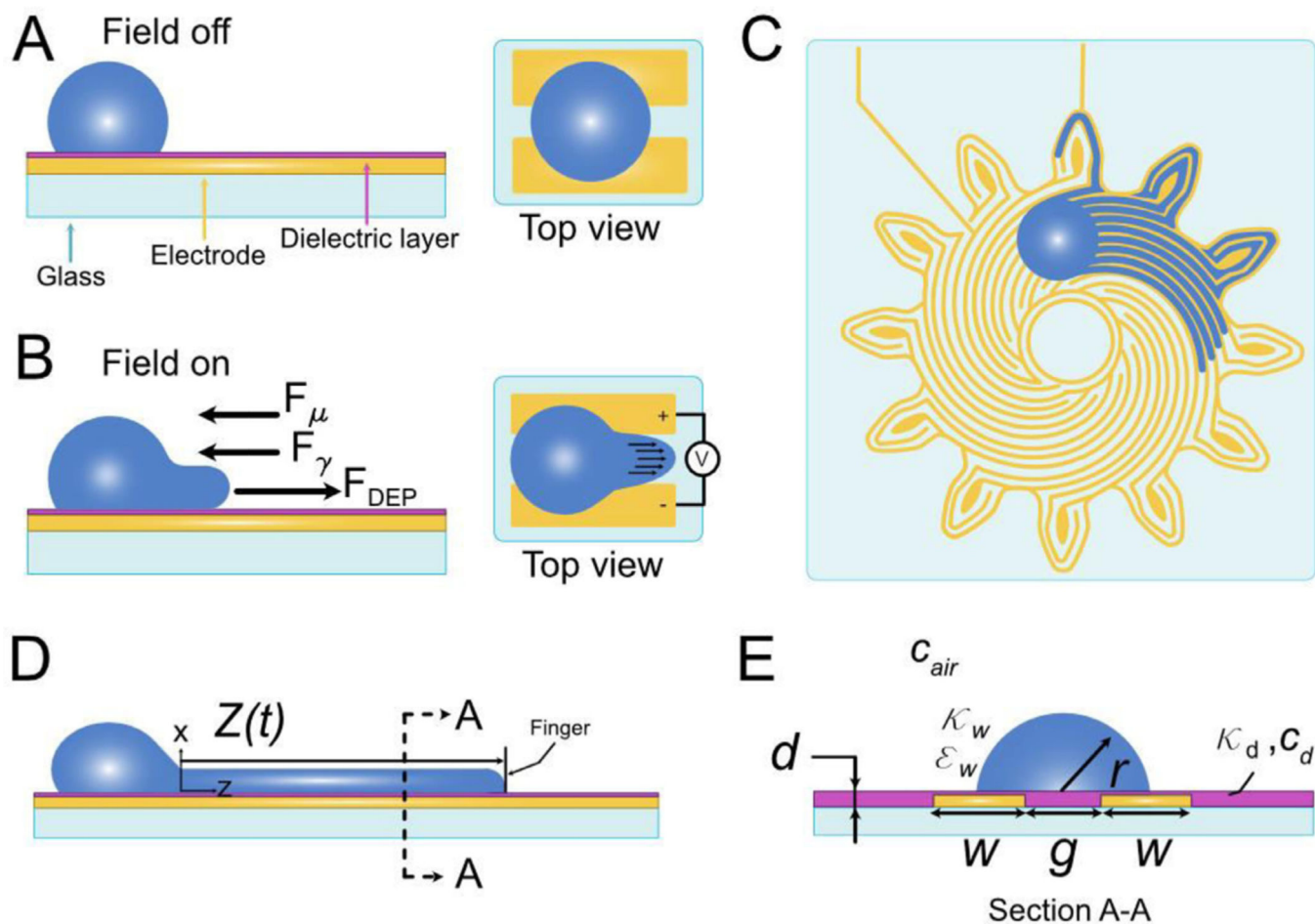


Fig. 1. Dielectrophoresis is used to manipulate and shape a droplet in the presence of a non-uniform electric field.

A) A sessile droplet on a non-energized electrode array. When the field is turned on, B) the dielectrophoretic force overcomes the viscous and capillary forces to drive the liquid along the electrode. In an interdigitated electrode array, C) multiple fingers are driven simultaneously forming a layer across the pattern of the electrodes to form a single layer, in this case: a gear. The distance traveled by the liquid D) is a function of time and E) the cross-section of the droplet defines the liquid and device properties.

