SOFT OPTOELECTRONIC SKINS FOR
FLEXIBLE AND FOLDABLE STRUCTURES

A Thesis in

Aerospace Engineering

by

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ABSTRACT

The development of flexible electronics has shown great potential in medical applications. The focus in recent studies has been on wearable electronic devices for monitoring various human health conditions. However, fewer studies have been conducted in other disciplines. This thesis will present two potential applications of flexible electronics in the discipline of aerospace engineering. The first application is targeted at structural behavior monitoring. Traditional electronics that are rigid and heavy have now shown their disadvantages for integration with the more flexible and deployable structures, which have been adopted for most spacecraft vehicle and structure designs. The results presented in this thesis provide an alternative approach to overcoming such issues with the concept of flexible structural electronic skin. The fabricated devices are demonstrated to be able to monitor various aspects of a deployable tape-spring hinge, including motion, surface orientation, vibration mode and frequency, crease development and crease dislocation.

The second application is targeted at monitoring environmental conditions in harsh environments. By combining flexible electronics with thin-film ZnO/SWNT-based ultraviolet photodetectors, this thesis demonstrated that the fabricated devices can survive the various harsh environments, including extreme temperatures (-80 °C to 100 °C), UV irradiation, and low pressure (6.7 kPa), which are commonly experienced in Antarctica, low-earth orbit, and Mars. In all scenarios, the UV photodetector has shown stable response to UV irradiation, with an increase in photoresponsivity as temperature increases. The performance of the fabricated sensor is also compared to that of a commercially available UV photodetector, which further demonstrate the comparable sensing capability and the advantage of flexibility. This thesis also successfully incorporated the near field communication protocol to enable wireless data acquisition, which
further demonstrated the potential of the fabricated devices when compared with commercially available ultraviolet photodetectors.

This thesis, in both applications, provides a platform for designing and fabricating a structural electronic skin with various sensing capabilities. The sensors presented are for demonstration purpose, whereas other thin-film sensors can be adapted to fit the proposed fabrication process. In addition, with the help of programming, it is possible to convert the data acquisition process to be completely automatic. In the case of the electronic skin for a tape-spring hinge, it is also possible to computationally regenerate the in-situ shape of the hinge at any given time with the data acquired.
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Chapter 1

Introduction

1.1 Overview

Flexible electronics have proven to have great weight-accuracy efficiency in recent studies. The focus of these studies has been lying within the bio-medical realm. The ultimate goal of using flexible bioelectronics is to substitute heavy, traditional medical devices with thin, light-weight devices which provide equally accurate measurements of human conditions. These include, but not limited to: human motions, breathing patterns, body temperature, cardiovascular characteristics, and voice recognition. **Fig. 1** shows a comparison between the potential of flexible electronics (right) and the undesired characteristics of current rigid devices (left) in the case of neonatal intensive care.

**Fig. 1** Comparison between traditional medical devices and envisioned flexible electronics for neonatal intensive care. Image credits: PA/ John A. Rogers and Todd Coleman.

It is noticeable that in **Fig. 1 a)** the wired connection of traditional devices, to a large extent, limits the normal movements and behaviors of the infant under monitoring. The proposed use of flexible electronics in **Fig. 1 b)**, on the other hand, tremendously alleviates the burden placed on the infant by removing the cables and heavy patches. Due to the flexibility and stretchability of human skin, it is essential to have medical devices with the same or comparative characteristics.
This make flexible electronics more promising for human health monitoring. For the scenario of structural health monitoring, this has not been a widely adopted concept. With rigid structures, flexible electronics are only advantageous due to their light weight and small volume. However, in recent developments of space vehicle and structure designs, the potential of deployable or stretchable structures have been extensively studied. Traditional rigid electronics can no longer match with the flexibility or stretchability of these structures, therefore making the usage of flexible electronics more prominent.

1.2 Objective

The objective of this thesis is to demonstrate the possibilities of using flexible electronic skins (e-skin) for the health monitoring of foldable and deployable structures. Two major research objectives are pursued. Firstly, a flexible e-skin is developed for monitoring the structural integrity of a deployable hinge by providing in-situ measurements of the mechanical behavior of the hinge, during and after deployment or stowage. Secondly, this fabrication method is adapted to allow in-situ wireless measurement in the harsh environments that are commonly experienced by a launched spacecraft or space structure.

1.3 Layout of Thesis

Chapter 1 consists of a short overview of the motivation and objectives of this thesis.

Chapter 2 presents an in-depth literature review on the related topics, including the aspects mentioned in section 1.2. Firstly, the current general development in flexible electronics is discussed, with a follow-up discussion on how flexible electronics are currently integrated with aerospace applications. Secondly, a discussion on deployable structures focused on deployable
booms is presented, in order to introduce the target structure for structural health monitoring. Thirdly, challenging harsh environments are discussed to further explain the final goal of the fabricated devices. Finally, a discussion on carbon-nanotube-based thin-film sensors is presented to introduce the key sensing materials used for the fabricated devices.

Chapter 3 shows the experimental results which demonstrate the functionality of the fabricated device as an e-skin for structural health monitoring. These results show measurements of the mechanical behavior of a deployable tape-spring hinge, including bending, twisting, dynamic and quasi-static release.

Chapter 4 shows the experimental results for an adapted e-skin for sensing in harsh environments, including extreme temperatures (-80 °C to 100 °C), UV irradiation, and low vacuum (6.7 kPa), which are commonly experienced by launched space vehicles and structures.

Chapter 5 concludes the thesis, summarizing the results and noting future research directions.
Chapter 2

Background and Literature Review

2.1 Review on Flexible Electronics

Flexible electronics refers to a category of electronic devices that can be conformed to any shape and surface, with excellent capabilities for bending, twisting and other motions that lead to strain development which traditional devices cannot withstand. The concept of developing flexible electronics is inspired by the multifunctionality of human skin [1], which is a combination of surface coverage, sensing, and regulatory organs. The composition of flexible electronics normally consists of three layers of materials. The first layer is commonly a flexible plastic membrane which serves as the flexible or stretchable substrate. Polyimide is one of the most commonly used substrates for its excellent temperature and chemical resistance. The second layer generally forms the electronic circuit with thin-film conductive metals (commonly silver, copper, and gold). The third layer can be described as the functional layer where electronic components are attached. The electronic components can include both organic and inorganic materials to form transistors or sensors depending on the desired functionality.

