

Printing Methods to Fabricate Receptor Layers of Gas Sensors

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Contributor: Victor Sysoev , Nikolay Simonenko , Nikita Fisenko , Fedor Fedorov , Tatiana Simonenko , Artem Mokrushin , Elizaveta Simonenko , Ghenadii Korotcenkov ,

Printing technologies are nowadays an integral element of contemporary materials science applied to development of low-cost gas sensors and multisensor arrays for many applications including a development of lab-on-chips. These protocols ensure automating of technological processes, a reproducibility of microstructural and functional characteristics with a reduced time necessary for the receptor material deposition over substrates. At the same time, using an accurate positioning system improves significantly the targeting of the substance, while the dosing setups allow to ensure a high control over the volume of discretely or continuously applied inks. The printing technologies enable forming planar receptor structures, even under a complex geometry, at various thicknesses and porosity with the required spatial resolution to be in nanometer micrometer ranges. Some methods, as dip-pen nanolithography, nano-imprinting lithography, and microcontact printing, are more suitable for discrete miniature devices with unique characteristics owing to the labor-intensive and multi-step procedures, while other ones, as ink-jet printing, aerosol jet printing or microextrusion printing, can be used quite easily in scaling the procedures to design gas sensors, including a rapid tuning of their geometric parameters without a necessity to prepare appropriate stencils and masks in advance. While designing the gas-sensor receptor materials, a great variety of printing technologies are used these days which vary both in the principle of operation and in such parameters as printing speed, spatial resolution, thickness of the formed coatings, and their microstructure, etc.

gas sensor

additive manufacturing

deposition

multisensor array

lab-on-chip

printing

1. Introduction

One of the major characteristics of a gas sensor is its sensitivity. This parameter matures by several factors which can be formally separated into two groups ^[1]. The first group includes microstructural properties—shape, size of particles, and porosity of the material ^[2]. In dense layers, the interaction with analytes of interest takes place almost exclusively on the surface, while in the porous layer, it occurs in the bulk of gas-sensitive material. The second group envelops characteristics of the chemical composition of the functional layer—the type of doping, stoichiometry, composition of sensing layer, etc. ^[3]. This approach suggests that the sensitivity of sensors is controlled not only by the morphology of the receptor gas-sensitive layers but also by the chemical composition of the surface which affects the concentration and type of surface defects as well as the catalytic activity of the surface ^[4]. All these parameters are usually defined by receptor, transduction, and utility functions, as originally

described by Yamazoe [5]. This means that the technology employed to manufacture the sensors must be able to deposit gas-sensitive layers in the necessary composition with the required surface properties. In other words, the composition of the ink utilized in the printing process should be selected in such a way that, on the one hand, it promotes the formation of a porous gas-sensing matrix, and, on the other hand, it does not contain components that reduce a surface activity. The process to form a gas-sensitive layer should also consider a minimum number of post-printing treatments to supply the applied material with the properties of interest.

To build multisensor arrays, gas sensors with different gas-sensing characteristics are ordinarily required, which is achieved by varying the layer thickness, particle size, and composition of the deposited semiconductor layer [6]. As a rule, the solution of this problem goes via taking a number of discrete sensors manufactured in frames of various technologies [7]. In order to reach the higher gas discrimination, the number of fabricated sensors in the array is usually rather high; for example, E. Baldwin et al. [8] employed 26 different sensors for this purpose. As a result, the measuring gas-sensor unit appears to be quite large which makes applying these devices rather difficult in currently developed miniaturized techniques. It is possible to solve this problem only if there are technologies which allow people to design the entire number sensors over the smallest possible area [9]. This means that the manufacturing technology must provide the possibility to reduce sensors to the given size, frequently at submillimeter or micrometer range. Reducing power consumption through the use of micro hotplates and integrating gas sensors with silicon electronics also requires a reduction in sensor size. The optimal method should also provide the possibility of varying the parameters of the formed layer during the printing process. For example, be able to apply simultaneously or sequentially the materials of different composition.

