Journal of Diabetes Science and Technology
Volume 5, Issue 5, September 2011

SYMPOSIUM

Future Opportunities for Advancing Glucose Test Device Electronics

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Abstract

Advancements in the field of printed electronics can be applied to the field of diabetes testing. A brief history and some new developments in printed electronics components applicable to personal test devices, including circuitry, batteries, transmission devices, displays, and sensors, are presented. Low-cost, thin, and lightweight materials containing printed circuits with energy storage or harvest capability and reactive/display centers, made using new printing/imaging technologies, are ideal for incorporation into personal-use medical devices such as glucose test meters. Semicontinuous rotogravure printing, which utilizes flexible substrates and polymeric, metallic, and/or nano "ink" composite materials to effect rapidly produced, lower-cost printed electronics, is showing promise. Continuing research advancing substrate, "ink," and continuous processing development presents the opportunity for research collaboration with medical device designers.

J Diabetes Sci Technol 2011;5(5):1077-1086

Introduction

Opportunities often present themselves when new developments and advancements from traditionally different areas of focus come together. In the areas of portable glucose sensing and printed electronics, opportunities for electronic circuitry, energy storage/harvesting devices, and sensor design are relevant examples.

Predominant Technology in Glucose Meters/Sensors and Strips

Glucose sensors with digital display ("meters") have not significantly changed in basic design concept since the 1990s. Improvements have continued to be focused on consumer products' ease of sampling and smaller sample volume requirement features, software enhancements, and device compactness.[1]

The principle of glucose detection is still primarily based on the enzymatic reaction of glucose (in blood) with glucose oxidase, glucose dehydrogenase, or glucose quinoprotein glucose dehydrogenase. Enzymatic reaction products hydrogen peroxide or reduced form of nicotinamide adenine dinucleotide or the product pyrroloquinoline quinone (red), respectively, are then detected when combined with a suitable transducer. Amperometric detection of hydrogen peroxide after

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Abbreviations: (PET) polyethylene terephthalate, (pHEMA) poly-hydroxyethyl-methacrylate, (R2R) roll to roll

Keywords: flexible electronics, printed biosensors, printed electronics, printed sensing system, roll to roll, rotogravure

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enzymatic reaction of glucose with glucose dehydrogenase remains the primary method of measuring glucose in blood and has become the standard for clinical diagnostics.[1]

"Glucose strips" refer to the scaffold onto which the sample is placed (or contacted) for reaction. Typically this consists of glucose oxidase immobilized between two membrane layers, the outer layer being polycarbonate (preventing larger components of blood from passing but allowing glucose to pass through and then react with glucose oxidase) and the inner being cellulose acetate adjacent to a platinum electrode. The inner layer allows the hydrogen peroxide reaction product to pass but acts as a size exclusion barrier to other electroactive compounds that might be present. To help reduce the operating potential below that of other oxidizable sample components and thereby minimize interference signals, the addition of electron transfer compounds—known as "mediators" (e.g., ferrocene and derivatives)—can be used in the vicinity of the electrode.[2]

Although major limitations to implementing design changes are the industry's desire to maintain market share of existing products and the reluctance of patients to change established testing habits,[1] new innovations in *in vivo* biosensor technology have been tested. Design challenges in this area include minimizing device size, minimizing oxygen dependency, biocompatibility, and overall long-term stability.[3]

Traditional Electronics and Their Limitations

Sensor design typical of personal "glucose meters/strips" has traditionally relied on a reagent reaction at the sample surface affecting a change in potential at that point, which is then detected via transducer to wafer board electronic circuitry, resulting in numerical display readout. This requires a minimum sample amount, appropriate conditions for a reaction to occur (temperature/humidity), comparison of the product amount to a calibrated standard code, and step-time-measured display readout. Test-surface and sample-junction cleansing (rezeroing) are no longer concerns affecting the performance of the meter due to the advent of disposable test strips. Cost, reliability, stability, and life of the strip remain areas of interest.[1]

Electronic circuits are traditionally manufactured using processes that basically involve precise layering of components onto substrate wafer-type surfaces. As the push to reduce the size and weight of these wafers gained momentum for use in smaller and smaller devices, technologies involving thin film metals deposition, laser etching, and chemical etching of silicon wafers took hold. These technologies offer the major advantage of resultant high-precision circuitry but at a high fabrication cost due to the requirement for meticulous material clean controls and long stepwise processing times. In addition, the relatively larger amount of rare materials required per unit circuit is a growing cost concern.[4]

For biosensors, commercialization has been achieved through the use of screen printing (squeezing or forcing an "ink" through a patterned screen onto a solid support held on the reverse of the screen).[5] A major limitation, however, has been the range of "inks" available.[1] Screen printing deposits a thicker ink film than other print methods that takes longer to dry. Fine line printing is possible with line resolutions of 15 μm being reported. These "inks" are high in viscosity, so screen printing is mostly used to print materials where a thick ink layer is required for performance, (e.g., metallic conductors, electroluminescent materials, and some dielectrics).

