

Additive manufacturing for nano-feature applications: Electrohydrodynamic printing as a next-generation enabling technology

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Abstract

Regardless of the technology, additive or subtractive, the miniaturization trend is constantly pushing for smaller resolutions. The rise of global challenges in material availability, fabrication in three dimensions (3D), design flexibility and rapid prototyping have pushed additive manufacturing (AM) into the spotlight. Addressing the miniaturization trend, AM has already successfully answered the challenges for microscale 3D fabrication. However, fabricating with nano resolution still presents a challenge. In this review, we will present some of the most reported AM-based technologies capable of nanoscale 3D fabrication addressing resolutions of ≤ 500 nm. The focus is placed on Electrohydrodynamic (EHD) printing (also known as e-jet printing), as EHD printing seems to have the best trade-off when it comes to technique complexity, achievable resolutions, material diversity and potential to scale-up throughput. An overview of the smallest achieved resolutions as well as the most unique use cases and demonstrated applications will be addressed in this work.

Introduction

Additive manufacturing (AM) is often referred to as one of the key enabling technologies capable of answering major challenges of tomorrow and according to the ECS report [1], by 2025 the AM market is expected to reach a value of €6.3 billion mainly based on the consumer electronics, automotive and aerospace industries. The most (well-known?) distributed AM technologies are for sure inkjet, aerosol printing, direct ink writing and laser-induced forward transfer (LIFT) just to name a few. The major advantages of AM are quite obvious: maskless fabrications, flexible and rapid prototyping, printing over 3D structured substrates, waste reduction and responsible material consumption. However, AM also faces disadvantages such as relatively low diversity of the printable materials, low printing speeds, low throughput (restricted printing size & small volumes) and relatively high electrical resistivity of the printed metal structures. Despite having all of those limitations, the advantages of AM are still deemed to be of great value for applied R&D and therefore are being intensively investigated and reported within the scientific community. The constant pursuit of Moore's law and consequently miniaturization are the main drivers for the development of novel next-generation AM technologies. The main goal put in front of them is the capability to deliver submicron resolution, with higher speeds, higher throughputs and a large portfolio of printable materials. Up to this date, the challenge of microscale fabrication has been pretty much answered and a variety of AM technologies for microscale 3D fabrication are reported

[2]–[6]. In our opinion, the challenge for the AM of nanoscale fabrication will be the next goal following the miniaturization pursuit. To our best knowledge, there are no reports available addressing the AM technologies for nanoscale 3D fabrication and therefore we have taken up the challenge of providing one. There is no exact definition of where nano labeling exactly starts and where it stops, therefore for this review we took the freedom and defined 500 nm resolution to be the upper limit for nano labeling. In addition, to identify the technologies, we will also engage in a detailed analysis of the most prominent technological representatives, focusing not only on the nanoscale fabrication aspect but also on the printed materials and demonstrated applications.

Additive Manufacturing Technologies for nanoscale metal structuring

As already stated, there are several comprehensive reports addressing the AM technologies for microscale 3D fabrication [2]–[6], therefore our work will build on them. By applying the self-defined nano criterion of ≤ 500 nm, several technologies are fulfilling this requirement such as: laser-induced photoreduction (also known as two-photon printing), meniscus-confined electroplating, electroplating of locally dispensed ions in liquid (fluidFM or SICM-configurations), focused electron/ion beam induced deposition (FEBID/FIBID) and electrohydrodynamic (EHD) printing.

A. Laser Induced Photoreduction

Here the metal salt solution is exposed to a laser source resulting in photochemical reduction at the laser focus spot and consequently printing of 3D-structures. The photocuring is typically driven by a combination of two photons (two-photon polymerization - 2PP). The major shortcoming is the limited possibility to print materials which in general do not support non-linear absorption, typically opaque materials. In the case of materials with a high refractive index, the resolution can be compromised and interaction between long wavelength radiation and coinage metals can result in the excitation of plasmonic modes causing the generation of bubbles and deformations [7].