2. Ink-Jet Printing

Ink-jet printing is a non-contact method for manufacturing many functional layers. It enables a programmable setting of the trajectory to follow for a functional ink on the substrate surface with the ability for rapid changes in the pattern unlike printing methods employing stamps or stencils. The ink-jet printing unit, i.e., printer, includes a cartridge (container) with ink, a printhead, and a control system for the position of the printhead (drives, step motors, etc.). Usually, small droplets, normally at a volume of ca. several picoliters, are jetted from the printhead based on various mechanisms, such as gravity, hydrostatic pressure, as well as piezoelectric, via applying electric and thermal actuators [10]. Ink-jet printing can be classified at two basic categories, continuous ink-jet printing (CIJ) and drop-on-demand (DOD). In the case of the CIJ approach, the liquid flow is generated by the application of hydrostatic pressure followed by transformation of the flow into the droplets under the action of surface tension. To design the required pattern, unwanted droplets are deflected by an electric field to be collected in a gutter. The DOD approach of droplet formation is realized with the help of the thermal, piezoelectric, and electrohydrodynamic protocols. The method generates separate drops to be applied precisely to defined places at the substrate. The parameters of the printed layers primarily depend on the characteristics of the printing unit, of functional inks and substrates [11][12]. One of the major problems associated with ink-jet printing is the possible appearance of so-called “coffee spots” which, when dried, lead to the formation of an inhomogeneous layer. One way to minimize the effect of the “coffee spot” is to maintain a high temperature of the substrate so that the solvent can quickly

evaporate prior to undergoing a hydrodynamic spreading of the surface [13][14]. Another solution is the addition of minor amounts of solvents with a high boiling point and a low surface tension to slow evaporation at the contact angle [15]. This technology seems to be quite promising to design electrochemical sensors [16][17][18], photodetectors, electronics [16][18][19][20], and alternative energy applications [17][21]. The advantages usually include a compatibility with biological materials, low cost, programmed control, relatively high spatial resolution down to 20–50 μm , relatively high speed, and performance. Still, this technology has a low printing speed due to the limited number of nozzles, therefore, possibility of clogging the nozzles with solid particles make the scalability of this technology not yet feasible. Ink-jet printing is the most popular method for the machine designing gas-sensor layers due to the widespread use of corresponding devices; it is enough to note household printers which are widely spread out. The composition of the manufactured coatings can vary greatly from noble metals to metal oxides, carbon nanostructures, etc.

The prepared sensor demonstrated quite good performance in forward to ethanol detection. Ink-jet printing is also quite effective to apply while employing inks to appear as solutions of necessary reagents or organometallic precursors. For example, nanosized thin films of TiO_2 –10% ZrO_2 composition developed using alcohol solutions of titanium and zirconium alkoxoacetylacetonates as inks were found to have a high reproducible sensor response to O_2 [22].

3. Aerosol Jet Printing

Aerosol printing is a direct non-contact method of fabrication of layers on various substrates including ones with curved surfaces. Aerosol printing enables one to reach the resolution of about 20 microns [23]. The mechanism of particle transfer is the following: Primarily, the functional ink is vaporized, after that, a carrier gas is injected capturing the ink vapors, then this mixture passes under pressure through the print head along with the sheath gas, usually N_2 , which prevents the mixture from interacting with the walls of the print head. The mixture is sprayed onto the substrate through a nozzle [24]. A branched technique, aerosol lithography, has been recently developed by W. Jung et al. [25], who demonstrated 3D printing of flexible metallic nanostructures with the size of elements around 100 nm. Various spirals, lettering, rings, columns, and hanging structures were printed on the Si/SiO_2 wafer. The gas flow rate through the shell (SHGFR), the flow rates of carrier gas (CGFR), the temperature of the substrate [26], and the printing speed were shown to influence the printing [27]. In the case of aerosol printing, nozzles are resistant to clogging unlike ink-jet printing systems. At the same time, the viscosity range for functional inks employed in frames of this technique is usually much greater, from 1 cP to 1000 cP for a pneumatic sprayer, and from 1 cP to 10 cP for an ultrasound sprayer, which allows one to use a larger range of inks and to more finely adjust the characteristics of the ink, and, so, the layer parameters [28]. One of the actual problems of aerosol printing is an excessive spraying, i.e., overspray (OS) [29], which results in inaccuracies during the printing. In particular, the relationship between OS, SHGFR, and CGFR has attracted reasonable attention.