Energy storage device design (e.g., batteries) has been an active field of research due mainly to the need to boost power delivery and storage in ever-smaller, lightweight devices. Over the years, the industry has advanced from acid-reservoir cells to dry-cell technologies, with lithium-based, solid-state technology that relies on porous substrates. Material (rare metals) cost, thicker functional layers (high volume/weight), and limited storage life are current limitations.[4] However, advancements in the efficiency and communication software for near-field wireless power may be an enabling technology on the near horizon. Fulton Innovations has shown the ability to efficiently power packaging displays with this technology.[6]

Energy-harvesting devices (e.g., photovoltaic cells) have evolved over the years for use in consumer products amenable to solar exposure for battery charging. Technology limitations include lack of low-temperature ambient stable semiconductor materials. Inefficient manufacture, complicated thin-layer production, and higher costs remain concerns.

Electronic and lighting displays have advanced from conductive wires to liquid crystals to light-emitting diodes. Light-emitting diodes are amenable for use in displays on glucose meters provided appropriate materials are established. Displays continue to be one of the

most active areas of printed electronic research where advancements have been made in energy efficiency and image quality.[7–9]

New Materials in Electronics Printing

The printing industry has historically concerned itself with printing media onto flexible substrates (e.g., paper, film, foil, and textiles) for graphics and communications. Printing has come a long way from depositing colored pigmented inks onto paper, with tremendous advances taking hold in the printing of electronic materials. Ink-jet, aerosol-jet,[9–11] screen, and rotogravure printing technologies are used alone or in combination with traditional fabrication methods to produce lower-cost electronic devices. New engraving and press technology has been developed that is capable of printing features comparable to current photolithography electronic fabrication methods.[12]

Research in smart packaging and graphics imaging by consumer product companies, driven by globally expanding markets and increased security-tracking needs, coincident with advances in optics electronics and solution-based electronic materials, has resulted in the application of those root ideas of paper printing to printing on new materials.

New flexible and durable materials can be printed with designs that can impart the physical function of an electronic circuit, store and/or harvest energy, and be coated with specific materials that impart specific detectable electronic changes on contact with a sample. Such printed materials are as lightweight as paper and are capable of being manufactured on high-speed presses.

Low-cost, thin, and lightweight materials containing printed circuits with energy storage or harvest capability and reactive/display centers, made using new printing/imaging technologies, are ideal for incorporation into personal-use medical devices, such as glucose test meters.[13]

Though relatively macro-sized components made by "thick film" screen printing have dominated the field since the early 1980s, micro components having full functional capacity, printed onto flexible substrates, is a new field of research. Further advancements in material substrates and "inks," incorporating new developments in nanotechnology and semicontinuous printing, present exciting opportunities for research collaboration with medical device designers.

Developments in Printed Electronics

Screen printing is now used to make microelectronic circuits and circuitry components, including storage and display devices. Energy storage capability and remote data transfer (e.g., electrical induction and radio telemetry) are also elements that can be incorporated into these printed electronics. New developments include multilayered circuit coils for powering remote sensor devices.[14]

Combined components ("hybrids") consist of flexible and functional printed electronic layers. These hybrids can perform multiple functions synergistically. For example, layers of bi-stable inorganic and organic elements (one being reflective and the other being active or passive in nature), used in concert with electroluminescent inks, can be engineered to display characters and images (e.g., "e-paper").[15] Laminated layers can also be fashioned to function in energy-storage photovoltaics or powerable coils or to act as sensors.

"Ink" development, in this context, development of fluids containing carbon, metals, and/or polymers that ultimately comprise printed electronic component structures, is now a flourishing area of research, especially with the emergence of new nanomaterials. A multitude of electronics inks are now available, with new formulations becoming applicable to larger-scale production. A materials properties registry is being established through a joint effort between the Center for the Advancement of Printed Electronics at Western Michigan University and FlexTechAlliance.[16] Different "inks" and layering/overlay schemes allow customization of designed circuits and electronic components. These overlays can additionally incorporate reaction sites, channels, or sample wells. New inks include intrinsically conducting polymers and nonconducting polymers, nanocomposites, and metal composites.[17–19]

Chemical laboratories on "chips"—integrated into biosensors—can contain multiple chemistries, enzyme reactions, and reagents. These can be fabricated in large arrays within printed electronics, provided extremely small sample volumes are effective and high sensitivity is achieved.[3] Combination of sample reaction site(s) with transducer electronics can result in such innovation as biosensor "patches." It is easy to envision the ideal flexible biosensor contact "patch" that samples, analyzes, and displays results all in one unit.[1]

Printable circuit coils within the layered structure allow for signal powering. Combined with implantable devices, such as insulin minipumps, remote detection capability (e.g., of glucose level) may be combined with treatment/dosing capability (e.g., providing insulin needed) via signaling semiconductive imbedded components.[14] Powering applications can be expanded if printed coils can be printed with finer, smoother lines.[20–22]

As components of devices become smaller, the electronics that power, respond to chemical/biochemical changes, and display/communicate results can become smaller also. This is what is achievable through printed electronics at the micro scale.

Research Opportunities for Advancing Printed Electronics Applicable to Glucose Testing Technology

The next step on the development horizon for these printed electronics is to incorporate continuous manufacturing technologies for *all* layers of materials in the construct to make products that are more conformable to the human body or clothing and to minimize cost and weight.

