

Development of multiple-droplet drop-casting method for the fabrication of coatings and thin solid films

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Abstract Spray coating is a commercial and low-cost technique for the fabrication of large-area coatings and thin films, but it is a stochastic process that is hard to control, as far as the fabrication of thin coatings and solid films is concerned. On the other hand, drop-casting is a facile and more controllable coating technique than spray coating, but its application is limited to small-area thin solid films and coatings. The objective of this work is, therefore, to study the feasibility of impinging an array of droplets, rather than just one droplet, to fabricate polymeric and other solution-processed thin films with larger surface areas than those produced by conventional drop-casting. To this end, in this study, four droplets of poly(3,4-ethylenedioxythiophene)–polystyrene sulfonate (PEDOT:PSS) solution are released simultaneously and impinged on the four vertices of a square on a wettable solid surface to make a thin film. The effect of the substrate texture on the spreading and the film formation process is studied. As a novel idea, the substrate is excited by ultrasonic vibration to improve the droplet spreading and coalescence. It is shown that as time elapses, the impinged droplets successfully coalesce and make a thin film. Surface morphology and roughness of the resulting PEDOT:PSS thin solid films show that, except on the edges, the resulting thin solid films are uniform. This leads us to conclude that the application of equal-sized and equally-spaced multiple droplets released simultaneously and impinged on vibrating substrates could be considered as a new coating technique, which has some of the benefits of the spray coating, but it is much more controllable than spray coating.

Keywords Drop-casting, Multiple-droplet impact, Ultrasonic substrate vibration, PEDOT:PSS, Coatings, Thin solid films

Introduction

Coatings and thin solid films may be deposited from the vapor or liquid phases, depending on the nature of the precursors and the desired functionality and expectations of the resulting films. The vapor-phase methods, which are categorized under either physical or chemical vapor deposition routes, require a well-controlled atmosphere and are usually performed in a vacuum, using expensive equipment and energy intensive processes. Therefore, highly ordered and defect-free thin films and coatings are usually deposited from the vapor phase. This paper focuses on the deposition of materials that could form a liquid solution or a fine emulsion or ink at room temperature, such as organic materials. The liquid-phase methods for the deposition of solution-processed or colloidal mixtures are less expensive, but are also less controllable. Therefore, it is highly desirable to perform basic research and develop new ideas to make the solution-processed deposition methods more precise, reliable, and reproducible.¹

Solution-processed thin solid films and coatings are obtained by the drying of thin liquid films or liquid patches or islands (e.g., impinged droplets in spray coating). The thin liquid films or liquid patches are formed on the substrates based on two main methodologies. In slot-die coating, blade coating, spin coating, and other similar methods, a continuous thin liquid film is cast on the substrate. In contrast, in drop-casting, inkjet printing, and spray coating, the process is based on the release, impact, spreading and deposition of one or more droplets.² The impinged droplets may form a continuous liquid film before drying or may dry individually to make a thin solid film comprised of

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numerous liquid islands that may overlap with one another. The focus of this work is on the droplet-based deposition methods, owing to their touch-free nature, scalability, and the potential to make very thin coatings and films. Among the droplet-based methods, spray coating is the most facile, inexpensive, and rapid method, but it is a stochastic process and, therefore, it is hard to control the repeatability and uniformity of the spray-on films. Inkjet printing is more controllable, but more suitable for the fabrication of lines rather than films because of its low throughput. Drop-casting is another simple and low-cost deposition method for the fabrication of small-area films.³ Drop-casting relies on the release of large droplets with controlled sizes and momentum that spread and wet the surface upon impact; thus, drop-casting is more controllable than spray coating, although its application is limited to small-area films and coatings. Hence, this work focuses on investigating the feasibility of releasing multiple solution droplets simultaneously, instead of a single droplet, in an attempt to fabricate uniform thin films with larger areas than those obtained by conventional drop-casting. In a sense, this process is similar to inkjet printing and spray coating. Compared to inkjet printing, multiple-droplet drop-casting requires simpler and less expensive equipment, without the need for using inks with prescribed physical and rheological properties and chemical formulation and it can coat an area in a shorter time, and compared to spray coating, it is a more controllable process. We have studied the impact dynamics of a single PEDOT:PSS droplet and the process of thin film formation in a previous work.⁴ This work, therefore, focuses on the coating characteristics of the process, and not the droplet dynamics. In the following, the literature reports concerning the impact of multiple solution droplets on dry solid substrates are reviewed.