B. Meniscus-confined Electroplating

In meniscus-confined electroplating, the nozzle is filled with a metal salt solution and is connected to a backside pressure regulator (typically a pump) to enable the excitation of the solution. The nozzle (or the metal salt solution) and the printing substrate are connected to an electrical power source (typically a direct current (DC) source). Once the metal salt solution is exited from the nozzle, at the interphase point of substrate and solution the electrochemical reduction will occur, resulting in 3D metal printing. Minimal resolutions of 100 nm have been reported by Hu et al. [8]. The major drawback of this technology is that the printing substrate needs to be conductive and that it is limited to conductive materials because of the necessity of current flow for deposition.

C. Electroplating of Locally Dispensed Ions in Liquid

This approach is quite similar to meniscus-confined electroplating. The add-on is the supportive electrolyte bath which is in this case connected to one end of the power supply (again typically DC), while the second end is connected to the metal salt solution. The same shortcoming as in the case of meniscus-confined electroplating is applicable. Minimal resolutions of 400nm have been reported. So far, only printing of metals (copper, platinum and copper-nickel alloy) has been reported. [9], [10]

D. Focused Electron/Ion Beam Induced Deposition (FEBID/FIBID)

In the case of the FEBID/FIBID, the precursor gas interacts with the electron/ion beam, resulting in the dissociation of the precursor and consequently 3D printing. The shortcoming of this technology is that the homogeneity of the gas distribution needs to be ensured, resulting in confined space and limited working volume. Processing of large areas or 3D structured substrates as well as simultaneous multi-material deposition is hardly possible with this technology. For Cryo-FEBID, all the above is applicable and additionally, the temperature in the print chamber must be held at -155 °C [11]. Minimal resolutions of 1-10nm and the deposition of a variety of single-metal and bi-metal combinations have been reported [12], [13]. Deposition of C, Si, Si₃N₄, SiO_x, TiO_x [14] as well as superconductors [15] are also reported.

E. EHD printing

Electrohydrodynamic (EHD) printing is a droplet-based technology developed from conventional inkjet printing. Instead of “pushing” the ink out of a nozzle, an electric field between the ink and substrate plate is generated. This electrical field forces charge carriers inside the ink towards the substrate and when the electrostatic force overcomes the surface tension of the liquid a Taylor cone is formed and nanodroplets or a cone-jet an order of magnitude smaller compared to the used nozzle diameter is ejected. EHD printing requires neither adapted process conditions (high temperature or vacuum) nor electrolytic baths or specially adapted precursor gases. For this reason, there is a high degree of process compatibility and yet even structures smaller than 100 nm can be fabricated with this technology. [16]–[24] Furthermore, the printability of many different materials such as metals, semiconductors, polymers, ceramics, carbon nanotubes and graphene has already been demonstrated. [25] In addition, EHD printing is capable of printing large areas due to the relatively high process speed. This process speed can be multiplied by the use of parallel glass capillary nozzles [26], [27], which also enables simultaneous multi-material printing [28]. In comparison, electrohydrodynamic printing is one of the most promising technologies for the realization of structures with resolutions <500 nm. For this reason, the following chapter summarizes the technological fundamentals, as well as the publications with the highest already achieved resolutions and special applications for the technology.

Electrohydrodynamic printing

A. Fundamentals

The additive manufacturing of structures with the electrohydrodynamic printing process is based on two different forces. The first consists of mechanical pressure applied to the ink by a pump to ensure that the nozzle (Fig. 1a) is filled, which is similar to traditional inkjet printing [29]. This pressure has to be adjusted for optimal high-resolution printability so that the ink is getting pushed to the orifice of the nozzle (Fig.