2.1.1 Fabrication Methods of Flexible Electronics

Various fabrication methods have been developed for the preparation of flexible electronics. Depending on the materials used, the fabrication methods need to be varied and combined to meet with the compatibility of the processed materials. In this section, some of the most common and general approaches of fabricating flexible electronics are introduced.
2.1.1.1 Direct Printing on Flexible Substrate

Direct printing is one of the straightforward adaptations from the fabrication of traditional, rigid PCBs (printed circuit boards). As the demand of flexible electronics has increased, researchers have made tremendous efforts in enabling direct printing on flexible substrates. **Fig. 2** shows a general process of direct printing on flexible substrates with liquid metal alloys [2].

![Fig. 2 Schematic of direct printing using liquid alloy for electrical circuitry on coated paper](image_url)

In order to achieve controllable printing profiles, the surface chemistry of the substrate is carefully studied. Surface-coated paper, one of the most promising substrates, has been the focus of this development [2], due to its cost-effectiveness. Tobjörk and Österbacka gave an excellent review and a detailed summary of the properties of surface-coated paper serving as the substrate for direct printing method, and also of the available printing materials for the electronics [3]. They stated that there is no universal guidance on the surface chemistry of the paper substrate, but rather it varies widely for different applications and printing materials, therefore, making the development of this technique very specific to each application. Although the requirements depend heavily on the application, some major challenges are more generally considered, including large surface roughness, porosity, and chemical impurities.
Since this method was not adopted for this thesis, more in-depth explanations will not be included. For readers interested in more details of the different processes and materials developed for direct printing, a few review articles are cited here for references [4] [5] [6].

2.1.1.2 Photolithography and Transfer Printing

To overcome the challenges faced by direct printing method, many have adopted photolithography and transfer printing methods to prepare devices with much consistence designs. Fig. 3 shows a schematic of a standard photolithography process. The first step of the process is to fabricate a photomask with designed patterns. The masks are normally clear glass with a thin layer of high-density chromium or iron oxide. After the sample is spin coated with photoresists, the sample will be placed in close contact with the photomask, normally aided with vacuum. Then, UV irradiation will activate the photoresist in the non-patterned region. Depending on the polarity of the photoresist, one of the regions (patterned or non-patterned) will be dissolved by photoresist developers, leaving the targeted pattern on the sample. The photoresist shown in Fig. 3 is an example of positive polarity, where the exposed area became soluble. Then, depending on the conductive materials used, a specific etchant will be used to selectively etch the non-patterned region. A dry etching process, for example plasma etching or reactive ion etching (RIE) can be used to pattern the flexible substrate, if needed. Generally, all polymeric materials will be spin coated in a cleanroom environment to achieve low impurities and great surface evenness and smoothness. Thin-film metals can be deposited onto the spin coated substrate through e-beam evaporation or thermal evaporation.
Fig. 3 Schematic of photolithography process. Step 1: photolithography is used to form patterned photo resist to protect the designed circuitry; step 2: removing excessive, unprotected materials through wet or dry etching; step 3: removal of photoresist to expose the desired circuitry. (Image credits: Yao, Ning, et. al. AIAA 2019.)

Fig. 4 Schematic of transfer printing process. Step 1: removing the electronic circuitry from stiff substrate with water-soluble tape; step 2: bounding the transferred circuitry to soft substrate via surface chemistry; step 3: dissolving the water-soluble tape; step 4: achieves self-standing flexible electronics. (Image credits: Yao, Ning, et. al. AIAA 2019.)

Critically, in order to perform photolithography, a stiff substrate is required, which poses a great obstacle towards the final goal of self-standing flexible electronics. To overcome this issue, a transfer printing technique has been adopted. This method has been adopted in many of Prof.
John A. Rogers’ publications. Fig. 4 shows a schematic of a generic transfer printing process. It can be noted that, to enable such process, a sacrificial layer was required to be spin coated onto the stiff substrate prior to photolithography steps. A commonly used sacrificial material is polymethyl methacrylate (PMMA), which is soluble in acetone. The sacrificial layer needs to be removed prior to transfer printing. In case of using PMMA, the fabricated device needs to be immersed in acetone for an extended period for complete removal. Then, a water-soluble tape is used to help pick up the device from the stiff substrate, and to transfer the device to a softer substrate. A final removal of the water-soluble tape with water droplets yields the final self-standing flexible electronics. This method is adopted for the purpose of this thesis.

As mentioned before, such a process mostly requires a cleanroom environment to achieve higher accuracy and quality. Vacuums, chemicals, and other laboratory setups are considerably expensive, which yields a concern that for fast-prototyping purposes, such a process would not be cost-efficient.

2.1.1.3 Laser Patterning

The need for a more cost-efficient prototyping process attracts scholars to the idea of using direct laser patterning. The requirement of this process is that the patterning material can be activated, cross-linked, or melted by focused incidence of a laser. Yeo et. al. demonstrated a laser direct patterning method using silver nanoparticles and semiconducting polymers, with comparative resolution to the photolithography process [7]. The silver nanoparticles are melted upon incidence of laser (continuous wave green wavelength) to form continuous metal traces, which serve as the electrical circuitry. The semiconducting polymer, poly-4-vibylphenol (PVP), is also laser patterned to form the transistors required for functionalities. The substrate used in this
study is polyimide, since it is stable at the melting temperature of the silver nanoparticles. In another report by Ko et. al., a similar approach was conducted [8]. Instead of silver nanoparticles, they used gold nanoparticles to form the circuitry. An argon laser was used to enable melting of the nanoparticles to form continuous lines. An interesting topic of this work is that the laser patterning technique was used to aid with the direct printing technique, making the printed circuitries more consistent and robust. Others have also developed methods of laser-induced graphitization, to enable the usage of graphite-like materials for sensing and other functionalities [9]. However, due to the requirement of interacting with incidence lasers, the types of materials that can be used in this process are still limited until further development.

### 2.1.2 Applications Realized by Flexible E-Skin

Recent studies of the applications for flexible electronics have been focused on the medical field, targeted at mimicking, and interacting with human skin. The types of signals harvested are summarized into three major categories: electrical, physical, and chemical signals [10], with the physical measurement closely related to the topic of this thesis. Applications on physical measurement have included strain [11] [12] [13], pressure [14] [15] [16], temperature [17], light [18] [15], vibration [19] [20], etc. Such measurements have also served as the environment input of a feedback loop of many soft robotic projects [21], making it more promising to act as structural e-skin.