Depending on the liquid physical properties, droplet size and velocity upon impact, and therefore the magnitude of the nondimensional Re , We , and Oh numbers, as well as the surface roughness and energy, an impinged droplet on a dry surface may experience several phenomena, including splashing, spreading and deposition, receding, and rebounding.^{5–7} It has been shown that for ordinary liquids, if the parameter $Oh \cdot Re^{1.25}$ is small enough (<57.7), splashing is suppressed and an impinging droplet spreads and deposits on the surface.⁸ This is the mode associated with or ideal for the drop-casting process. While there are numerous studies on the impact of single droplets of a wide range of materials from pure liquids to solutions and molten metals, e.g., references (9–12) there are fewer studies on the impact of multiple droplets on a surface and their interaction and coalescence. Lee et al.¹³ studied the effect of the liquid viscosity on the coalescence of droplets to simulate the process of line printing. Roisman et al.¹⁴ performed experimental observations and theoretical modeling of impact of two droplets on a dry solid surface with various droplet spacing. They substanti-

ated that the mass, momentum, and energy conservation laws play pivotal roles in spreading and the formation of thin films. Ristenpart et al.¹⁵ investigated slow spreading of two sessile droplets on a wettable surface and their coalescence dynamics. Barnes et al.¹⁶ studied the crown interaction of two droplets successively impacting the substrate. Li et al.¹⁷ and Graham et al.¹⁸ experimentally investigated the coalescence dynamics of a falling droplet colliding with a sessile droplet. In another study, Dalili et al.¹⁹ studied the coalescence of highly viscous liquid droplets deposited sequentially on straight lines or square arrays. They showed that the initial droplet diameter, droplet spacing, and the impact We number affect the thickness of the deposit. Yang et al.²⁰ studied the oscillations and spreading dynamics of single and consecutively ejected droplets on substrates with different wettabilities. In another work, it was found that droplet deposition frequency, spacing, and evaporation time scale are the main factors that determine the morphology of the coalesced droplets.²¹ In a study pertinent to the present work, Sarojini et al.²² studied the impact and coalescence of PEDOT:PSS solution droplets produced by an inkjet printer head and corroborated that the solution concentration and properties and droplet spacing affect the spreading dynamics and the deposit pattern. Soltman and Subramanian²³ studied the effect of the droplet spacing, as well as the temperature on the morphology of the inkjet-printed lines, in an attempt to obtain uniform and high-resolution lines. It is known that a gradient in temperature and/or concentration in a spreading droplet may lead to the generation of internal motions, creating coffee ring and Marangoni effects.²⁴ Soltman and Subramanian²³ showed that an elevated temperature triggers stronger coffee ring effect, affecting the quality of the line. In this work, in order to suppress the coffee ring effect and increase the uniformity of the resulting thin solid films, all experiments were conducted at room temperature.

The above-mentioned works have considered the interaction of multiple droplets in which falling droplets impinge on sessile droplets. This is in fact to simulate and optimize the process of inkjet printing. To the best of our knowledge, there are few or no studies on the impingement and coalescence of multiple droplets that arrive at the substrate, simultaneously. Thus, in this work, we study the phenomenon of the thin film formation as a result of the simultaneous impaction of four droplets of PEDOT:PSS, arriving at the four vertices of a squared area of the substrate. This is simply to investigate the feasibility of multiple-droplet drop-casting as a viable coating method. To this end, the effect of the substrate roughness and texture is studied by using fluorine-doped tin oxide (FTO)-coated and bare glass substrates. Besides, in an attempt to improve the droplet spreading and film uniformity, the substrates are excited by ultrasonic vibration. This is because it has been shown that the imposed ultrasonic vibration improves droplet spread-

