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Flexible Bioelectronics with Power Autonomous Sensing and Data Analytics



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Preface: Introduction and Overview of Flexible Bioelectronics

Motivation for This Book

Advances in materials, processing, and microfabrication have spurred the digital revolution of today. These advances have revolutionized the way we process and communicate information using computers and smart phones. They have changed the way we entertain ourselves using television and video games. And importantly, they have greatly improved the quality of our lives through advances in healthcare and assistive technologies. However, majority of these platforms still rely on designs and materials that are rigid and bulky. They may be well suited for the environment around us; however, they are ill equipped to adorn our body as wearables or be utilized as implantable, which requires conformability and flexibility. The need for flexibility brings new challenges in their fabrication that conventional microfabrication approaches that rely on top down lithography or high temperature processing using hard substrates may not be able to provide without further advancements. This requires new thinking and approaches to process soft materials into flexible devices that can then be integrated into systems for wearable, implantable, or other applications. Over the last decade, there has been tremendous research progress in this emerging area of flexible electronics. The research progress has spanned areas of innovative materials development such as soft polymers, including biomaterials as substrates, to the use of exciting two-dimensional ultra-thin nanomaterials such as graphene as transistors and sensors. Research has also focused on converting existing bulky and rigid materials like silicon into thin micro- and nano- membranes to make them flexible and conformal for integration onto flexible substrates. Ageold methods of roll-to-roll printing and inkjet printing have been transformed to allow fabrication of micron-scale features for flexible electronics. New device developments for transistors and sensors that are inherently more suitable for flexible devices such as electrolytic gating of transistors have also evolved. Finally, there have been a lot of peripheral developments in supporting technologies to interface and communicate with these new flexible devices. These developments may not be flexible themselves but are extremely important for practical realization

of integrated flexible systems. For example, packaging and integration of flexible sensors with conventional Complementary Metal Oxide Semiconductor (CMOS) integrated circuits is key. Another example is that of wireless energy and power harvesting for flexible platforms, which may not be on flexible substrates. Yet another effort is on algorithms and circuits that are more amenable for processing data from flexible devices. These topics while not directly related to fabrication and realization are also quite relevant when it comes to the practical realization of flexible electronic platforms.

Flexible Bioelectronics

Flexible electronics in general has been touted as the next frontiers for consumer electronic devices like flexible displays, curvilinear televisions, and folding phones. However, the biggest driving force for advances in flexible electronics has been the need for devices that can provide intimate interface with the human body for monitoring our health and wellness. For example, there is a need to make next generation of prosthetics that can conform and interface well to the contours of the human body. Flexible bioelectronics is a niche area where flexible electronics overlaps with biomedical applications and where the key goals are to develop materials, devices, and systems that can reside safely on the human body or within it for the purpose of monitoring and augmenting human health and performance. Smart watches and smart rings are commercial products today that fit this space; however, these devices are still rigid and bulky and cannot be adapted for other biomedical needs. More promising examples of flexible bioelectronics take us beyond the smart watch to smart skin, where flexible devices conform directly on the human skin for health monitoring and provide intimate human-machine interfaces. Yet another example of flexible bio-electronics is that of smart bandages for the treatment of chronic wounds, where a biocompatible interface containing flexible sensors and drug delivery is needed to monitor and repair deep wounds. An example in the realm of implantables is that of a flexible neural electrode array for stimulation of and recording from neurons in the brain and peripheral nervous system. The next generation of neural interfaces also incorporates drug delivery and/or chemical stimulation for truly remarkable multi-modal neural implants. In such applications, there is an even more stringent requirement on the biocompatibility to ensure long-term chronic implantation. Every material, structure, or design used in the development of such flexible bioelectronic implants needs to be scrutinized for long-term toxicity and inflammation all the while ensuring continued functionality and performance over months and years of implantation. Different biomedical applications elicit different requirements for materials, design, and structure with unique processing and fabrication techniques more suitable for their form and structure. For example, neural implants are expected to have softness and mechanical properties of brain, which are more adequately achieved using hydrogels or soft biopolymers, which also happen to be more biocompatible. And these materials can only be patterned or shaped into devices using low temperature solution-based processing rather than conventional cleanroom techniques. These requirements are unique to flexible bioelectronics and form a subset of all approaches and methods available for flexible electronics. This emphasis will be the focus of the discussion in this book. It is also well understood that the progress in flexible bioelectronics will have to go through multiple phases of development, where the earliest and nearest developments will focus also on the practicality of merging simple concepts/devices for flexible sensors and electronics with conventional electronic devices that may be rigid but small (e.g., CMOS IC). These hybrid platforms combining small integrated circuit chips on flexible substrates through flexible interconnects can provide amazing flexibility and conformability adequate for many applications such as wearable electronic skins. The book will attempt to focus on such hybrid integrations, and also on conventional and novel circuits and algorithms that are suitable for such hybrid platforms.