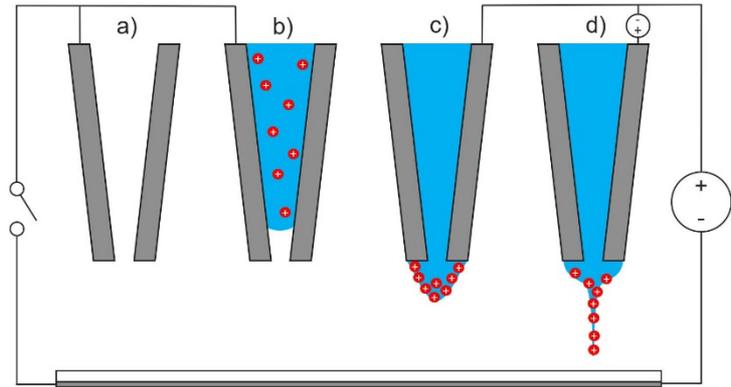


Fig. 1: simplified illustration of the electrohydrodynamic printing process, cross-sectional view of one nozzle a) empty b) back pressure applied c) Taylor-cone in equilibrium d) cone-jetting

1b)). In addition to this mechanical pressure, a voltage is applied between the conductive nozzle and the substrate during EHD printing. Due to the high electrical potential of the nozzle, charges are injected into the ink, resulting in the creation of ions in the liquid, which are forced toward the substrate by the applied electric field. This effect is called the injection phenomenon as also explained in more detail by Paillat et al. [30] and is referred back to the experiments of Michael Faraday [31]. The ions accumulate at the lowest point of the meniscus and exert tensile stress on the ink (Fig. 1c)). By exceeding a threshold value at which the electrostatic force becomes greater than the surface tension of the ink, a stream of ink called cone-jet (Fig. 1d)) is pulled from the tip of the meniscus and is accelerated to the substrate.

F. High-resolution EHD printing

In the last decade, electrohydrodynamic printing of critical dimensions in the nanometer range with many different materials has been published. Most publications describe the printing of organic materials because their properties are easier to tailor to the requirements of electrohydrodynamic printing. Cho et al. [32] printed 312 nm (polyvinylpyrrolidone)PVP-polymer lines with a 1 μm capillary nozzle on silicon. As an alternative method, they demonstrated subtractive printing of PMMA by jetting the solvent and produced masks for non-printable materials. They used this technique to fabricate a bottom-gate, top-contact thin film transistor without any photolithographic processing. The design of the transistor as well as its output characteristics are shown in Fig. 2a).

Another application for nano/micro-manufacturing of polymers is the fabrication of cellular structures for tissue engineering as showed by Zhang et al. [33] They used EHD-cone-jetting of PEO-PCL biopolymer lines on silicon down to 130 nm line width to build the scaffold for improved cell adhesion. In combination with the possibility of stacking several printing layers on top of each other, the production of the scaffold by EHD printing is a promising method for enhanced tissue regeneration. The deposition of DNA droplets with a minimum size of 100 nm has also already been demonstrated with electrohydrodynamic printing by Park et al. [34] as illustrated in Fig. 2b). They proposed that this technique can be applied to the alignment of DNA nanoparticles and the biosensing of RNA constituents.

Even the production of structures below 100nm has already been presented with the technology of electrohydrodynamic printing. Zou et al. [21] reported cone-jetting of 68 nm photoresist lines on gold with a 2 μm -inner-diameter capillary. Their work demonstrated the capability to use EHD printing as an alternative approach for the rapid additive manufacturing of etch masks. Another approach to fabricate masks for etching by EHD-printing is shown by Su et al. [23]. They conducted a study on the dependence of the electrical signal characteristics such as bias voltage, pulse amplitude, duty cycle and pulse frequency on the droplet diameter. With their optimized process parameters, they demonstrated cone-jetting of 90 nm NOA-61 UV-curable adhesive droplets and PEO lines widths down to 30 nm as shown in Fig. 2c). Furthermore, self-aligning materials such as PS-b-PMMA block-copolymers are promising materials to reduce the resolution of printed structures even further as reported by Onses et al. [22]. They used a 1 μm capillary nozzle to print lines with 800 nm width, but thermal annealing results in the self-assembly of the polymer to even smaller structures of around 50 nm.

G. Unique Applications of EHD printing

The technology of electrohydrodynamic printing can also be utilized for diverse applications. Kim et al. [35] demonstrated cone-jetting of semiconductor particles dissolved in dichlorobenzene.