ing,⁴ as well as the uniformity and intactness of the resulting thin solid films and the performance of thin film photovoltaic devices deposited by drop-casting,³ spray coating,^{25–30} and spin coating.^{31–33} To explain the reason behind an improvement in the uniformity of the solution-processed thin films excited by a mild or low-amplitude ultrasonic vibration, and breakup of the films overexcited by large amplitude vibration,³⁴ Rahimzadeh and Eslamian³⁵ revisited the hydrodynamic theory of thin liquid film evolution and instability under excitation by vibration developed by others, e.g., references (36–38) and applied it to the liquid solutions and derived practical criterion for the film instability. They concluded that excitation by vibration, and in particular vertical vibration, tends to destabilize the liquid film; however, if the excitation is of low amplitude, and the liquid film could resist the perturbations, it will remain stable due to the very small perturbation growth rates; meanwhile, the created nanoscale motion, known also as microstreaming, in the liquid film has a mixing effect on the precursors, levels the film, and enhances the solvent evaporation rate,³⁹ all of which contribute to the formation of a uniform and homogenous thin solid films after complete drying. Therefore, in this work horizontal ultrasonic vibration, which has a less destabilizing effect,³⁵ is imposed on the substrates to enhance the coating characteristics. It is noted that besides the above-mentioned emerging application of ultrasonic vibration for the effective fabrication of solution-processed thin films, the positive effect of excitation of the bulk or thin films of liquids is widely employed in other applications, such as sonochemistry to synthesize powders,^{40,41} microfluidics to manipulate small droplets and thin liquid films,^{42,43} generation of microdroplets in ultrasonic atomization,⁴⁴ controlled crystal growth,⁴⁵ self-assembly of nanostructures⁴⁶ and thin films deposited in the vapor phase.^{47,48}

In this work, PEDOT:PSS is used as the model solution, due to its popularity and wide range of applications in thin film devices. In the rest of this paper, the deposition methodology of the films prepared by simultaneous arrival of four droplets on the nonvibrating and vibrating substrates is described, and the physical characteristics of the resulting thin solid films are studied. In the end, a scalable coating method and the device layout based on the idea of multiple-droplet drop-casting is proposed.

Materials and methods

Pristine PEDOT:PSS aqueous solution (Clevios PH1000, Heraeus, Germany) was further diluted with isopropyl alcohol (IPA, 99.7%) with a volume ratio of 1:2, respectively, to increase the spreading of the PEDOT:PSS droplets. The physical properties of the resulting solution are listed in Table 1.⁴ Glass-based substrates (50 mm × 50 mm × 2.5 mm) with two surface conditions were used in this work: FTO-coated glass with a roughness of 45.9 nm and bare glass with a roughness of 16.3 nm.⁴ Substrates were chemically and thermally treated based on the following procedure in order to remove the surface contaminations and to increase their surface energy and therefore their wettability. The substrates were washed by detergent, acetone, and deionized water in sequence, and dried in a vacuum furnace and cleaned in an ultraviolet cleaner. An ultrasonic transducer installed inside of a cubic metal box was used to generate horizontal vibration. The transducer was actuated by a signal generator operating at a frequency of 40 kHz and power of 10 W. The substrates were placed and secured atop the metal box so as to allow the transmission of the ultrasonic vibration to the substrates and the impinged droplets.

Four equal-sized droplets were released simultaneously using four micro syringes fixed on the vertices of a squared array from a distance of 150 mm above the substrate, with initial diameter of $0.55 \text{ cm} \pm 0.03$, as depicted in Fig. 1. In order to ensure simultaneous and complete drainage of the syringes, a rigid metal plate connecting the end of all four plungers was pressed down carefully and rapidly. Although the four droplets were released simultaneously, they impacted the substrate with delays of about a few milliseconds, which has a negligible effect on the deposition process. The nondimensional parameters *Re*, *We*, and *Oh* numbers of PEDOT:PSS droplets upon impact are listed in Table 1. Droplet center-to-center spacing was optimized and set to 12 mm to ensure that the impinged droplets coalesce and form a thin film as time elapses. High-speed images were taken using a color high-speed camera (FASTCAM SA5, Photron, Japan). The optical mode of a confocal laser scanning microscope (CLSM, model LMS700, Zeiss, Germany) was used to observe the surface morphology of the prepared deposits under 10× magnification. A 3D surface profilometer (KLA-Tencor P7, USA) was utilized to measure the roughness of the PEDOT:PSS deposits.

Table 1: Physical properties of PEDOT:PSS solution measured at 25°C, and the corresponding nondimensional numbers of PEDOT:PSS droplets for the conditions of the experiments of this work (impact height of 15 cm and initial diameter of about $0.55 \pm 0.03 \text{ cm}$)

Density (gr/cm ³)	1.59	<i>Re</i>	681
Surface tension (mN/m)	28.9	<i>We</i>	890
Shear viscosity (mPa S)	22.0	<i>Oh</i>	0.044
Contact angle on bare glass (°)	6.7	Contact angle on FTO-coated glass (°)	2.5