Contents of This Book

Flexible electronics and bioelectronics has been a very active field of research and development in recent years. There have been many review papers written on this topic with select books. The focus of those papers and books has been solely from the point of view of material scientists who extol the virtues of polymers and flexible substrates, or those who focus on engineering hard materials through thinning and patterning to achieve flexibility. There are yet others that focus on specific applications. However, there is no discussion on flexible bioelectronics from the view of practicing engineers on how to apply their knowledge and expertise in devices, circuits, and algorithms to this emerging field. This book tries to address such a gap in understanding by first providing a gentle introduction to the materials and processing (Chap. 1) followed by basics in sensing (Chap. 2). Two detailed case studies of flexible bioelectronic platforms, namely smart flexible wound dressing and textile-based wearable diagnostics (Chap. 2), will provide a comprehensive review and deeper understanding of the challenges and promises of this field. Chapter 3 will finally delve into CMOS-based integrated circuits for readout from flexible sensor platforms covering both resistive and capacitive sensors. This discussion assumes a hybrid flexible bioelectronics approach where flexible sensors and flexible substrates interact with small CMOS ICs for readout and signal conditioning. Chapter 4 will continue the discussion of further digitizing signals using data converters amenable to sensing applications. Beyond circuits, efforts on energy harvesting and power management are critical for flexible bioelectronics platform and will be discussed in Chap. 5. Chapter 6 will finally discuss algorithms for processing and data analytics pertaining to flexible bioelectronic applications. Majority of the discussion will be on intelligent sampling and data acquisition to reduce power dissipation without sacrificing accuracy through compressed sensing approaches, and also data recovery and remediation in the presence of noise and motion artifacts.

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Chapter 1: Materials and Processing of Flexible Bioelectronics

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Chapter 2: Sensors and Platforms for Flexible Bioelectronics

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Chapter 3: Low-noise CMOS Signal Conditioning Circuits

- Dr. Maryam Shojaei Baghini (IIT Bombay), Dr. Meraj Ahmad (IIT Bombay) and Dr. Shahid Mailk (IIT Delhi)
- Chapter 4: Data Converters for Wearable Sensor Applications
- Dr. Maryam Shojaei Baghini (IIT Bombay), Dr. Laxmeesha Somappa (IIT Bombay) and Dr. Shahid Malik (IIT Delhi)
- Chapter 5: Power Management Circuits for Energy Harvesting
- Dr. Maryam Shojaei Baghini (IIT Bombay), Dr. Meraj Ahmad (IIT Bombay), Dr. Shahid Malik (IIT Delhi) and Dr. Gaurav Saini (IIT Bombay)

Chapter 6: Sampling and Recovery of Signals with Spectral Sparsity

Dr. Shuchin Aeron (Tufts University) and Dr. Laxmeesha Somappa (IIT Bombay) Chapter 7: Compressed Sensing

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Chapter 1 Materials and Processing for Flexible Bioelectronics



1.1 Materials for Flexible Bioelectronics

A fully functional wearable device may integrate different physical, chemical and biological sensors with electronics for data collection, battery to power them and a display to show the results. In some cases, data can be wirelessly transmitted to a phone or a PC using flexible antennas and a wireless communication module, which can be used instead of a display. In some other bioelectronic applications, there may be actuators such as electrodes and circuits for electrical stimulation or drug delivery. A conceptual schematic layout of an example bioelectronic platform realized on a flexible substrate is shown in Fig. 1.1 [1].