Adapting the printing of microelectronics to include sheetfed, rotogravure, and roll-to-roll (R2R) methodologies is a current area of research.[22] Manufacturing optimization of product and process variables (e.g., gravure cell geometry, press material compatibility, and "ink" application uniformity) and performance testing (in process and end product) are underway.

With the overarching goal of improving ease of use, performance, and affordability of personal testing devices, opportunities for collaborative research include the areas of manufacturing processing, sensor design and performance, and materials ("ink" or coating materials).[23]

Advancements in the Printing Process

Printing Sequence

The most economically suitable printing sequence (e.g., one process using multiple single prints or multiple prints using more than one process) for low-cost manufacture of printed electronics must be identified for the broadest set of functional materials. For researchers, this requires overcoming the limited availability of large-scale printing and coating facilities. Compared with relying on traditional, high-cost integrated circuit technologies (e.g., single layer printing combined with nonprinting deposition methods), sequence-printing processes require greater knowledge of printing process technologies.[23]

Sheetfed, Rotogravure, and Roll-to-Roll Processing

Process demands for sheetfed and R2R printing are vastly different than screen printing in terms of environmental stability, drying requirements, antiscratching, contamination sensitivity, pH and temperature control, electrostatic discharge, and registration control (i.e., print-to-print alignment specification tolerances).

Sheetfed printing involves passing individual sheets through a press. Sheetfed printing has the ability to maintain tight registration control, is easily off-line tested, and results in less material waste than R2R printing.

An example of a single station precision laboratory sheetfed press is shown in **Figure 1**. A sample of printed glass and fine lines printed on this press are shown in **Figure 2**. The press is designed to precisely print multiple layers.

Our research team is currently focusing on transitioning materials (nanometallic particles, conductive polymers, organic semiconducting materials, organic and inorganic dielectric materials) printed successfully using conventional print methods for printing to a sheetfed AccuPress 3 equipped with a Micro Gravure (<50 μm) engraved cylinder.[12] The ability to print <50 μm features on a high throughput sheetfed rotogravure press offers multiple advantages relative to photolithography: fewer processing steps, less waste, and cleaner processing.

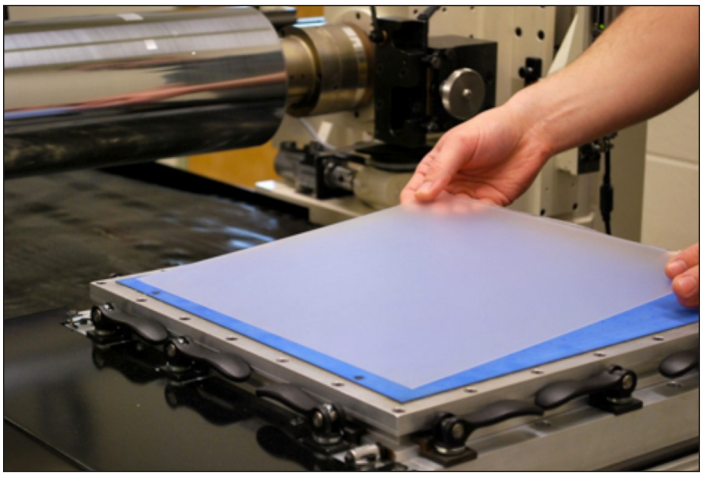


Figure 1. Loading of rigid substrate onto platen of precision sheetfed rotogravure press, which can be run with or without glove box enclosure in place. (Reproduced courtesy of Erika Rebrosova, Center for the Advancement of Printed Electronics.)

Rotogravure printing has the advantage of imparting high print resolution. The engraved chrome cylinder can withstand aggressive solvents. Geometries of the engraved cells can be altered to control ink film thickness and to accommodate inks of different viscosities, solids loadings, surface tensions, and particle sizes. **Figure 3** shows 5 μm cells engraved by Daetwyler R&D.[12]

Roll-to-roll printing presents many more control challenges. These include concerns with web tension control (which affects registration control), speed constraints for use of online characterization controls, material waste during webbing and startup of the press, scratching, and sheet breaks. Greater precision registration is required in web tensioning, control and imaging, and electrical signal capturing systems to align printed features online.

In order to fully evaluate R2R printing methodologies, qualification systems need to be established for all materials used and online metrology/inspection tools need to be developed for unique device attributes (e.g., uniformity, thickness, cell electrical testing, position check, and layer–layer registration) to allow for real-time monitoring and corrective action. It is preferable to use noncontact technologies. Until such tools are created, cost benefits of high-speed R2R printing will not be realized. In the interim, sheetfed printing provides the benefits of higher speed throughput with less waste and investment into higher-end online control systems.