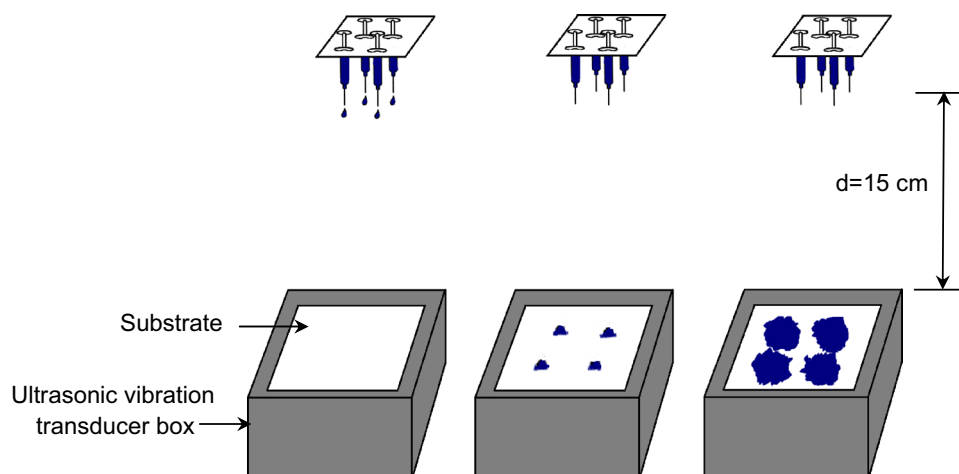


Fig. 1: Schematic of the experimental setup for simultaneous release of four equal-sized and equally spaced droplets

Although the substrates used in this work are $50\text{ mm} \times 50\text{ mm}$ in size, the resulting thin solid films excluding the edges were found most uniform in a squared area of $25\text{ mm} \times 25\text{ mm}$. The average roughness of each film was therefore measured on three lines of approximately 1 mm each, passing through the center of the above-mentioned squared films. ImageJ software was used to estimate the percentage of coverage of the films with respect to the substrate area.

Results and discussion

The parameter $Oh \cdot Re^{1.25}$ associated with the impinged PEDOT:PSS droplets of this study is ~ 152 , which is higher than 57.7 , the threshold for the transition from the splashing mode to the deposition mode, according to reference (8). However, owing to the high viscosity of the polymeric PEDOT:PSS solution, only minor splashing was observed, as substantiated by the images in Fig. 2, displayed after 30 ms from the time of the touchdown. Thus, it is safe to state that the droplets are in the deposition mode, suitable for the film formation. Figure 2 shows sequential images of the spreading and deposition of four simultaneously impinged PEDOT:PSS droplets on bare glass and FTO-coated glass substrates, with and without imposed vibration, in a prolonged time window of 60 s , and Fig. 3 shows the time evolution of the percentage of the surface coverage for each of those four cases. As elaborated in the introduction,⁵⁻⁷ an impinging droplet may undergo several dynamic phenomena within several milliseconds, owing to its momentum upon impact. At the end of the rapid dynamic stage, the droplet slows down and assumes a stable condition with an apparent contact angle. In this stage, the droplet behavior is governed by thermodynamics and surface science, rather than hydrodynamics. The images in Fig. 2 and curves in Fig. 3 are, therefore, associated with the aforementioned slow deposition process. As Table 1

shows, the contact angles of PEDOT:PSS droplets on bare glass and FTO-coated glass are small, and therefore, the substrates are of high surface energy and are partially wettable. On a partially wettable surface, the droplets may continue to spread^{49,50} after the initial impact stage, although very slowly, a phenomenon that Figs. 2 and 3 show for the four impinged droplets. The details of the dynamics of contact line motion, droplet bridging, and complete coalescence are beyond the scope of this work and have been studied by others for simple cases, e.g., reference (15). Nevertheless, Fig. 2 clearly shows the deposition and coalescence process of the four impinged droplets. Given that the droplet spacing is small enough, as time progresses, the four spreading droplets come in contact with one another and completely coalesce to form a film (Fig. 2), although the substrate texture and imposed vibration influence the merging time. Figure 2 shows that the droplets impinged on the FTO-coated glass substrate coalesce earlier than those impinged on the bare glass substrate. Another effect of the FTO-coated glass is the formation of finger-like patterns on the rims, as observed by others as well.⁵¹

In addition, Fig. 2 substantiates that the imposed vibration promotes the deposition speed and accelerates droplet coalescence. This is because the imposed vibration results in contact line depinning,⁵² in that the energy needed to overcome the energy barrier for contact line motion is partly furnished by vibration. The images taken after 10 s clearly show that at this time, only the droplets impinged on the vibrating and textured FTO substrate show a complete coalescence. In all cases, however, the four droplets finally coalesce and form a liquid film. When the droplets have already reached a small contact angle close to their equilibrium contact angle, they might be pinned to the substrate, and therefore, the continued fluid flow from the center toward the boundaries would result in the accumulation of the liquid in the rims, as observed in Fig. 2. This effect is amplified if the substrate is vibrating. Figure 3