Since bioelectronic devices are expected to interact intimately with the human body, their flexibility and biocompatibility with the underlying human tissue is of paramount concern. Mechanical flexibility (or stiffness) of the material is captured by its Youngs' modulus and varies widely based on the tissue or organ you are interfacing with as shown in Fig. 1.2 [2, 3]. As Fig. 1.2 suggests, there are large differences in the mechanical properties between different human tissues and materials (silicon, metal electrodes) used in conventional electronic devices. Notably silicon-based devices made using conventional photolithography are hard and brittle and not compatible with majority of tissue or organs such as brain or skin, and thus poorly suited for direct integration with them. There are two ways to minimize this gap in flexibility between and underlying material and the target tissue, one is to replace these electronic materials with more soft options. The first is a bottom-up approach where each material and device is flexible and stretchable at all length scales and under integration. The second approach is to miniaturize rigid devices and integrate them within a flexible polymeric material those that possess

Dr. Sameer Sonkusale (Tufts University) and Dr. Narendra Kumar (Boston College) contributed to this chapter.

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Fig. 1.1 A conceptual schematic of flexible bioelectronic device carrying different sensors, electronics to provide interface and data processing, a battery to power the electronics, and an electronic display for results. Some bioelectronic platforms may have an antenna and wireless communication device instead of a display, while some other platforms may also have actuators such as electrical stimulators or chemical drug delivery modules as part of the platform (not shown in the figure) [1]



Fig. 1.2 Young's modulus of different target tissues and organs, compared to different materials to realize bioelectronic devices [3]

the elastic moduli close to the human tissues. Options to reduce thickness of bulk rigid materials such as thinning or making ribbons and using geometric patterning such as using serpentine layout imparts are also utilized to achieve flexibility. Going with the first approach requires extensive investigation on the properties and their implementation to develop electronic devices—several efforts are already being made since last 15 years with several successful examples [4–8]. The second approach is more convenient and practical, as one only needs to focus on developing

smarter ways of integration that can result in a flexible bioelectronic device using rigid electronic and sensing components, or thinning bulk materials and patterning them [8-11].

We first begin our discussion by going through some choice of materials for substrates and materials for realizing electronics and sensors. Following this, we cover some standard methods of processing and fabrication of flexible devices and ways to integrate them. We also separately dedicate discussion on the second approach listed above to integrated miniature conventional electronic devices on flexible substrates to realize flexible and/or stretchable bioelectronics.

1.2 Substrates

Substrates serve as a foundation over which sensors, electronics, and other functional components are built to realize integrated devices. Therefore, their choice is crucial in determining the flexibility, biocompatibility, and performance of wearable flexible bioelectronic devices. The choice also depends on application. For example, one may need them to be transparent. In other application, they may need to be resistant to humidity and temperature, and while in some other cases, swelling of the substrates may be acceptable or even desirable. There are two major classes of materials being utilized as substrates, one is a class of polymeric materials and others are cellulosic substrates, which include papers and fibers/textiles.