Aerosol jet printing can be used to produce transparent electrodes for flexible electronics [30], carbon nanotubes [31], and photodetectors [32]. Due to a high productivity, rather high resolution of the formed objects, a large number of degrees of freedom for spatial moving of the printing nozzles, resistance of nozzles to clogging, as well as a

wide range of acceptable viscosity of the ink, this method is one of the most effective when designing gas sensors based on receptor materials of different chemical composition. In particular, the work of [24] considers the application of this technology in using a high-sensitive ammonia sensor with the response to be ca. 4.64% to 4.35 ppm and ca. 52.01% to 97.19 ppm of NH_3 . In this case, graphene was utilized as the receptor material, which was applied to the substrate surface by spraying an aqueous dispersion of graphene to form an aerosol. Arsenov et al. [33] showed that aerosol printing can also be effectively used in the fabrication of metal electrodes which are components of miniature gas sensors. For this purpose, dispersed systems based on Pt particles with an average size of about 100 nm, to be sprayed with a pneumatic atomizer, were employed as inks. The technique is also used for the development of gas sensors containing metal oxide layers as receptor components. In a recent work [34], it was reported that the denser, as compared to screen printing, thick-film structures formed by aerosol printing in the $\text{Y}_2\text{O}_3\text{-ZrO}_2$ system exhibit lower noise and high sensitivity to NO and NO_2 . Therefore, this method is highly versatile and, in dependence on the chemical composition of the receptor material, layer thickness, and printing conditions, makes it possible to fabricate chemical sensors sensitive to various analytes [24][35][36].

4. 3D Printing

3D printing always requires building a 3D model of an object and converting it to the STL format, which carries only information about the geometry of the surfaces of the three-dimensional model. Then, this file is processed using “slicers”, programs that “slice” the model (for example, Simplify3D, Cura, etc.), and data on the technical parameters of printing, as nozzle movement speed, nozzle temperature, table temperature, etc. After installing the entire set of parameters, the slicer outputs a G-code file written by a software language with a numerical programmed control [37]. The width of the lines and the resolution of 3D printing depends primarily on the diameter of the nozzle, on the characteristics of stepper motors installed in the printer, on the temperature of the nozzle and the table, as well as on the executable G-code.

Several different protocols have been developed to implement the 3D printing. In addition to stereolithographic printing (SLA), there are the fused deposition modeling (FDM) or fused filament fabrication method (FFF), “ink-jet” 3D printing (or, binder jetting, BJ), selective laser-sintering method (SLS), selective laser melting (SLM), electron beam melting (EBM), by light processing (digital light processing, DLP), and laminated object manufacturing (LOM). Additionally, 3D printing is frequently distinguished to classify into two groups as nozzle-based printing (or nozzle-based 3D printing), which includes, for example, functional ink printing, and light-based 3D writing which includes, for example, two-photon lithography and projection micro-stereolithography [38]. Since the printing protocols which involve a photopolymerization make it possible to achieve a high printing resolution, the extensive work is actively underway to create new materials—photoinitiators [39]. In general, 3D printing, is possibly the most popular method to manufacture gas sensors, similar to ink-jet printing. In particular, this method was effectively used to fabricate a composite sensing material based on Cu and Fe microparticles whose surface was subject to a partial oxidation as a result of additional heat treatment in air [40]. The coating fabricated by this protocol has been characterized as rather selective to acetone vapors to yield a 50% chemiresistive response to 100 ppm of the analyte. The 3D printing, under fused deposition modeling (FDM) protocol, was recently employed to design an