Examples of offline characterization tools that have been developed to date, complete with integrated computer software packages, are given in **Table 1**.[15]

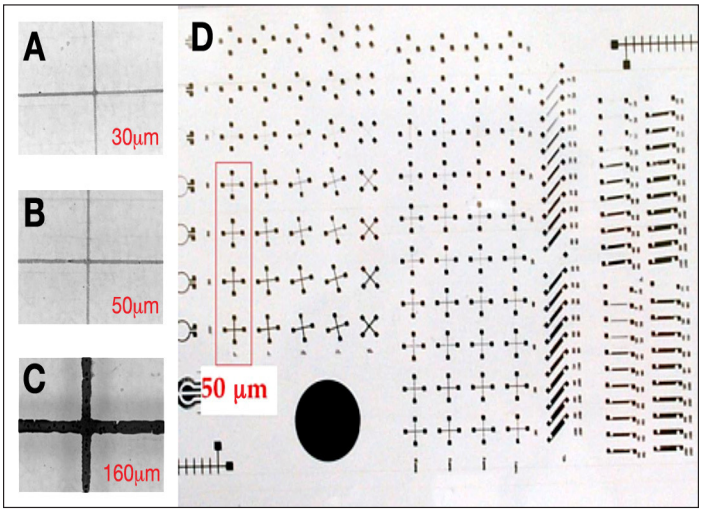


Figure 2. Photo of **(A)** 30, **(B)** 50, and **(C)** 160 μm silver printed lines taken from **(D)** the printed glass sample on the right.

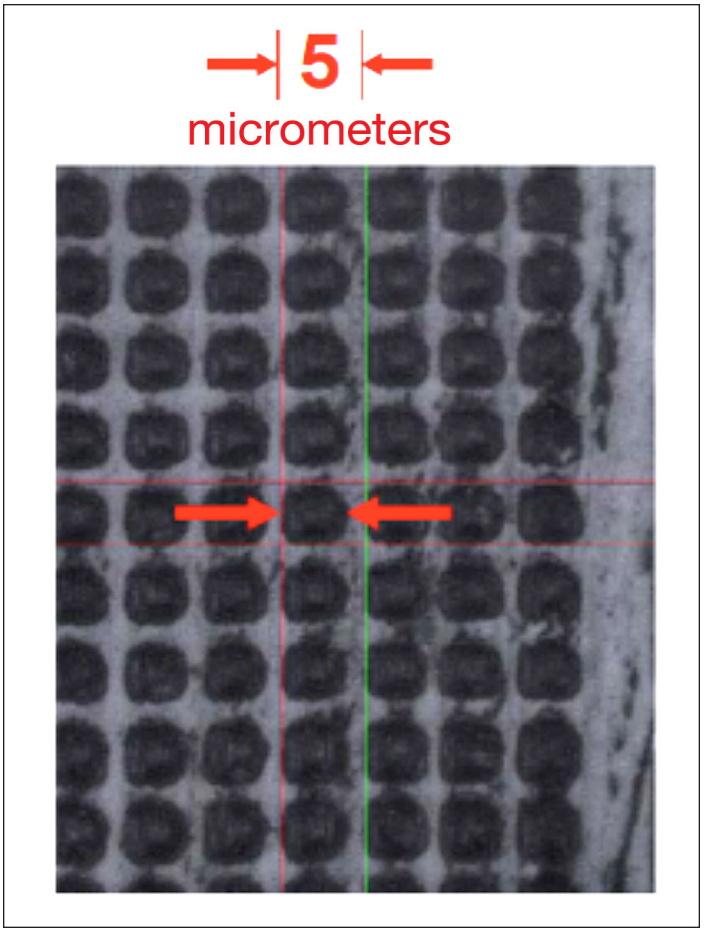


Figure 3. New engraving technology developed by Daetwyler R&D illustrating the accuracy of engraved 5 μm cells.[12] (Reproduced courtesy of Daetwyler R&D.)

Table 1.
Offline Characterization Tools[a]

Measurement need	Description
Electrical	Device: I–V, leakage, capacitance, parametric model extraction Passive: resistance, C–V, high-frequency impedance Circuit: Output voltage and current, impedance, speed, logic, performance with temperature
Material and interfacial structure	Structure of functional inks at critical interfaces: molecular order, grain size, crystalline morphology
Pattern quality	Registration, printing quality, dimensions
Mechanical	Layer thickness, surface roughness, surface defects, material wetting, substrate dimensional stability

[a] Reproduced courtesy of iNEMI, from the 2009 iNEMI Large Area Roadmap. Also see physics.nist.gov/GenInt/STM/stm.html and http://www.chembio.uoguelph.ca/educmat/chm729/afm/operate.htm.

Sensor Design and Performance

Researchers are working on printed sensors to replace photolithographically fabricated sensors for toxic/chemical detection applications. The performance of these printed sensors is comparable to that of photolithographic sensors.