### 2.2 Review on Deployable Structures

Deployable structures have been a crucial development in space structure and vehicle design. Fig. 5 shows an image of the Starshade (currently under development by NASA), which
is designed to function as a telescope to enable direct imaging of exoplanets of interest. The designed structure is capable of expanding to the size of a 10 m disk to provide higher resolution of imaging.

Fig. 5 Starshade in deployment stage. Image credits: NASA Exoplanet Exploration.

Many other strategies have also been demonstrated in recent year publications. Gdoutos et. al. demonstrated a two-step deployable structure prototype, where the structure is first folded along design battens through mechanical hinges, and then is coiled to a compact hub through bending of membrane materials [22]. Deleo et. al. introduced a combination of the fabrication process of CFRPs and origami inspired tessellation designs to achieve deployable CFRP composite structures [23]. Liu et. al. provided an in-depth analysis of shape accuracy optimization of the mechanical-hinge-based deployable antenna [24]. Numerous articles have also been dedicated to investigating deployable booms and springs as structural hinges [25] [26] [27]. Due to the discrepancy in deployment strategies, it is currently not possible to develop a sensing strategy that suits all. Therefore, I will be focusing on deployable structures using deployable booms, particularly tape-
spring-based hinges.

2.2.1 Tape Spring Hinges

This thesis focused on the analysis of a tape-spring hinge, which is a commonly used structure model for many deployable applications. A detailed analysis of a standard tape-spring structure can be found in Soykasap’s publication [28]. A tape-spring hinge adopts the shape of a section of a cylindrical shell. Two bending conditions are generally considered for this structure, shown in Fig. 6. Upon bending, the change in the pre-existing curvature within the deformed region stores energy by inducing moment against the motion of bending, for later self-deployment.

![Folding of a tape spring](image)

Fig. 6 Folding of a tape spring (a) equal sense bending, (b) opposite sense bending. [28]

The moment required for bending a tape spring is also well studied, to form a curve shown in Fig. 7. Equal sense bending takes smaller amount of moment to reach the steady state, whereas opposite sense bending requires much larger moment. It is also important to notice that a “snap” of moment drop occurs at maximum moment applied for opposite sense. Such phenomenon indicates that, for a real application scenario, if the hinge is bent in an opposite sense fashion, the structure will suddenly deform after reaching the maximum moment and requires less moment to withhold the folded state. The analysis performed by Soykasap also provides information on how to design a multiple hinge system with mathematical supports, but it is out of the scope of this
Recent studies have also made an effort to complicate the design of a tape spring in order to achieve much stronger and controllable hinges. Mallikarachchi has presented an ultrathin composite tape spring hinge. The tape spring hinge is made out of carbon fiber reinforced polymer (CFRP), and essentially connects two tape spring at both ends with enlarged round ends. The fabricated hinge is shown in Fig. 8. A finite element method is provided in this study to accurately demonstrate the folding and deploying profile of the structure during a quasi-static motion [29]. The experimental setup includes mounting the structure to a station which has embedded strain gauges. A later publication by the same author further demonstrated the same capabilities with dynamical motions [30]. Images of a deployed state during the dynamical motion are captured using a high-speed camera.
2.2.2 Structural Health Monitoring

Structural health monitoring (SHM) has been an emerging field in recent years, due to the arising demands for long-term, non-destructive in-situ testing on structures [31]. The target information includes but is not limited to structural dynamics, materials, signal processing, and microelectronics. During the cycle of stowage and deployment for deployable structures, fatigue can easily propagate through the structure. This is a result of material decomposition, material fatigue due to cyclic strain development, and material loss due to abrasive shear effects. These fatigue mechanisms eventually lead to inaccurate structural behavior or even catastrophic structural failure. Although an SHM system, to a large extent, has a similar functionality as currently developed non-destructive testing methods, it is not possible to simply miniaturize some of the current devices to integrate with the structure for long-term monitoring [31]. Therefore, novel approaches are in demand.

Fig. 9 shows a summary of the main categories that concern an SHM system. For this thesis, I focused on the environmental aspect and shape sensing. The fabricated devices are designed to provide environmental information: including temperature monitoring, UV irradiation monitoring, vibration, and acceleration that the structure experiences, and shape sensing information on crease development, crease dislocation, and structure surface orientations.
2.2.3 Methods of Monitoring Mechanical Behavior of Deployable Structure

To better demonstrate the novelty and advantage of my designed monitoring approach, a number of studies targeting in-situ measurement of the mechanical behavior of deployable structure are introduced in this section. The focuses of measurement will be confined to structural motion and vibration during deployment or contraction for the purpose of this thesis.

The non-invasive approach is one of the most widely adopted approach for SHM system. The NASA roll-out solar array, one of the state-of-art structures, was tested in 2018. The structural behavior of the deployed structure was measured in two ways: accelerometers at key locations, and video of photogrammetry scattered throughout the structure [32]. This type of video-based structural monitoring is also seen in other studies. Jorgensen et. el uses a CCD camera to set up a videometry system to measure the deployment of a deployable solar array with z-fold hinges [33]. Rothberg et. el. made an effort to review the current development of laser Doppler vibrometry,
which is widely used in structural health monitoring [34]. Yu et. el. demonstrated the potential of using single-camera digital image correlation (DIC) for measuring plate vibration excited by an impulse hammer [35]. Deflectometry, as a sub-category of DIC, is also promising in measuring plate vibration without requiring a speckle pattern on the test sample surface, but rather a reflective surface [36]. However, the aforementioned optical-based measurement methods generally require that the test subject maintains a small deviation from the focus plane of the camera. Larger degrees of motion will result in distorted images for analysis. Especially for DIC based methods, quality of the speckle pattern affects the validity of the test result to a great extent. In my experience, surfaces with the following characteristics are exceptionally challenging for DIC analysis: high degree of waviness, smooth surfaces that will cause dispersion of the sprayed speckles, and non-uniform thickness.

To overcome these challenges, others have also demonstrated alternative methods. Warren et. al. studied the correlation between the stowed energy of a lenticular hinge and its deployment rate and status [37]. Talon and Pellegrino developed a computational model to help with shape reconstruction by detecting the vibration modes at different locations of a structure [38]. However, the designed circuitry was realized using traditional PCBs.