NH₃ gas sensor operated at room temperature [41]. A composite based on metallic Cu particles and polylactic acid (PLA) was made, which was further subjected to a high-temperature treatment to remove the organic component and to oxidize the copper in order to obtain a monoclinic CuO phase. The appeared framework structure of the material has ensured the sensor to have advanced sorption properties and, consequently, a high stability, sensitivity, and selectivity for ammonia detection at room temperature. The 3D printing has also employed other functional layers in electrochemical detectors [41], biosensors [42], photoelectrodes [43][44], photoresist printing [45], biomaterials, porous materials [46][47], flexible electronics [48], optoelectronics [49], production of drugs [50], and perovskite layers [51].

5. Microextrusion Printing

The microextrusion printers are very similar to conventional 3D ones and consist of a heating element, a slide print bed, step motors that allow moving along the X, Y, Z axes, and ink/paste supply system. In the microextrusion process, ink is squeezed out through the nozzle (or nozzles) pneumatically or mechanically using a piston or a screw. Unlike ink-jet printing, microextrusion applies micro-balls on the surface of the substrate. There are commercially available bioprinters utilizing this method, for example, Inkredible+ (CELLINK, Sweden), which is employed for biomedical applications [52].

The factors to define characteristics of the printed layers include the nozzle size, starting from 0.5 μm to correspond best for liquid ink printing up to 1540 μm when using pastes, ink viscosity in the 1–1 × 10⁸ mPa·s range, and printing speed, from 1 μm/s to 500 μm/s depending on ink type and printing task [53]. This technique has been shown to print gas sensors [54], biological tissues [55][56][57], transparent flexible force sensors employed in wearable sensors under water and in robotics [58], graphene supercapacitors on flexible substrates [59], solid-oxide fuel cells [60][61][62], solar cells [63], electrochemical capacitors [64], thin films and membranes [65]. It is worth noting that the microextrusion printing is still rarely used in the fabrication of inorganic materials [61][66] that limits its application. However, it has great prospects, particularly to form structures at thick-film architecture. In this way, microextrusion printing can be compared to screen printing in terms of its efficiency, although there is no need to prefabricate templates that correspond to the geometrical parameters of the target coatings.

These protocols normally enable a microdosing of ink in a specified area of the substrate, which can have a complex shape as shown, for instance, in one of the first works devoted to prototyping gas sensors with such a printing [54]. Using the hydrolytically active organometallic nickel compound as a precursor, the NiO nanosheets were obtained under hydrothermal conditions and used to prepare a paste-like ink. After applying the coating to the Pt/Al₂O₃/Pt chip surface, it has been shown that the layer has a sufficiently high porosity and the average NiO particle size is about 32 nm. In course of chemisensor measurements, it was found that the material exhibits a high sensor response in forward to detection of hydrogen sulfide. In particular, the response to 100 ppm H₂S for the prepared oxide coating was 9786%.

6. Pen Plotter Printing

Pen plotter printing started taking a lot of attention recently, which is supported by high publication activity. Usually, pen plotters are employed with a pen utilized to print the pattern. The pen is filled with inks and further installed in a special holder. Plotters move in the X and Y planes and are unable to move in the Z plane, giving a chance to obtain planar layers only. This technology allows one to apply layers of various geometries, and to fabricate the pattern using a special software to enable further multiple reproductions. The quality of the printed layers depends on the geometry of the tip of the pen, ink viscosity, substrate wettability, and application mode with particular speed and resolution [67]. The thickness of the single layer can be reduced down to 400 nm. Applications of the pen plotter printing method include fabrication of sensitive layers for gas sensors [68], electrodes for supercapacitors [67], microfluidics field [69], deposition of electrodes (AgNP and CNT) on paper substrates [70], and biosensors on paper substrate for diagnostics-related applications [71]. Despite its simplicity and the availability of equipment, this method is not often used in the fabrication of gas sensors today [68].