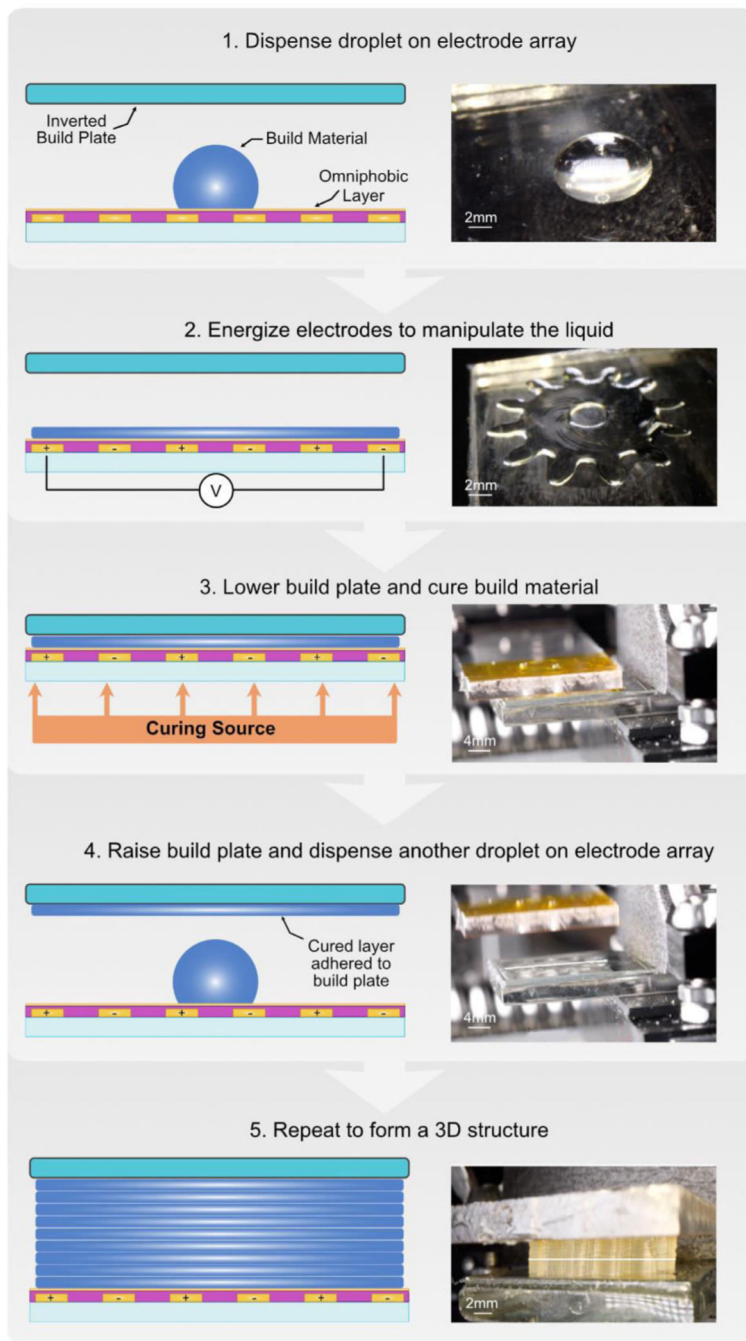


Fig. 2. Electric Field Fabrication printing process.

First, a liquid droplet is deposited on an IDE. The electrodes are energized to manipulate liquid along the IDE. The inverted build plate is lowered on top of the liquid and the layer is cured. The inverted build plate is then raised and a new droplet is deposited on the IDE. This process is repeated to build a 3D structure, layer-by-layer, as each layer adheres to the previous layer on the build plate.

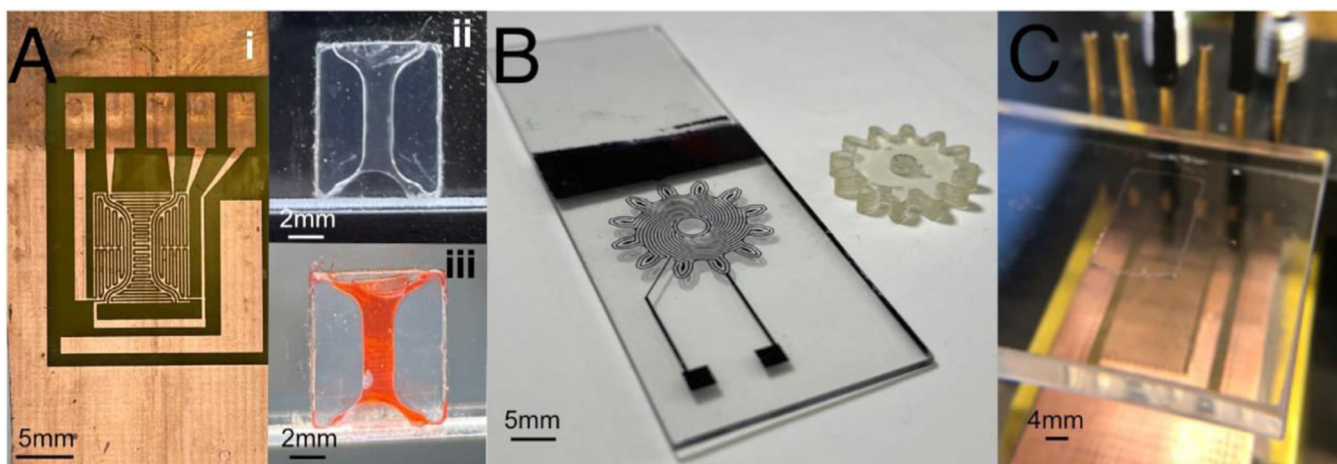


Fig. 3. Parts printed on the EFF platform.

A) A lidded dogbone-shaped flow channel i) copper IDE, ii) the top view of the resulting device, and iii) the channel filled with food coloring to illustrate the unobstructed channel. B) A representative IDE pattern and the resulting 3D resin gear. C) A strategically placed PDMS membrane that occupies only a portion of the glass slide to be interfaced with a copper coupon.

Table 1

Specifications of the EFF platform.

Platform Specifications	Value
Build Area	25mmx25mm
Maximum Build Height	75 mm
Build Speed	1–3 mins/layer *
Layer Thickness	100 μm (epoxy resin) **, 400 μm (PDMS) **

* This describes the entire process for a single layer including dispensing the droplet, moving the build plate, and curing. The variability results from the type of build material used.

** This has not been optimized.