1.2.1 Polymeric Materials

The most common substrates for flexible electronic devices are polymers such as polyethylene terephthalate (PET), polyethylene naphthalate (PEN), polyimide (PI), parylene, and polydimethylsiloxane (PDMS). PET substrates fulfill most of the requirements for substrates for flexible bioelectronics such as flexibility, chemical inertness, thermal and mechanical stability, and smoothness. Cost-effectiveness and extensive utility of these substrates in other areas such as for food packaging, liquid containers, and face shields make them highly desirable. PEN provides comparatively better thermal stability and chemical resistance compared to PET and is also a popular choice when conventional clean room fabrication process is desired to make devices. PI is another popular flexible material for the same reasons and is also heavily used in flexible printed circuit boards. Another material that is also FDA approved for medical and implantable devices is parylene, which is a highly inert substrate and is very popular as hermetic coating in implantable devices. They have been used for making electrode arrays for direct brain implants or making peripheral nerve interfaces. However, one of the main challenges with the materials listed above is that while they are flexible, their Young's modulus may still be quite higher than skin or brain, the organs they are used to interface with. Polydimethylsiloxane (PDMS) is an excellent choice for implantable sensors due to

their high stretchability and Young's modulus closer to skin and other implantable tissues. They also show high biocompatibility and can be used for fixing sensors and devices directly on body parts especially at joints. They meet most of the criteria for future flexible bioelectronics because they are cost-effective, robust, and compatible to human tissues and conventional microfabrication techniques. It also offers opportunity to couple well-established microfluidics technology on PDMS whenever required for such devices and can be integrated together with electronics. PDMS is also a well-suited elastomer to be utilized as a matrix to integrate different electronic components to realize a wearable device, more details are provided in the later part of this chapter. However, PDMS is not always chemically inert. It also has its own limitations such as swelling in organic solvents and nonspecific adsorption of cells and proteins in case of microfluidics and implantable applications.

Hydrogels (e.g., poly(ethylene glycol), poly(vinyl alcohol), collagen, gelatin, chitosan, alginate) are class of materials which have made significant advancement to provide flexible interconnects and conducting electrodes [12–15]. They possess strong stretchability, biocompatibility, and closely match elastic moduli to human tissues. Moreover, they provide flexibility of modulating their electrical, mechanical, and biological properties making them a unique bridging material to biological world [14]. Hydrogels are made from cross-linked polymeric network and have high degree of hydrophilicity because of excess amount water contents, which easily allow the transport of biological and chemical analytes desired for bioelectronic devices [16]. All these properties of hydrogels rendered their growing utilization in different bioelectronic devices and being utilized either as interface material or to form composite matrix to modulate the properties of metallic and carbon-based materials [13]. Recent advancements in the field are utilizing hydrogels to develop tissue like wearable bioelectronic platforms [17, 18].

1.2.2 Fibers/Textiles

Substrates based on fibers, threads, and textiles offer a highly versatile option to realize flexible bioelectronics [19, 20]. First of all, they are cheaper than other substrates, and benefit greatly from established textile making and processing infrastructure. Fibers and threads also offer a rich diversity of material options (e.g., rubber, nylon, cotton) which can be knitted, woven, or patterned to achieve flexibility or stiffness over a wide range of values for Young's modulus. Tunable mechanical properties and long lifetime of fibers and textiles provide a promising platform to realize wearable sensing devices. Compared to top-down fabrication approaches to realize electronic devices on other substrates, textile-based flexible bioelectronics utilize more bottom-up approach. This is because, textile-based substrates are nonuniform and porous making uniform patterning and coating harder. Better results can be achieved if functional smart threads are first made using conventional dyeing or coating approach, and then threads are sewn or knitted together to make functional devices [19, 20]. Several well-known knitting and

weaving processes allow integration of the functional fiber-based electrodes and electronic components on desired textiles. The major advantage of fabrics/textile is that they do not need cleanroom-based fabrication methods and use ambient processing. A sister substrate to textile, and one that is just as ubiquitous is paper, a cellulosic substrate that is environmentally friendly and amenable to simple processing to make functional devices such as inkjet printing or screen printing. However, the paper-based substrates are suitable for short-term temporary applications as they are biodegradable and have poor thermal and chemical stability.