An efficient electrochemical sensor that features interdigitated electrodes for detection of various toxic bio/chemical species was successfully fabricated using photolithography. Gold, an element known for its inertness and affinity toward biomolecules, was chosen as the electrode material. The sensor is shown in **Figure 4**. Analysis of the impedance-based response successfully demonstrated the feasibility of the biosensor to distinguish between various chemical substances and biological proteins at picomolar concentrations.[24–28] **Figure 5** shows the impedance percentage change of the sensor toward HgS at various frequencies. Detection well below the U.S. Food and Drug Administration's limit of picomolar concentration was achieved.[29]

After characterizing the performance of the photolithographic sensors, the interdigitated electrode geometry and impedance-based analysis techniques used were extended to printed sensors on flexible substrates. Conventional printing methodologies were explored to address the challenges of fabricating miniaturized, low-cost, flexible sensors via high-throughput techniques, which are used for applications in bio/chemical detection.[30–33]After multiple trials with different printing methods, rotogravure printed interdigitated silver electrodes were fabricated on polyethylene terephthalate (PET). A schematic of the device with eight pairs of electrodes is shown in **Figure 6A**. **Figures 6B** and **6C** show photographs of a printed array of sensor devices and a single sensor device on flexible PET, respectively. Sensor response was tested in a similar fashion to the photolithographically printed sensor using a custom-built acrylic flow cell shown in **Figure 6D**.

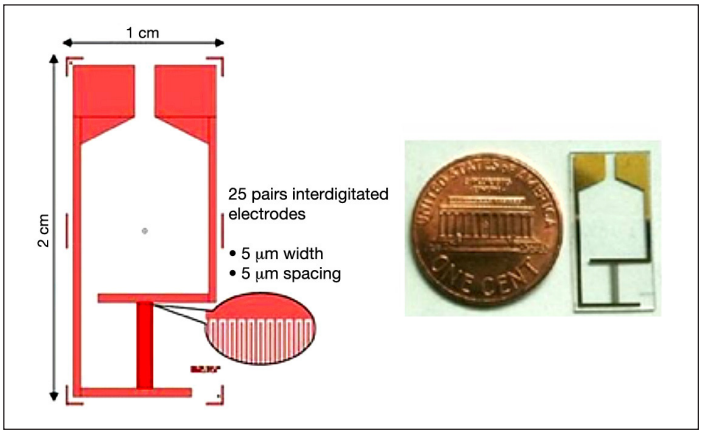


Figure 4. Schematic and photolithographically fabricated biosensor for the detection of various toxic bio/chemical species. (Reproduced courtesy of IEEE Sensors Conference Proceeding, 2009,[28] and *Biosensors and Bioelectronics*.[27])

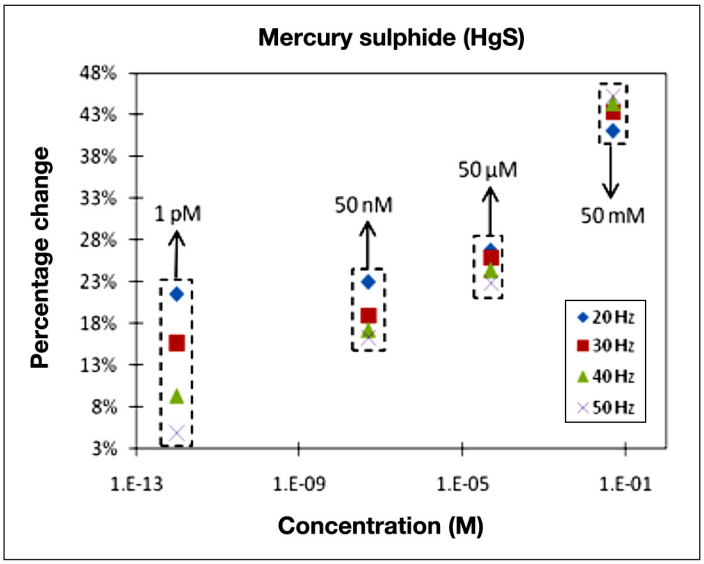


Figure 5. Change in sensor impedence with concentration of HgS as a function of frequency (potential of 1 mV). (Reproduced courtesy of Binu Baby Narakathu, WMU).

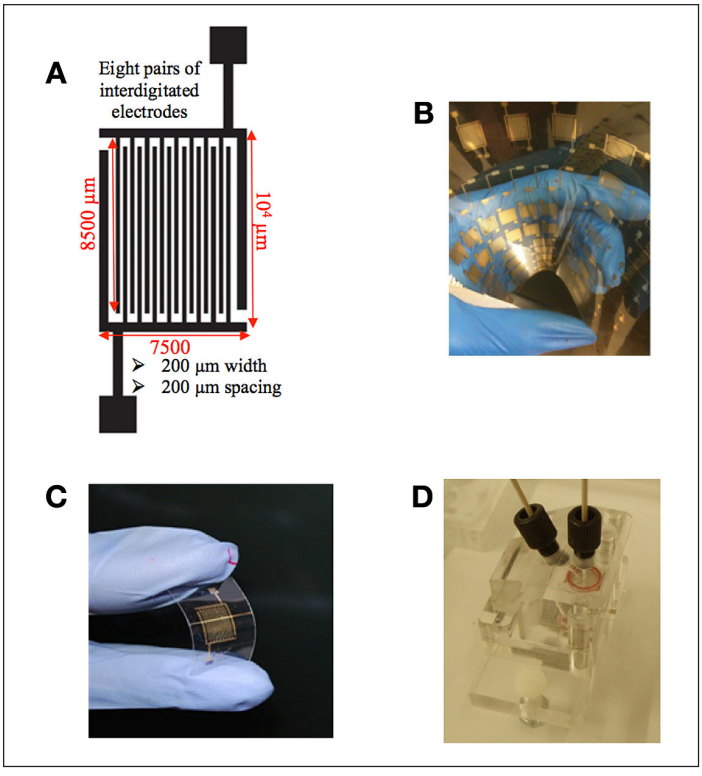


Figure 6. (A) Schematic of bio/chemical sensor, **(B)** array of interdigitated electrodes, **(C)** single sensor device printed on PET, and **(D)** sensor placed in an acrylic flow cell. (Reproduced courtesy of IEEE Sensors Conference Proceeding, 2010.[33])