Considering the advantages and disadvantages of these alternative methods, I envision that flexible electronics would provide an excellent alternative approach. The non-invasive attachable flexible electronics can greatly reduce the possibility of inducing defects. And the attachment can be customized with various type of adhesives for different surface characteristics.
2.3 Review on Harsh Environments

Exploring distant destinations on Earth and in outer space, such as the Antarctic and Mars, is one of the most challenging and influential efforts of mankind that has greatly advanced science and technology. However, the harsh conditions experienced during these exploration tasks pose great risks to human and structure health conditions. Most of the well-known harsh conditions include extreme temperature cycling, UV irradiation, atomic oxygen, vacuum, and high energy radiation [39]. For the purpose of this thesis, I will confine the concern of harsh environments to the following: extremely low temperature, low vacuum, and UV irradiation.

The ultraviolet (UV) irradiation at these remote locations, which is often accompanied by harsh environments, is usually strong due to the absence or depletion of ozone. Studies have shown that UV irradiation is one of the primary causes of skin cancer [40] [41]. Additionally, UV irradiation can degrade polymeric materials that are widely used in aerospace, automobile, marine, and similar industries [42] [43] [44] [45] [46] [47]. For example, long-duration scientific balloons usually cruise in the stratosphere, in or above the ozone layer, where the strong UV irradiation can damage the polymeric membranes and reduce the lifetime of balloons [48]. In outer space such as low-earth orbits, UV irradiation is one of the most dangerous environmental threats to the health of astronauts and the integrity of spacecraft materials such as Kapton, Kevlar, and carbon-fiber composite [42] [44] [45] [47] [49] [50]. Therefore, it is particularly important to monitor the UV irradiation in distant locations with harsh environments, on Earth and in space, for the safety of both human explorers and engineering materials.
2.4 Review on Thin-film Carbon-Nanotube-based Sensors

2.4.1 ZnO/SWNT-based Ultraviolet Photodetector

Despite recent advances in UV photodetectors (PDs) [51] [52], three major challenges have hindered their applications in explorations of distant destinations with harsh environments. Firstly, these missions often demand extreme requirements on the weight and size of structures and instruments. Therefore, they widely employ ultrathin, ultra-flexible, ultralight shells and membranes, which can be folded and deployed on demand. The mechanical and dimensional mismatches between thick, rigid conventional UV PDs and those unusual structures have greatly limited their integration for in-situ UV monitoring. Secondly, UV light only accounts for less than 10 percent of solar light; hence, visible light can interfere with the measurements by narrow-bandgap UV PDs or commercial Si or GaP-based UV photodetectors that are not visible-blind [52] [53] [54]. Thirdly, the harsh climate including high/low temperature and low atmosphere pressure at the distant locations of interest to scientific exploration is often beyond the operating conditions of conventional UV PDs.

The results presented in this thesis aim to address the above challenges through the introduction of routes to ultrathin, ultra-flexible nanocomposites based on zinc oxide (ZnO) nanoparticle and single-walled carbon nanotubes (SWNTs) for visible-blind UV sensing in mild and harsh environments. The ZnO/SWNT-based UV PDs achieve highly stable, repeatable, and high-quality visible-blind measurements of UV intensity in temperatures from -80 °C to 100 °C and atmospheric pressure as low as 6.7 kPa. This temperature range covers the typically harsh climates in widely explored destinations on Earth and in space such as the Antarctic, Arctic, low-Earth orbits, and Mars. The atmospheric pressure of 6.7 kPa corresponds to an altitude of 18.7 km in Earth’s stratosphere, where scientific balloons often cruise [55]. Moreover, the ZnO/SWNT-
based PD can detect low UV light intensity that the commercial PD cannot reach. The ZnO/SWNT-based UV PDs can integrate with flexible near-field communication (NFC) circuits for wireless, battery-free data acquisition, potentially eliminating the cumbersome wires and reducing the weight and size [56] [57]. This wireless, battery-free optoelectronic system can also serve as a wearable platform for monitoring the UV exposure of human explorers [58] [59]. Additionally, the ultrathin, ultra-flexible ZnO/SWNT UV PDs realize non-invasive and conformal integration with other thin, intricate, flexible materials widely used in space and polar exploration, further demonstrating their potential in in-situ monitoring of UV exposure of unusual structures in demanding environments.

2.4.2 rGO/SWNT-based Strain Sensor

For strain sensing, I am using a rGO-based thin-film strain sensor. Recent studies have shown multiple methods for fabricating rGO thin-film sensors. For example, in a study performed by Ye et. el., a self-assembly approach was used to fabricate a PEI-rGO strain sensor [60]; in another study by Wang et. el., a resistive type of rGO/TPU strain sensor was used for human motion monitoring [61]. There are also studies reporting a rGO/CNT-based thin-film, in combination with metal oxide to achieve other functionalities like piezo-electric effect or gas sensing [62] [63] [64]. The strain sensor I investigated in this study is a rGO/SWNT thin-film resistive-type sensor. The rGO solution and the SWNT solution were mixed and drop-casted onto a PDMS mold in the designed area. This fabrication method is easy to be realized in mass production. After evaporating the remaining solvent, the rGO/SWNT mixture will automatically form a thin film on the substrate. The thickness of this film can be controlled by controlling the amount of liquid mixture used in the mold.
Chapter 3

Structural E-Skin for Measurements of Tape-spring Hinge Behavior

3.1 Structural Electronic Skin

Fig. 10 a) shows the schematic of the fabricated e-skin. The displayed e-skin is a three-layer structure. The polyimide film serves as a flexible substrate to support the e-skin. The copper film is pre-laminated to the polyimide film (Dupont, AR1820) and patterned to form the electrical circuit connecting all electronic components and the thin-film strain sensors (third functional layer). In the deformation region of the tape-spring, I designed the Cu/PI traces to adopt a serpentine shape in the longitudinal direction for enhanced stretchability and to be transversely connected (PI film only) at a few locations for structural integrity. This design is to maximize the allowable deformation in the longitudinal direction, while the wires can deform in the perpendicular direction of the tape-spring surface to mitigate the strain development. A detailed study on the effect of serpentine traces can be found in a previous study by Wang et. al [65]. A top view of the completed structure is shown in Fig. 10 b). Fig. 10 c) demonstrates the structure in bending condition. After releasing the bended structure, it was observed that no wrinkling formed. This implies that the strain developed within the e-skin is well distributed and prevents structure from sliding on the tape-spring surface.