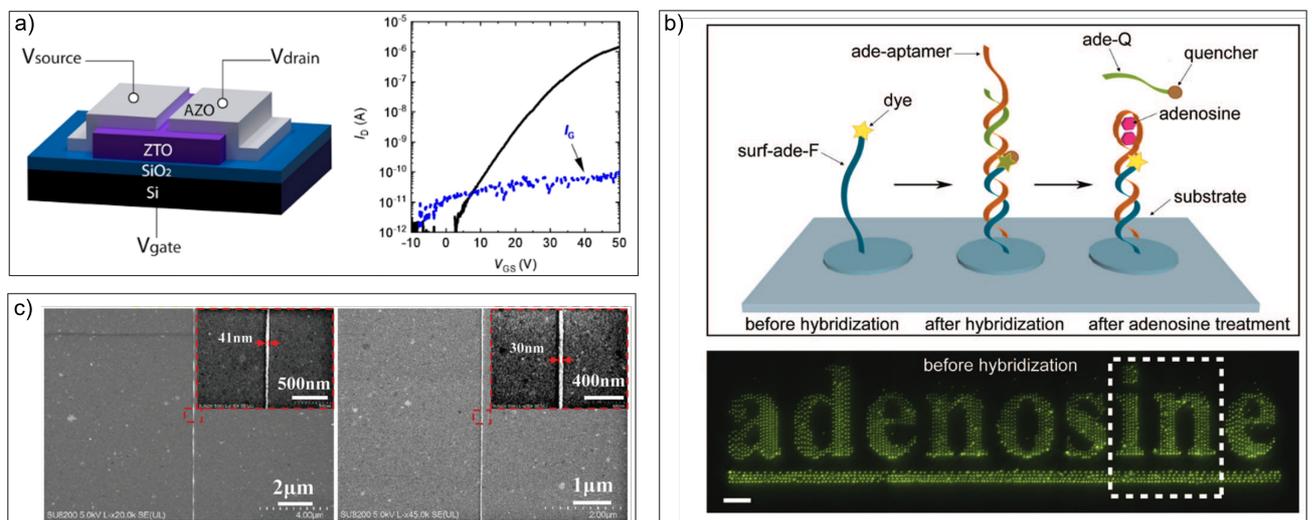


Fig. 2: EHD-printing of organic materials a) utilization of the EHD-printed PVP line as etch-mask for a photolithography free TFT fabrication. Reproduced with permission. [32] Copyright 2020, American Chemical Society b) EHD-printing of DNA droplets for biosensing applications. Reproduced with permission [34] Copyright 2008, American Chemical Society c) high-resolution PEO lines fabricated by microtip focused EHD-printing. Reproduced with permission [23] Copyright 2009, Royal Society of Chemistry

They printed lines down to 410 nm for the potential fabrication of micro-LEDs on a glass substrate. Another different approach to traditional EHD printing is reported by Wang et al. [24]. They reported cone-jetting 40nm PZT nanowires by using an outer high viscous silicone solution to stabilize the inner functional ink. After completing the printing, the abundant silicone had to be removed. That is the reason why this process is only semi-additive and can't be scaled to high throughput, but it is a possible way to fabricate PZT structures for nanoscale piezoelectric applications. In addition, the printing of metallic structures by nanoparticle suspensions has been demonstrated. Lee et al. [36] presented the EHD-printing of a silver nanowire tip (height: $\sim 4.5 \mu\text{m}$, body diameter: $\sim 150 \text{ nm}$) on an AFM-cantilever for deep trench profile measurements as illustrated in Fig. 3a). They

reported the simplicity and flexibility of fabricating different measurement tip sizes by adjusting the substrate-nozzle-distance printing process parameters. Furthermore, Schneider et al. [19] and Rohner et al. [37] reported the nanoscale fabrication of conductive nanoparticle materials by printing silver and gold grids and walls with a line width down to 80 nm as illustrated in Fig. 3b). By applying an AC voltage charge agglomeration on the borosilicate substrates can be prevented to fabricate conductive and transparent grids exceeding the optoelectronic performance of conventional ITO. Rohner et al. investigated the electrical properties of the gold nanoparticles and showed that the resistivity of the printed nanowalls was only 2.5 times that of bulk gold. Additionally, Schneider et al. [18] demonstrated the printing of 50 nm thick 3D-gold scaffolds on cyclic olefin copolymer with a potential biomedical application in monitoring cancer cell migration. Galliker et al. [17] reported the fabrication of 60 nm gold lines and 75 nm diameter gold nanopillars. These pillars are adjustable in their angle and show great potential for the development of nanoantennas.