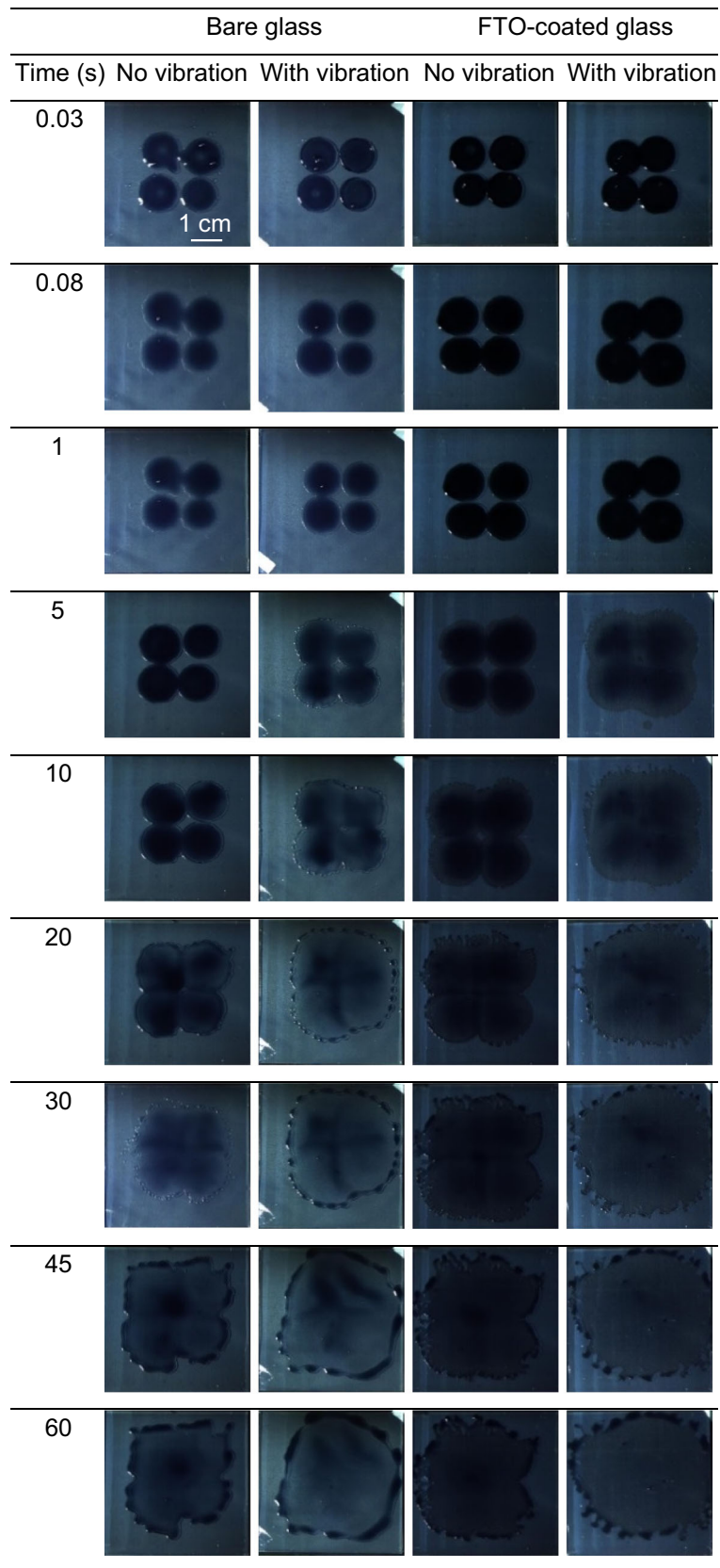


Fig. 2: Effect of the substrate vibration on spreading of PEDOT:PSS droplets on bare glass and FTO-coated glass, in a prolonged time window of deposition