The sensor system shown in Fig. 10 a) includes two MEMS-based accelerometers at two non-deformed locations on the tape-spring (both pre- and post-hinge regions), one MEMS-based gyroscope at post-hinge region, and a series of rGO/SWNT stain sensors within the hinge region. The accelerometer and gyroscope can be substituted by any other surface mount microchip sensors to realize more functionalities.
3.1.1 Fabrication of the Flexible Structural E-skin

This section will cover the fabrication method of the E-skin demonstrated in Fig. 10, including the fabrication of the flexible circuit using photolithography. And the fabrication of the thin-film rGO/SWNT strain sensors.

3.1.1.1 Sample Preparation

The Cu/PI laminate (Dupont AR1820) was attached to a PDMS coated glass slide. The PDMS coating was formed by spin coating uncured PDMS mixture (Dow Sylguard 184 silicone elastomer, with a ratio of 10:1 Part A to Part B) at 1000 rpm for 30 seconds, and was baked at 110 °C for 1 minute to allow partial curing. The attached substrate was then placed in a 70 °C oven
for 2 hours minimum to allow fully cure. The sample surface was then rinsed with acetone and IPA prior to further processing.

3.1.1.2 Selective Etching of Cu and PI film

The Cu/PI/PDMS/glass sample resulting from the previous step was patterned first using photolithography. A thin film of AZ 5214 was spun on the cleaned surface at 3000 rpm for 30 seconds. The desired circuit traces were patterned using UV exposure of 60 mJ and developed by 1:4 AZ 400K Developer. Then the exposed area of the underlying Cu film was etched with Copper Etchant Type 100 (Transene). After successful removal of all unwanted Cu traces, the sample surface was cleaned with acetone and IPA. Then, the Cu/PI film was flipped on the PDMS coated glass slide to allow back-side patterning of the PI layer.

A 50 nm Aluminum thin film was deposited on the back side of the Cu/PI substrate using Temescal Deposition tool to serve as a shadow mask for plasma etching of the PI film. The Al film mask was then patterned through photolithography (AZ 5214, 3000 rpm, 30 s; 1:4 AZ 400K Developer). Conveniently, the AZ 400K developer was able to etch the Al film while etching the photoresist. The resulting sample was then etched by Vision 320 Plasma Etching tool (45 sccm O₂, 5 sccm SF₆, 200 W, 200 mTorr, 2 hr). After removing the Al mask, the patterned Cu/PI circuit was then removed from the glass slide.

3.1.1.3 Fabrication of the Functional Layer—Sensors and Electronic Components

The required electronic components were soldered to the flexible circuit with low temperature solder paste (TS391LT, Chip Quick Inc.). Graphene oxide (GO) bought from Carbon Solution Inc was reduced by hydroiodic acid to get reduced graphene oxide. A probe sonication
dispersed 10 mg rGO powder in 100 ml DI water for 0.5 h to get GO dispersion, which was reduced by N$_2$H$_2$ at 90 °C in water bath for 12 hours. The resulting rGO dispersion was washed by vacuum filtration method using DI water and then re-dispersed in 100 ml 1% sodium dodecyl solution by probe sonication for 1 hour. Humidity sensor was prepared by casting rGO dispersion on the interdigital electrodes on PI substrate and dried it at room temperature followed by putting it in the oven and kept it at 50 °C overnight. To prepare rGO/SWNT dispersion, 3mg P2-SWNT powder and 9mg GO powder was dispersed in 100 ml DI water by probe sonication for 30 minutes. The resulting GO/SWNT was reduced and dispersed by the same process with rGO dispersion. Strain sensor was fabricated by casting rGO/SWNT dispersion on PI substrate with Cu electrodes and left air-dried at room temperature.

3.1.2 Finite Element Mechanical Analysis

A finite element analysis was performed to simulate the structure (tape-spring with e-skin attached) under bending condition. The finite element mechanical analysis was performed using Abaqus Standard/Explicit Model. The simulated tape-spring was constructed as a 3D shell extrusion with a length of 152.4 mm (6 inch) and a thickness of 0.115 mm. The curvature of the tape-spring at rest was set as 16.9 mm, with an arc span of 86 degrees. The assigned material, steel, has the following properties: a density of 7.85 E-9, a Young’s modulus of 210 GPa, and a Poisson’s ratio of 0.3. The generated part was then processed as a composite material, where partitions are formed using the e-skin design. Materials used for the e-skin are Polyimide (Young’s modulus: 2.5 GPa, Poisson’s ratio: 0.3) and Copper (Young’s modulus: 117 GPa, Poisson’s ratio: 0.3). The corresponding materials were assigned to designated partition areas to simulate the attachment of the e-skin to the tape-spring structure. Each end of the tape-spring is given a 45 degrees rotation
boundary condition to deflect the structure to a 90 degrees angle at the middle. In-plane stress and strain are calculated and exported as output. **Fig. 11** shows the output strain values in the transverse and longitudinal directions. The induced strain sits well below the elastic limit of copper (1%) [66]. Therefore, the copper-based e-skin will not experience significant plastic deformation.

![FEA on steel tape spring bending](image)

**Fig. 11** FEA on steel tape spring bending, where color map shows the induced strain in transverse (T) and longitudinal (L) direction.

### 3.2 Measurement and Analysis of Quasi-static Release

A gyroscope is used in the structure to measure rotation speed of the tape-spring hinge. Upon rotation, the voltage reading from the gyroscope (LPR503AL) can be related to the change in the angular acceleration around the x-axis and y-axis, both of which are in the plane of the structure surface. **Fig. 12** shows the rotational angle calculated from the angular acceleration. **Fig. 12 a)** shows a cyclic equal sense bending and releasing profile with the experimental setup shown in first row. The x-axis data indicates the rotational angle in the twist direction (around the longitudinal direction) of the tape-spring structure, whereas the y-axis data indicates the rotational angle in the bending/deployment direction (around the transverse direction). Similarly, **Fig. 12 b)** shows the cyclic opposite-sense bending and releasing profile, where **Fig. 12 c)** shows the cyclic twist profile.
Fig. 12 Quasi-Static release with gyroscope and rGO/SWNT strain sensor. Row 1: experimental setup. Row 2: roll rotation angle. Row 3: pitch rotation angle. Row 4: strain values calculated from sensor voltage change (black: sensor 1; red: sensor 2; blue: sensor 3). Column a: Multiple equal-sense bending-release cycles. Column b: Multiple opposite-sense bending-release cycles. Column c: Multiple twist cycles.