Summary

This article summarizes the additive manufacturing technologies capable of fabricating structures $\leq 500 \text{ nm}$ and briefly describes their process-related characteristics. The following section describes the EHD printing process and presents the possibilities of this technology. It is shown that a wide range of organic and inorganic materials can be processed and that by optimizing the parameters

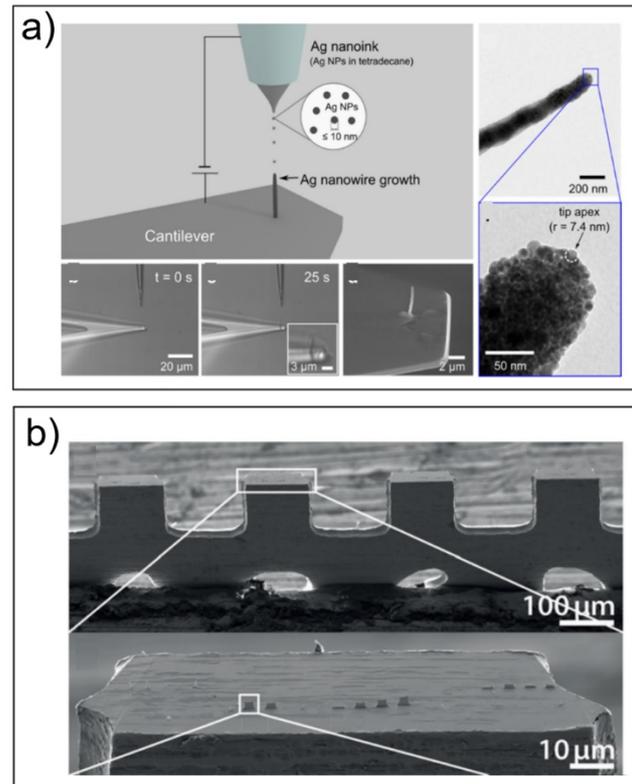


Fig. 3: EHD-printing of inorganic materials a) EHD-printing of silver nanoparticle pillars on cantilevers for AFM-measurements. Reproduced with permission [36] Copyright 2020, American Chemical Society b) fabrication of 100nm wide gold walls with 2.5 times resistivity of bulk

and the ink, structures with resolutions even below 100 nm can be manufactured. Due to the unique properties of the technology, even simple 3D structures such as free-standing pillars or nanowalls can be fabricated without intermediate treatments (curing/drying).

However, EHD printing is also facing limitations that prevent the technology from being utilized for industrial applications. On the one hand, all the systems presented are equipped with only a single nozzle, which means that throughput is very slow. The use of glass capillary nozzles prevents the technology from reaching industrial scalability since the electric fields of several glass nozzles would mutually interfere with each other and hence negatively affect the printing process. In addition, the electric field is generated between the top of the process table and the ink. The use of non-planar substrates or substrates composed of different materials also leads to a change in the electric field, which ultimately affects the generation of droplets or the cone jet and causes process instabilities.

Nevertheless, electrohydrodynamic printing has decisive advantages compared to other technologies for the additive manufacturing of sub-micrometer structures. For EHD printing no electrolytic baths or a special pre-treatment of substrates are necessary, which enables a high variability of processable substrates e.g. also with biodegradable and biocompatible properties and allows straightforward integration into microfabrication process lines. Furthermore, no prefabricated carrier substrates or precisely controlled process environments (vacuum chambers, process gases, high temperatures, etc.) are required. These aspects distinguish the EHD method from the other processes reviewed by Hirt et al. [3], which is why electrohydrodynamic printing is currently one of the most promising technologies for the additive manufacturing of features in the nanometer range. **This article is based on the authors' in-depth review of the topic, for further information and reference please see [38].**

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