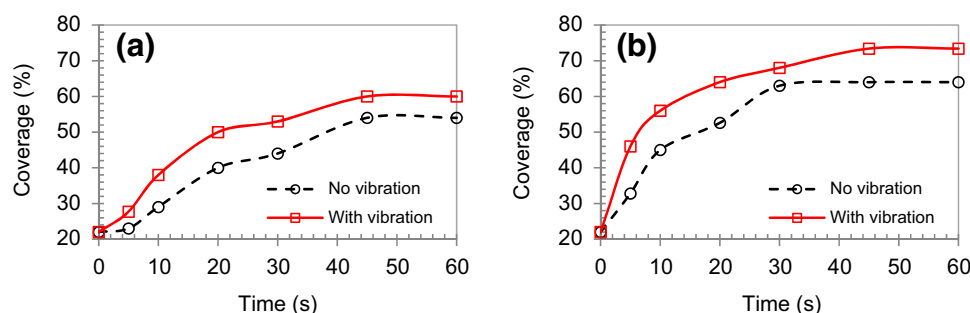


Fig. 3: Time evolution of the percentage of the substrate area covered (coverage) by four PEDOT:PSS droplets impinged on (a) bare glass substrates and (b) FTO-coated glass with and without the effect of imposed ultrasonic vibration on the substrate. Application of imposed vibration and FTO-coated glass promotes the film coverage

Table 2: Surface roughness of dried PEDOT:PSS drop-cast films and the associated standard deviations (SD)

Substrate	Vibration time (s)	Roughness ± SD (nm)
Bare glass	0	52.7 ± 21.5
Bare glass	1	43.6 ± 8.8
Bare glass	10	25.2 ± 7.5
Bare glass	30	22.15 ± 0.75
Bare glass	60	25.5 ± 4.2
FTO-coated glass	0	24.1 ± 1.3
FTO-coated glass	1	21.87 ± 6.9
FTO-coated glass	10	17.16 ± 1.2
FTO-coated glass	30	16.10 ± 1.3
FTO-coated glass	60	21.7 ± 6.7

confirms that the film coverage on the substrate increases in all cases as time elapses, and imposed vibration and employing FTO-coated glass improve the coverage. However, the rate of the increase in coverage due to spreading slows down with time according to the aforementioned discussion. Therefore, as far as obtaining a uniform film is concerned, there exists an optimum vibration time, beyond which the core of the film will become thinner, whereas the rims will become thicker while the droplets are pinned. Thus, the surface wettability and texture, as well as the time duration of vibration interplay a role in liquid accumulation in the rim. In other words, to eliminate the bulky rims and increase the film area, the surface wettability should be increased and the time of the imposed vibration should be controlled and limited.

Next we studied some of the characteristics of the PEDOT:PSS deposits, fabricated by drop-casting of four equal-sized impinged droplets after complete drying. Table 2 lists the roughness of the films fabricated on bare glass (smooth) and FTO-coated (rough) glass substrates, under various vibration times. Generally, the deposits made on the smooth bare glass substrates have a higher roughness. In other words, a smoother PEDOT:PSS deposit forms on the rougher

and textured FTO-coated glass. Considering that perturbation surface waves may form on the interface of the liquid film and air due to various effects, such as surface tension (capillary waves) and imposed vibration,³⁵ one may speculate that the textured surface tends to damp the instabilities, thus leading to leveling of the liquid surface and the formation of more uniform resulting solid film. The data in Table 2 show the effect of the vibration time on the roughness, as well. It has been shown in our previous works that imposing ultrasonic vibration could improve the film intactness and uniformity provided that the vibration time is below a threshold.^{31,34} Table 2 confirms the same trend, i.e., beyond 30 s of vibration, the film roughness increases again, because although the imposed vibration creates microstreaming or micro-scale motion that result in the film leveling, it also inherently tends to make the film unstable. Therefore, an excessively long or strong imposed vibration has a detrimental effect on the film uniformity. Figure 4 shows the optical microscope images of the same PEDOT:PSS thin solid films, where the influence of the imposed vibration and the choice of the substrate is demonstrated. The main images show the center part of the films, which contains the area where the moving