The electrochemical impedance spectroscopy response of the printed sensor toward varying concentration levels of toxic bio/chemicals demonstrated similar results to the photolithographically fabricated sensors. An example is shown in **Figure 7A**. The response of a poly-hydroxyethyl-methacrylate (pHEMA)-coated printed sensor toward humidity at 25 °C is shown in **Figure 7B**. These results demonstrate the ability to replace high-cost traditional electronic fabrication methods with printing.

Materials Characterization: "Ink" Coating Materials

Selection and testing of coating material "inks" for electronic printing includes characterization of electronic materials formulated as inks and ink composites (e.g., conductive polymer inks, nanopigments, silver composites) and assessing their performance properties relative to subsequent layer compatibility, drying and aging conditions, and method of deposition.[34–36]

For optimum print quality and surface leveling, an understanding of the substrate and wet ink properties is required. Roughness, surface energy, solvent resistance, and thickness of each receiving layer must be characterized and understood to assure compatibility and optimal electrical performance. As shown in **Figure 8**, ink compatibility can have a significant impact on the electronic properties of the printed layer.

Different tests have been successfully used by our group to study and predict the gravure printability of functional inks. Having printed and characterized a broad range of electronic materials, our group has developed an understanding of the relationship between ink, process, and print properties. The influence of ink rheology and process setup (in this case, gravure cell geometry) on print quality and ink coverage are shown in **Figure 9**. A picture of the engraved cells and their dimensions are provided.

Conclusions

Great strides have been made in the areas highlighted here toward producing printed microelectronics and sensors. Advancements can be applied to the field of diabetes testing. Further work at Western Michigan University is geared toward investigating new materials and demonstrating performance, minimizing cost, and establishing semicontinuous production of printed electronic circuit components and sensors while looking forward to implementing developed technologies for R2R processing.

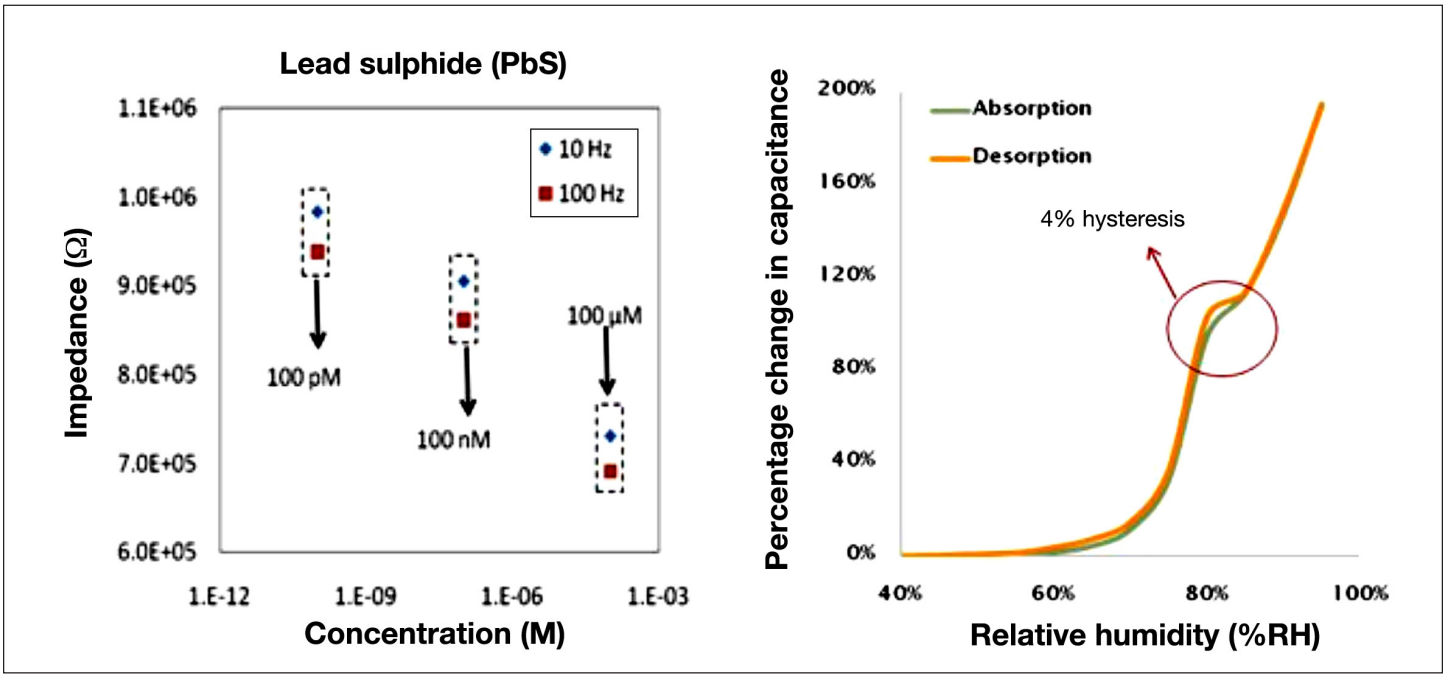


Figure 7. (A) Printed sensor response to PbS at applied potential of 100 mV and **(B)** capacitance response of printed pHEMA-coated sensor to humidity. (Reproduced courtesy of Binu Baby Narakathu, WMU).