Additionally, three rGO/SWNT strain sensors are designed at the folded location to monitor strain development in the hinge area. Sensor 1 (black curve in Fig. 12 row 4) is aligned at the table corner so that it is relatively at the center of the deformed hinge, whereas Sensor 2 (red
curve) and Sensor 3 (blue curve) is placed along the longitudinal direction away from the hinge center. In all scenarios, the sensors have shown relatively stable strain readings, despite minor resistance shift at rest, which can be caused by insignificant over-deployment or bending of the structure. As expected, since the crease location was setup to align with Sensor 1, Sensor 1 shows the largest response which correspond to the largest strain value. The calibration between the direct sensor voltage reading and the strain value is performed by linking the largest strain possible to be developed in the hinge area with the largest voltage reading from the strain sensor when the crease location is aligned. For the twist setup, due to a minor change in the curvature at the crease location, all strain sensors show minor response against motion of the structure.

The afore-mentioned strain gauges have shown a gauge factor for approximately 50. However, due to the fabrication method, the gauge factor various for different strain gauge. In the presented cases, sensor 1 has the highest strain gauge of approximately 66, whereas sensor 3 has the smallest gauge factor of approximately 37. For comparison, the widely adopted metal strain gauges generally have a gauge factor less then ten. For example, the strain gauge provided by OMEGA (KFH series), is a copper-nickel alloy-based strain gauge with a nominal gauge factor of approximately two. On the other hand, a lot of studies have reported on novel CNT-based strain gauges. The gauge factor varies significantly based on the fabrication method and material used. For example, Liu et. el reported a graphene-based strain gauge that is grown directly on PDMS fabric though fiber assembly [67]. The strain gauge is tested to have a gauge factor value of 151. Yu et. el. demonstrated a styrene-butadiene-styrene/CNT strain gauge, fabricated via wet-spinning, with a gauge factor value of 175 [68]. However, achieving an as high gauge factor value was not ubiquitous, and the majority of the strain sensors has been demonstrated with a gauge factor value less than 100.
3.3 Study of Crease Movement

The strain sensor system apart from monitoring strain development simultaneously, is also able to detect movement of the crease. Fig. 13 a) and b) shows two different bending location of the tape-spring structure. At the beginning, when the bending location is closer to sensor 1, sensor 1 shows the strongest response as compared to other sensors, shown in Fig. 13 c). When the bending location is shifted from its original location along the longitudinal direction of the tape-spring. Strain sensor 2 and 3 starts to show increasing strain readings. Fig. 13 d) is included to be compared to Fig. 12 a4), where sensor 1 now has the smallest strain reading.

![Image](image1.png)

**Fig. 13 Crease dislocation measurement and vibration mode detection.** a) image of equal sense bending with bending location closer to sensor 1; b) image of equal sense bending with bending location closer to sensor 3; c) strain values calculated from response of sensors upon active motion of crease along the longitudinal direction (black: sensor 1, red: sensor 2, blue: sensor 3); d) strain values calculated from response of sensors for crease forming at the final location (black: sensor 1, red: sensor 2, blue: sensor 3).

The above analysis from section 3.1.2 and 3.1.3 shows that the fabricated device is capable of monitoring general mechanical movement of the structure. While the gyroscope provides
information of the deployment angle in three dimensions, the strain sensors provide information of the crease formation or relaxation in a targeted region.

### 3.4 Measurement and Analysis of Dynamic Release

This section considers the vibration of the tape-spring structure after a dynamic release. **Fig. 14 a)** shows the experimental setup for the vibration analysis. The ACF cable connection site is attached closer to the table edge on the left side of the figure. Since the accelerometer further away from this end has larger degree of motion, the following study considers majorly the output from this sensor. In all three directions of the cartesian coordinate, the accelerometer is able to generate a voltage reading which changes upon variations in the motion acceleration. When at rest, the z-axis shows a reading equivalent to 1.0 g, which correspond to the gravitational acceleration. When bended to a 90° crease angle, the gravitational acceleration will shift to be aligned with the y-axis, as shown in **Fig. 14 b)-d)**.

A zoomed-in section of the measured data immediately after the dynamical release is shown in **Fig. 14 e)**. The oscillation of z-axis voltage reading indicates a dynamic vibration of the structure, which diminishes after approximately 0.5 seconds. Upon performing an FFT analysis on the z-axis output (**Fig. 14 f)** from the selected region, three vibration modes are captured with natural frequency of 90 Hz, 179 Hz and 468 Hz.
Fig. 14 Dynamic measurements with accelerometer. a) image of experimental setup; b-d) motion acceleration calculated from accelerometer voltage output; e) z-axis acceleration voltage output immediately after release, region between the dotted lines is selected for f) an FFT analysis, which shows 3 detected resonance frequencies at 90 Hz, 179 Hz, and 469 Hz.

3.4.1 Finite Element Frequency Analysis

In order to verify the measured vibration modes, a finite element frequency analysis using Abaqus Standard/Explicit Model was performed. The simulated tape-spring has the same geometry dimensions as the one mentioned in section 3.1.2. No partitions for the e-skin was created for this analysis, due to the significant increase in computational time. Therefore, steel was the only material assigned to the structure. This model was clamped at one end with a 6.4 mm (0.25 inch) clamped region. A distributed pressure load (static, general) of magnitude 0.01 N/mm² was applied at the other end of the structure to generate an initial deflection before the frequency analysis step. The frequency analysis step uses the Lanczos eigen solver and is set to generate the first 5 eigenvalues.
The first three modes of vibration calculated by the FEA analysis is shown in Fig. 15 a)-c), with corresponding natural frequency of 101 Hz, 184 Hz, and 471 Hz. The measured frequencies match the simulation with an acceptable error, which is a result from a more complex boundary condition in real experimental setup than a simulation setup.