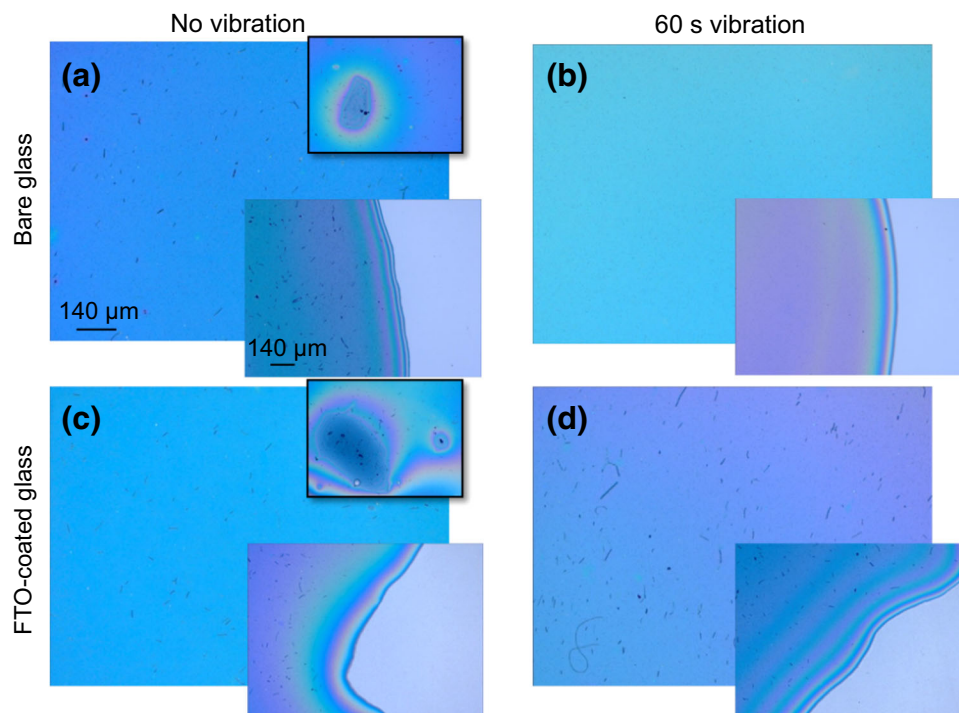


Fig. 4: Optical images of PEDOT:PSS thin solid films made by casting of four equal-sized and equally spaced droplets simultaneously impinged on the substrates: (a) on bare glass made without vibration; (b) on bare glass made using 60 s of vibration; (c) on FTO-coated glass made without vibration; (d) on FTO-coated glass made using 60 s of vibration. The main images show the center of the film, and the lower overlapped insets show the boundaries of the films. The top framed insets in (a) and (c) show some irregularities in the pristine films. (The field of view for all images is $1400\ \mu\text{m} \times 1000\ \mu\text{m}$)

contact lines of the four impinged droplets have met, and the lower overlapped inset images show the boundaries of the final solid film. The main images show no irregularities at the centers of the film, substantiating that the four droplets have coalesced effectively to form an integrated film. Some random defects or irregularities were detected on the nonvibrated or pristine films, as shown in the framed insets of Figs. 4a and 4c, which might have occurred during the drying process, due to various mechanisms of dewetting, such as heterogeneous nucleation dewetting,⁵³ presumably because of the presence of large agglomerated PEDOT:PSS particles that exist within the film (black dots and fibers), which may act as nucleation sites for dewetting. It is observed that the imposed vibration removes such defects because the fluid microstreaming and the mixing effect of the imposed vibration prevent particle agglomeration.

The successful fabrication of uniform thin films, excluding the edges, by impingement of four equally spaced and equal-sized droplets inspires us to put forward an idea for designing a coating device based on the principle of the simultaneous release and impingement of many equally spaced and equal-sized droplets. As Figs. 5a and 5b depict, such a device would be more effective than spray coating in which the droplets with

random size and position impinge on the substrate, making it hard to control the uniformity of the film. Compared to inkjet printing for the fabrication of thin films, the proposed device has a simpler design while it has a much higher throughput, and the anticipated device and ink price would be much less than that of the inkjet printer. Figure 5c illustrates a tentative design for the proposed device. The device would incorporate multiple holes with equal spacing made on a flat plate of a chamber, which would contain the liquid solution. The chamber would be connected to a syringe pump and step motor to control the liquid flow rate. Upon activation of the syringe pump, the liquid would be pushed through all orifices, leading to the ejection of one droplet from every orifice. The impingement and spreading of the multiple droplets would result in the formation of a thin liquid film. The substrate would be ultrasonically vibrated to improve the spreading, coalescence, and uniformity of the film.