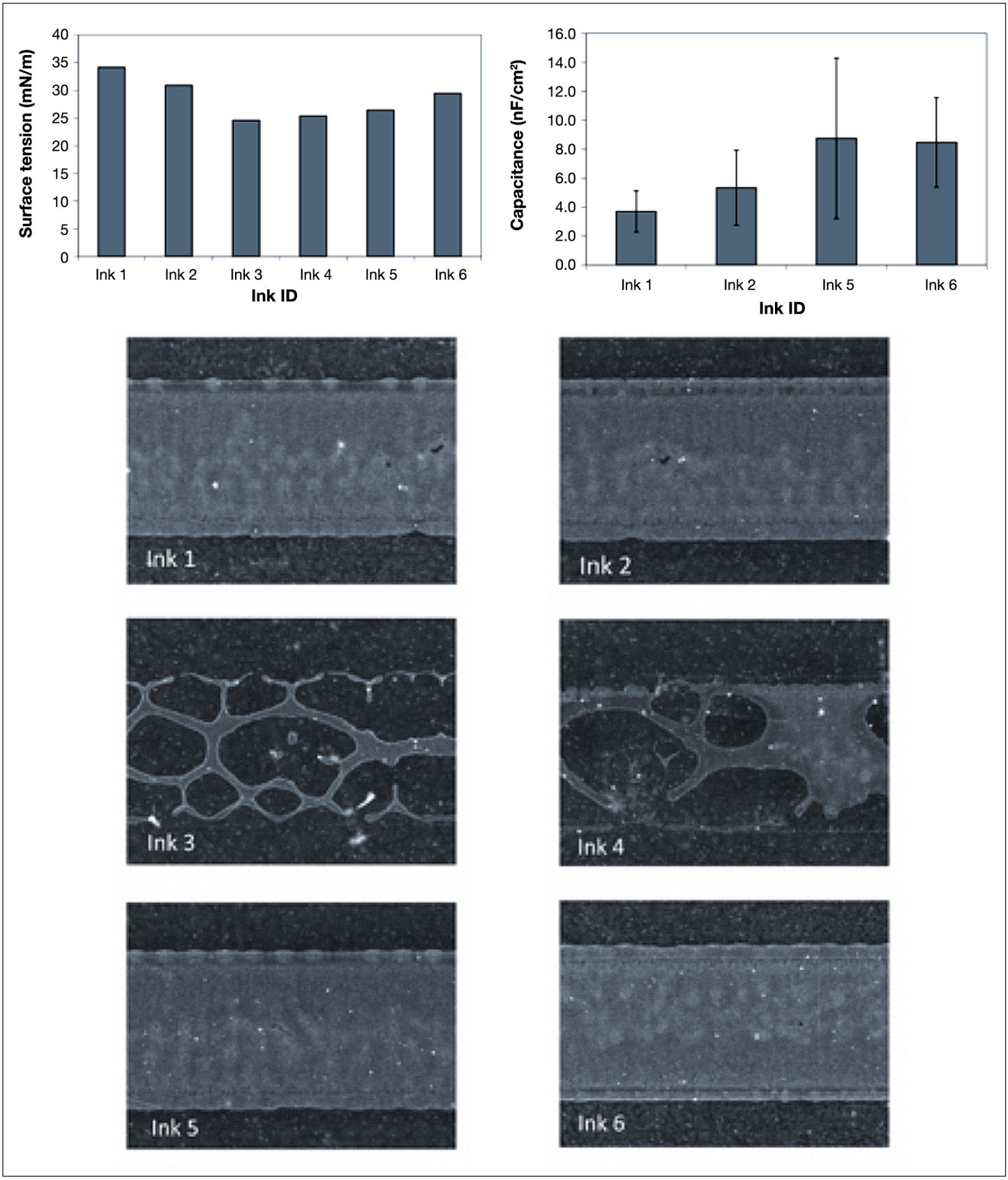
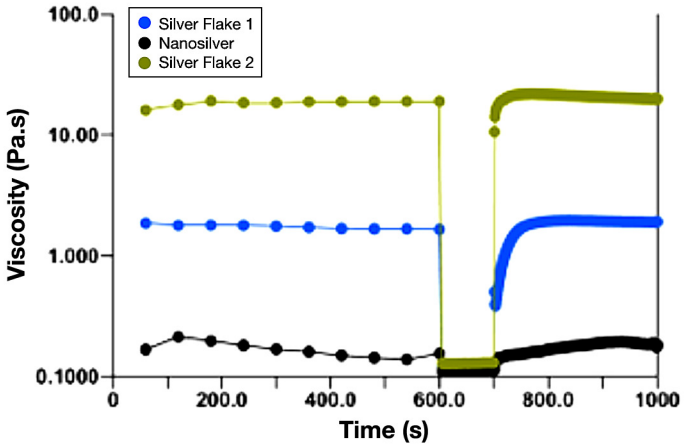


Figure 8. Silver-nanoparticle-filled ink printed on six different poly-methyl-methacrylate dielectric layers of different surface tensions (inks 1–6).