In general, the recommended substrates for flexible bioelectronics depend on the application and the type of functional devices one intends to make on them. As mentioned, the choices can range from PET, PEN, and PI for long-term flexible devices, to PDMS as matrix to fabricate and integrate conventional rigid electronic devices and circuits to textile-based substrates for wearable applications. We should mention here, as we did earlier, that in near term, the approach of integrating conventional electronics over flexible elastomeric support seems more reasonable over approaches that rely on fully novel material systems.

1.3 Electronic Functional Materials

As shown in Fig. 1.1, flexible bioelectronic platform consists of sensors, transistors, and other functional components. Herein we summarize different choices of materials used in making these components. The list may not be complete but provides a quick insight into available options.

1.3.1 Organic Materials

Organic electronic materials (OEMs) have been widely studied and utilized to develop flexible electronic devices such as solar cells, organic LEDs, and batteries [21–23]. OEMs are also interesting active materials for flexible bioelectronics because of their synthetic tunability, biocompatibility, and mechanical flexibility, which make them suitable to integrate with cells and biological tissue [24, 25]. Major OEMs being utilized as active semiconducting materials in bioelectronics are poly(3,4-ethylenedioxythiophene):poly(styrene sulfonate) (PEDOT:PSS), poly (3-hexylthiophene-2,5-diyl) (P3HT), and phenyl-C₆₁-butyric acid methyl ester (PCBM) [26, 27]. PEDOT:PSS is a class of highly conducting OEMs consisting of a polymer mixture of positively charged PEDOT with negatively charged PSS, and their conductivity can be tailored by choosing the ratio of these two compounds and their resulting particles size in dispersion. It is commonly used as a semiconductor that works in accumulation mode when utilized as an active channel material in transistors because of its high mobility >0.1 cm²/V.s and low

operating voltage as compared to PEDOT:PSS [30]. Both these OEMs are also being employed to fabricate organic electrochemical transistors for developing flexible biosensing devices [31, 32]. Doping or de-doping of electrolytes in PEDOT:PSS and P3HT can be achieved using a gate electrode in an aqueous environment providing transistor functionality [33, 34]. OEMs allow exchange of ions between electrolyte/biofluids and the organic semiconductor resulting in the involvement of the whole volume of the film interacts with biological environment, not just its surface, a feature which can be exploited to develop powerful biosensors and bioactuators [24].

1.3.2 Metal Oxides

Metal oxides and their nanostructures are another class of materials those can be utilized for flexible devices [35]. These materials are available in the form of dielectrics, semiconductors, and conductors. Zinc oxide (ZnO), indium oxide (In2O3), indium gallium zinc oxide (IGZO), indium tin oxide (InSnO), and many more combination possesses semiconducting properties and are being utilized as active layer in fabricating transistors [36]. Metal oxide semiconductors possesses excellent electrical performance (mobility >10 cm² V⁻¹ s⁻¹ at room temperature processing) along with their compatibility of fabrication over flexible substrates. On the other hand, aluminum oxide (Al_2O_3) , titanium oxide (TiO_2) , yttrium oxide (Y₂O₃), zirconium oxide (ZrO₂), tantalum oxide (Ta₂O₅) are utilized as gate dielectrics because of their high dielectric constant and smoother interface with metal oxide semiconductors [36]. For example, IGZO thin-film transistor showed a mobility of 14.5 cm² V^{-1} S⁻¹ processed below 350 °C, while it showed a mobility of 7.5 cm² V⁻¹ S⁻¹ processed at room temperature (RT) [37, 38]. These materials can be processed either by physical deposition systems at low temperature or through solution processing techniques discussed earlier. The solution processing of these materials by first preparing the nanomaterial precursor inks using sol-gel process offers a low-cost route that can then be used to make devices using spin/dip coating and screen/inkjet printing [39]. However, most of these materials still require high temperature (\geq 300 °C) annealing and suffer from limited flexibility and stretchability; additional modifications in the supporting substrates and encapsulation are needed to overcome these challenges [39–41].