Conclusions

In this work, the feasibility of using multiple-droplet drop-casting for the fabrication of thin solid films with larger areas than those obtained by the single-droplet

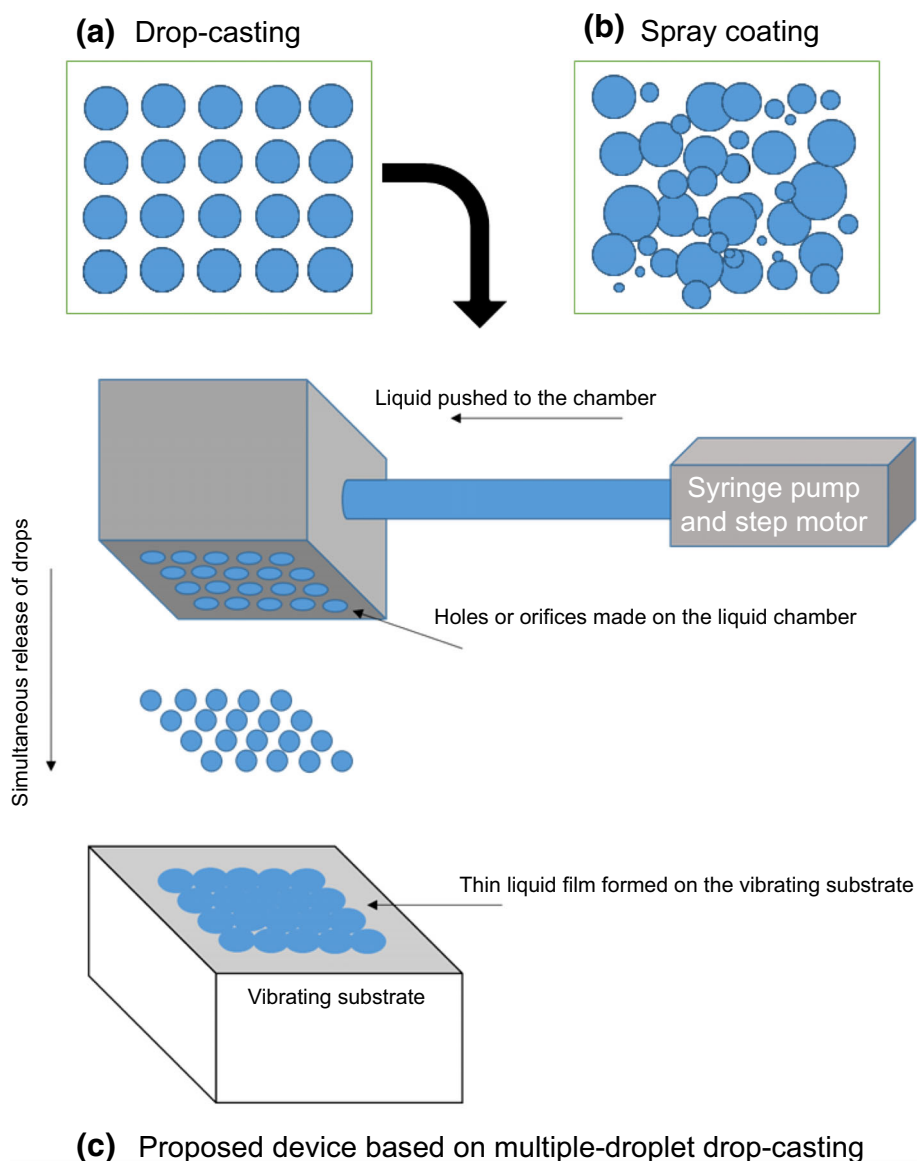


Fig. 5: (a) Pattern of the impinged droplets on the substrate in multiple-droplet drop-casting method to fabricate continuous thin films; (b) random droplet impingement in conventional spray coating; (c) tentative design of a device based on the multiple-droplet drop-casting concept

drop-casting was studied. To this end, the deposition of four identical PEDOT:PSS solution droplets, simultaneously released and impinged on the four vertices of a squared area of wettable smooth bare glass and textured FTO-coated glass substrates, was studied. In selected experiments the films were excited by horizontal ultrasonic vibration. Over a prolonged duration, it was found that PEDOT:PSS droplets spread out more and faster on vibrating FTO-coated glass substrate compared to the cases employing nonvibrating or bare glass substrate. In other words, the coalescence of the four droplets occurred earlier on FTO-coated glass excited by vibration and the entire film covered a

larger area of the substrate. Also, the imposed vibration resulted in a decrease in the film roughness and removal of the defects of the resulting thin solid films, provided that it was executed for a limited and optimum time. In summary, it was found that the drop-casting of multiple droplets assisted with ultrasonic vibration is a viable technique for the fabrication of thin solid films and has the potential to become a large scale coating method. A tentative schematic design of a coating device based on this idea was proposed. For some specialty applications, this method may be considered as an alternative technique to spray

coating, which is a stochastic process with limited controllability.

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