Line screen (lines/inch)	Cell opening (micrometers)	Cell depth (micrometers)
100	242	72
120	212	64
150	183	56
200	128	40



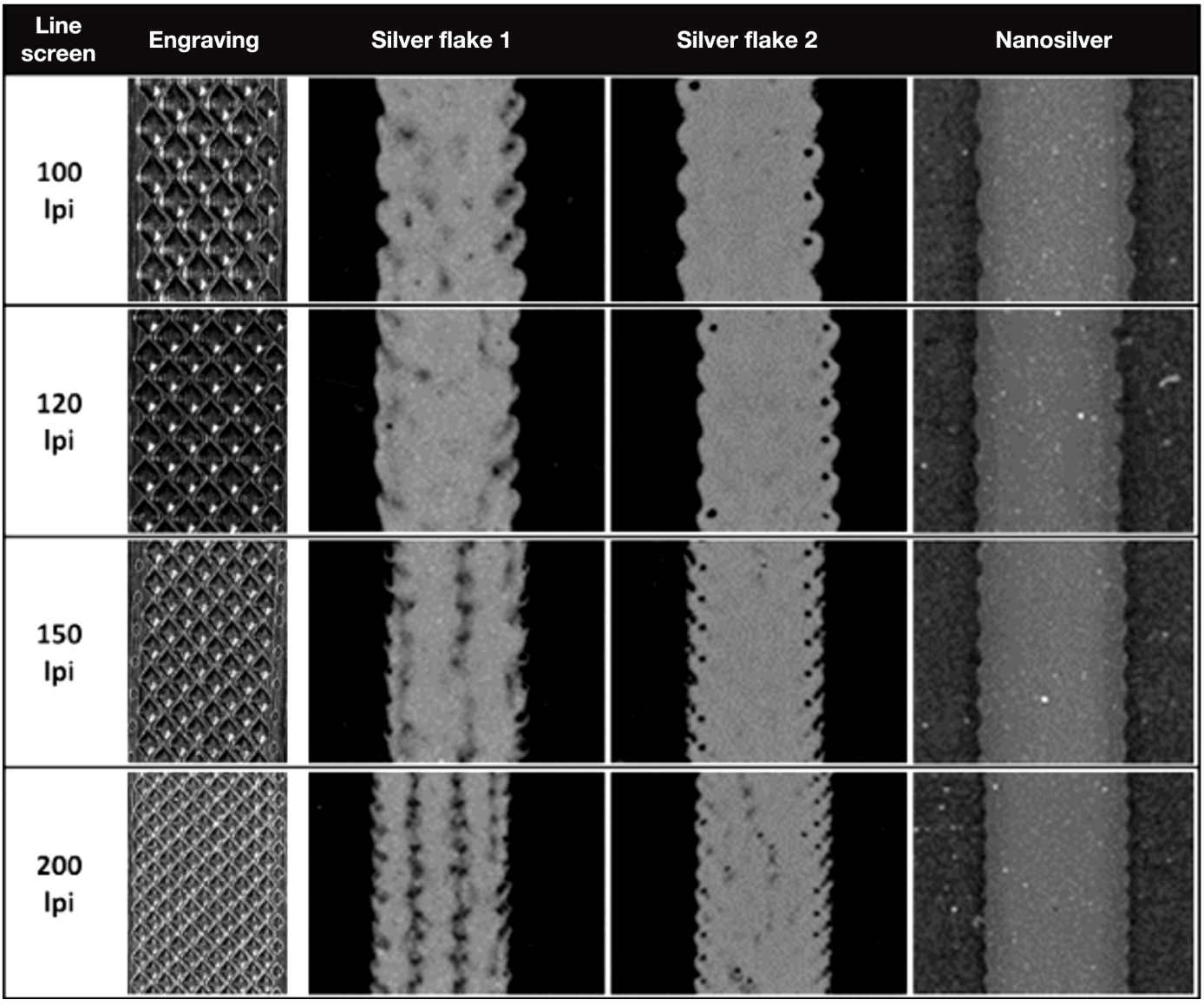


Figure 9. Rheological testing for printing inks performed to correlate and predict the printability of inks on the press, demonstrating the effect of gravure printing parameters and ink rheology on surface coverage and print quality. (Reproduced courtesy of Erika Rebrosova, Center for the Advancement of Printed Electronics, Western Michigan University.)

Funding:

This work was supported by the Center for the Advancement of Printed Electronics, Western Michigan University.

Acknowledgments:

The authors thank Erika Rebrosova and Marian Rebros (Center for the Advancement of Printed Electronics) for their permission to use their photos, figures, and data, as noted.

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