![Mode 1: f=101 Hz, Mode 2: f=184 Hz, Mode 3: f=471 Hz](image)

**Fig. 15 FE frequency analysis result** showing 3 resonance frequencies at (a) 101 Hz, (b) 184 Hz, and (c) 471 Hz;

### 3.4.2 Vibration Mode Detection

An additional vibration mode detection experiment is also conducted using the setup shown in Fig. 14 a). The deformed tape-spring is dynamically released, where one of the accelerometers is at the post-hinge region and one is at the pre-hinge region. Fig. 16 shows the vibration of the structure immediately after the release. It is noticeable that when the vibration amplitude is large, both sensors have in-phase acceleration readings, since the tape-spring is bouncing up away from the table surface and both sensors are moving synchronously. However, as the vibration amplitude drops, out-of-phase vibration starts to dominate the motion. A zoomed-in view of the acceleration reading is shown in Fig. 16.
On the other hand, when the structure is deployed quasi-statically, the acceleration in all three axes will diminish soon after the structure deploys at a constant speed. With the help of earth’s gravity, it is still possible to obtain information of the deployment stage and orientation of the deployed surface. However, in a gravity-absent environment, limitations are noted for using an accelerometer skin. When no gravity exists, all channels of the accelerometer will have a zero reading, regardless of the deployment stage of the structure in a quasi-static motion, leading to an insufficient data for reconstructing the structure status at all time. Therefore, a gyroscope is much more beneficial in this situation.
Chapter 4

Flexible Optoelectronics for

Visible-blind Ultraviolet Sensing in Harsh Environments

4.1 Ultrathin, Ultra-flexible, Visible-blind ZnO/SWNT-based UV PDs

Fig. 17 shows the optical image and schematic of a ZnO/SWNT-based UV PD consisting of ZnO/SWNT thin-film nanocomposite, interdigitated copper (Cu) electrodes, and a Cu thermoresistive temperature sensor, all of which reside on a flexible polyimide (PI) substrate (50 µm thick). The channel width and thickness of the interdigitated Cu electrodes are 40 µm and 100 nm, respectively. The temperature sensor (100 nm thick, 600 Ω) achieves in-situ measurements of ambient temperature.

Here, the ZnO/SWNT thin-film nanocomposites serve as the active material for UV sensing. The scanning electron microscopy (SEM) image of the ZnO/SWNT film shows the SWNT network is surrounded by ZnO nanoparticles (Fig. 18 b)), and the thickness is less than 100 nm (Fig. 18 a)). ZnO is an n-type semiconductor with a wide bandgap of 3.37 eV. It has a strong absorption in the UV region as confirmed by the UV-Vis absorption in Fig. 18 c), making
it a promising material for UV sensing. SWNT is a p-type semiconductor with diameter of 1.5~1.6 nm, corresponding to a bandgap of 0.68 eV as shown in the S11 absorption band in Fig. 18 d) [69]. The SWNT network serves as both flexible conducting electrode and p-type semiconductor in the ZnO/SWNT-based UV PDs.

Fig. 18 ZnO/SWNT nanomaterial synthesis and material characterizations. (a-b) SEM image of ZnO/SWNT film; (c-d) UV-Vis absorption of ZnO NP solution and SWNT thin film.

Current ZnO-based UV PDs largely rely on ZnO film grown by chemical vapor deposition (CVD) and molecular beam epitaxy (MBE) at high temperature on rigid substrates such as SiO₂ wafer or sapphire, significantly limiting their applications in flexible UV sensing systems as needed here [52] [70]. The fabrication methods presented here introduce a new approach to simple, low-temperature production of ultrathin, ultra-flexible ZnO/SWNT-based UV PDs on flexible substrates. In brief, a solvothermal method serves as the means for synthesizing ZnO nanoparticles
in methanol according to a previous report [71]. Adding the SWNT solution in the ZnO nanoparticle solution obtains ZnO/SWNT nanocomposite dispersion (Fig. 19).

![Fig. 19 Synthesis process of ZnO/SWNT nanocomposite.](image)

Drop-casting the ZnO/SWNT dispersion on a flexible PI substrate with microfabricated Cu interdigitated electrodes forms uniform ZnO/SWNT thin film at room temperature. The Methods section includes more details. Compared to conventional UV PDs with typical sizes of 1.0 mm × 3.0 mm × 3.0 mm, this ultrathin ZnO/SWNT-based UV PD achieves excellent flexibility. Fig. 20 a)-b) present a ZnO/SWNT-based UV PD under bending and conformal wrapping around a glass tube with an illuminating LED inside.

![Fig. 20 (a) Optical image of a ZnO/SWNT-based UV PD under bending condition; (b) optical image of a ZnO/SWNT-based UV PD wrapped around a LED, showing the flexibility of the UV PD; (c) optical image of ZnO/SWNT bended with sharp crease.](image)

Compared to brittle ZnO films grown by CVD and MBE, the ZnO/SWNT thin-film nanocomposites shows little change after more than 100 bending cycles (Fig. 21 a)). Moreover,
the ZnO/SWNT-based PD shows negligible change in both resistance and photoresponse (Fig. 21) under bending with sharp crease (Fig. 20 c)), indicating an ultra-flexibility of ZnO/SWNT-based PD. Fig. 40 includes more examples of exploiting this advantage for conformally integrating the devices onto intricate surfaces and highly flexible thin structures.

![a](image1.png) ![b](image2.png)

Fig. 21 (a) Survivability test of the ZnO/SWNT thin film up to 100 bending cycles; (b) photoresponse of ZnO/SWNTs-based PD before and under bending.