Nevertheless, pen plotter printing is a rather versatile approach, allowing one to use both true solutions of reagents or organometallic precursors and dispersions of nanoparticles in various solvents as inks.

Recently, this technique was applied to design thin-film receptor layers using alcohol solutions of hydrolytically active heteroligand complexes—indium and tin acetoacetylacetonates in order to obtain ITO films [68] or similar coordination compounds of cobalt, in the case of Co_3O_4 films' application [67], as inks. The gas sensor based on the printed ITO thin film demonstrated a high sensitivity to CO, down to 100 ppb concentration, and the sensor based on the printed Co_3O_4 thin film as a receptor component displayed a sensitivity to CO, down to 4 ppm, and NO_2 , down to 100 ppm, depending on the working temperature [67]. Thus, pen plotter printing is a rather convenient and affordable method for producing gas sensors based on thin-film structures of different chemical composition, including ones under a complex geometry.

7. Microplotter Printing

Microplotter printing technology is a fairly new method that has not been studied much yet. In the process of printing, microplotter moves the plotting unit, i.e., piezo element and glass capillary dispenser [72]. The piezoelectric element, in turn, helps to control the transfer rate of liquid to the surface of target; the gentle pumping of fluid to the surface occurs when the glass capillary dispenser is driven at frequencies in the range of 400–700 kHz. This method provides a high level of accuracy, both in the dosing of functional ink and the plotting, as well as enables a continuous printing, which yields a chance to obtain continuous lines and shapes. Frequently, the Sonoplot Microplotter II is employed for printing [72][73][74][75][76] which operates with inks at viscosity of 0–450 cP; the drop volume starts usually from 0.6 pL, and the resolution is about 5 μm .

Such a device, as the aforementioned, makes it easy to replace an ink without replacing a cartridge. The characteristics of the plotted layers depend mainly on the ink properties. For example, utilization of dispersions requires a control of the particle size and depends on the solvent, transition kinetics, and aging. Normally, major attention is paid to the viscosity and surface tension of inks. Glycerin, ethylene glycol, polyvinyl alcohol, and sodium carboxymethylcellulose are commonly added to adjust the ink's viscosity. Water and ethylene glycol are

often used to prepare inks based on particle dispersions. The thickness of a layer might be less than 100 nm [73]. The use of microplotter printing is possible in such fields as gas [77] and optical [76] sensors, biosensors [74], solid electrolytes [72], photocatalysts [75], and microlenses [78]. The technique is applied for metallization of the surface [79], as well as in flexible electronics [80][81], stretchable thin-film transistors and integrated logic circuits [82], RGB displays [83][84], and composite electrodes [85]. Nowadays, this method is not frequently used to design gas sensors. However, it is still promising because of a number of advantages [77]. For example, due to the possibility of a fast cleanup of the capillary dispenser from functional inks before filling it with inks of different composition, the microplotter printing is a very effective way to fabricate multisensor arrays such as combinatorial libraries of receptor components with different chemical composition over a single chip. For instance, using solutions of organometallic compounds as functional inks for microplotter printing, an array of 8 receptor semiconductor layers differing both in chemical composition (Mn_3O_4 , $\text{TiO}_2/\text{ZrO}_2$, $\text{CeO}_2/\text{ZrO}_2$, ZnO , TiO_2 , Cr_2O_3 , Co_3O_4 , SnO_2) and a conductivity type was formed on the surface of a multi-electrode chip [73]. This allowed the device to work in the mode of recognition of various analytes. At the same time, a sufficiently high resolution of printing makes it possible to implement the miniaturization of such devices. In addition to solutions, inks composed of dispersions of nanoparticles in various solvents can also be employed in these protocols. In particular, using a paste based on ZnO particles decorated with Pt nanoparticles, thick-film composites were obtained [86]. It was found that such a modification of zinc oxide particles with the noble metal reduces the sensitivity to NO_2 and CO but enhances it to benzene and hydrogen, which is explained by chemical and electronic sensibilization.

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