1.3.3 Carbon-based Materials

Carbon-based nanomaterials such as graphite, carbon nanotubes (CNTs), and graphene are expected to provide a more natural and biocompatible interface with the living universe that is entirely based on carbon. CNTs and graphene have shown great promise in the field of flexible bioelectronics with several examples from neural electrode arrays to flexible transistors or electronic tattoos [42-45]. Specifically, the CNTs can be metallic or semiconducting, grown by chemical vapor deposition (CVD) in bulk and easily dissolved in solvents to make ink formulations that can be used to fabricate electrodes/transistors using spin/spray coating, and inkjet/screen printing [45-48]. For instance, high-performance and low-voltage (3 V) flexible analog and digital circuits were fabricated using CNTs in solution form. The transistors fabricated on 4 inch polyimide substrate showed high mobility values ~34 cm²/V.s with 5 mm bending radius [49]. There is well established growth and transfer process of graphene making it one of the popular sensing and electronic materials at present. Recently, the roll-to-roll fabrication of these 1D/2D material-based devices has been reported indicating their potential for future flexible electronics [50, 51]. For electrochemical sensing, it is well known that graphitic carbon provides a stable working electrode. Carbon nanomaterial-based inks and paste are readily available commercially for screen printing of working electrodes and counter electrodes on any flexible substrates to make disposable electrochemical sensors [52–56]. Furthermore, the stability of carbon-based electrodes and materials in physiological environment, flexibility, and biocompatibility make it an excellent choice for future flexible bioelectronic devices [45].

1.3.4 Conductive Inks and Liquid Metals

Conductive inks (gold, silver, carbon, etc.) are easily available in the market and can be printed/coated/pasted on any kind of substrate using drop casting or inkjet printing. There are pens available filled with these formulated inks and are extremely helpful to draw the desired connection to make the circuitry. The properties of these inks can also be modulated as per the requirement of the application. There is also a eutectic alloy of gallium–indium that is metallic liquid at room temperature and has interesting wettability on select surfaces; this can be used to realize flexible interconnects that are self-healing in presence of breakage [57, 58].

1.4 Materials Processing

How do we go from materials to functional devices such as sensors or electronics is the subject of this discussion on materials processing. Since we are interested in making devices on flexible polymeric substrates, it is important that processing is done at low temperature since these substrates cannot handle high temperature. Solution-based methods and bottom-up approaches are more suitable. We cover two different techniques for materials processing to make flexible devices, one is a bottom-up approach using different functional inks and patterning them directly on flexible substrate, while the other is based on transfer printing from one substrate to another.

1.4.1 Printing from Inks

The various solution processing techniques such as spin, dip, spray, blade coating, and electrochemical deposition can be performed at low temperatures followed by evaporating the solvents at temperature susceptible to flexible substrates. In these processes (e.g., spin coating), the desired materials are synthesized as nanoparticles in a precursor having different solvents. After that, the solution is filtered to remove any remaining clusters. Coating is done by pouring the solution over the surface of substrates and running the spin coater at optimized spinning speed. In this process, centrifugal force spins out the solution from center to outward direction which depends on the speed chosen to obtain the desired thickness and uniformity. In the dip coating, the substrates are dipped in and out of the prepared solution at different speeds with varying number of dipping cycles. Spray coating, the substrate rotated at fixed speed and solution sprayed though nozzles, spray timing, and speed of substrate rotation decide the thickness and uniformity of film. The blade coating is done by injecting the ink (solution) while maintaining a fixed distance between knife/blade and moving substrates. The optimized annealing is done to evaporate the solvents followed by characterization of materials properties. See Fig. 1.3 for conceptual representation of these methods [59]. While these methods provide a uniform coat needed for any material, one needs to employ other methods to patterning them.

In conventional lithography, optical masks and photoresists are used to identify different areas for either ion implantation, or metal sputtering or oxide growth. However, this is not practical for realizing devices on flexible substrates due to their sensitivity to high temperature. Instead, solution-based patterning of different materials will enable realization of multilayered devices such as transistors or sensors. Some of the available methods for patterning with different inks are shown