4.1.1 Fabrication and Characterization of Thin-film ZnO/SWNT UV PDs

4.1.1.1 Synthesis of Ultrathin ZnO/SWNT Nanocomposites and Fabrication of ZnO/SWNT-based UV PDs

A probe sonicator (100 W) dispersed 10 mg P2-SWNT powder (Carbon Solution) in 100 ml 1% sodium dodecyl sulfate (Sigma Aldrich) to prepare SWNT solution. To prepare ZnO nanoparticles, 0.82 g Zinc Acetate was dissolved into 50 ml methanol in a beaker followed by fully dissolving the Zinc Acetate powder at 60 °C. 25 ml methanol with 0.5 g Potassium Hydroxide solution slowly and continually flowed into the Zinc Acetate solution. After two-hour reaction, the solution became white and then was centrifuged to remove the top methanol solution. Repeating this process for three times removed the salt in the solution to obtain ZnO nanoparticles. The
resulting ZnO nanoparticles were dispersed in 25 ml methanol [71]. 1 ml SWNT solution was added in 1ml ZnO nanoparticle solution followed by bath sonication for half an hour to obtain ZnO/SWNT solution. Drop-casting the ZnO/SWNT solution on a flexible PI substrate (thickness = 50 µm) with microfabricated Cu (100 nm thick) electrodes formed a uniform ZnO/SWNT thin film at room temperature. The device was heated at 50 °C in oven for overnight and finished the fabrication of UV PD. Fabrication of the Cu electrodes started from depositing a 100 nm Cu film on the PI substrate via E-beam evaporation. Photolithography patterned the interdigitated Cu electrodes with channel length of 40 µm and the temperature sensor with resistance of 600 Ω.

4.1.1.2 Characterization of ZnO/SWNT-based UV PDs

Scanning electron microscope (SEM, Zeiss G500) characterized the morphology of ZnO/SWNT nanocomposites, and the UV-Vis spectra of ZnO solution and SWNT film was obtained by using a Cary 5000 spectrophometer (Agilent Technologies). The I-V curves in dark and under UV light were measured by Kiethley 2600 B. A power supply (TENMA Laboratory DC Power supply 72-6610) connected with a digital multimeter (National Instruments NI USB-4065) achieved measurements of photocurrent. A function generator (Keithley 3390 50 MHz Arbitrary Waveform Generator) powered a UV LED (ThorLabs LED370E) to generate UV light with central wavelength of 370 nm. To test the photoresponse in the visible region, ZnO/SWNT-based PD was irradiated by LEDs with 405 nm (Thorlabs LED405L), 430 nm (Thorlabs LED430L), 490 nm (Thorlabs LED490L), 570 nm (Thorlabs LED570L), and 630 nm (Thorlabs LED630L) powered by function generator at 2.5 V. For the measurements of photoresponse at low temperature, the UV PD was housed in a thermal chamber (Thermacraft) that used controlled liquid nitrogen flow to regulate its temperature. For high temperature measurements, the UV PD was housed in an oven.
(Fisherbrand™ Isotemp™ General Purpose Heating and Drying Ovens) that precisely controlled the ambient temperature. For measurements in low atmospheric pressure, the setup was put in a glass chamber, which was vacuumed by a mechanic pump. The pressure can reach as low as 6.7 kPa. To test the flexibility of ZnO/SWNT based UV PD, the PD was bended with sharp crease and fixed by two clips, which was irradiated by UV LED.

### 4.1.1.3 Calibration of UV PDs

A system consisting of a lens, commercial UV PD, and UV LED was used to calibrate UV light intensity. The lens was put between UV LED and commercial UV PDs to tune the focus of light and get the max signal from the UV PD. By applying voltages from 1.8 V to 3 V on the UV LED, it generated UV light with various intensities that excited commercial UV PD to produce photocurrent. The light intensity can be calculated using the equation: \( R = \frac{I}{P_{\text{light}}} \), where \( I \), \( R \), and \( P_{\text{light}} \) are the photocurrent of commercial UV PD under light irradiation, photoresponsivity of commercial UV PD, and the light power on the effective area, respectively. **Fig. 22** presents the relationship between the bias applied on UV LED and its corresponding light intensity.

![Linear fit curve for UV light intensity and voltage across UV LED conversion.](image)
4.1.2 Characterizations of ZnO/SWNT-based UV PDs in Mild and Harsh Environments

Fig. 23 presents the current-voltage (I-V) curve of a ZnO/SWNT-based PD at room conditions in dark environment and under UV irradiation (370 nm) with intensity of 172 \( \mu \text{W/mm}^2 \). The device shows nonlinear I-V curve in dark, indicating a Schottky contact between ZnO/SWNT film and Cu electrodes. Under UV irradiation, the current significantly increases due to the generation of photocarriers.

![I-V curves of the UV PD in dark and under UV irradiation (172 \( \mu \text{W/mm}^2 \)).](image)

Fig. 23 I-V curves of the UV PD in dark and under UV irradiation (172 \( \mu \text{W/mm}^2 \)).

Fig. 24 a)-e) show the photoresponses under cyclic UV irradiation with increasing intensity from 172 to 525 \( \mu \text{W/mm}^2 \) at room temperature, high temperatures (50 °C, 100 °C), and low temperatures (-20 °C, -80 °C), respectively.
Fig. 24 Photocurrent of the UV PD under cyclic UV irradiation with increasing intensity at (a) room temperature, (b) 50 °C, (c) 100 °C, (d) -20 °C, (e) -80 °C, and (d) under low atmospheric pressure (6.7kPa).

The experimental setup consists of a UV LED, a lens, and the UV PD (Fig. 25 a)). An environmental chamber and an oven house the setup to respectively obtain the measurements at low and high temperatures (Fig. 25 d)-e)). For wired measurements, the wireless DAQ system shown in Fig. 25 c) was not included. A waveform generator periodically turns on and off the UV LED at 16.6 mHz. The irradiation time is 10 s, and the recovery time is 50 s. The photocurrent shows a fast increase under UV irradiation due to the production of photocarriers in ZnO nanoparticles and the efficient separation of electron-hole pairs by the ZnO-SWNT heterojunctions. The results are highly stable and repeatable with excellent signal-to-noise ratio even at -80 °C. As the UV intensity increases, the photocurrent shows an increasing trend in all temperatures because ZnO nanoparticles generate more photocarriers. When the ambient temperature increases, the photocurrent increases at the same UV incident intensity. Both SWNT and ZnO are stable in air at a wide range of temperatures [72] [73] [74]; therefore, the ZnO/SWNT-based PDs bring great
potential to UV sensing at harsh temperatures.

**Fig. 25 Experimental setup for measurement module.** (a) The setup of the measurement system; (b) The optical image of the UV sensor device; (c) The optical image of wireless patch and reading board outside of chamber for data collection; (d) The optical image of the environmental chamber and (e) the setup for UV measurement inside the chamber; (f) The optical image of vacuum chamber with all component enclosed: UV PD, UV LED, Lens, Wireless patch, and all wiring; power cable to the UV LED was connected through ACF cable; (g) The optical image of the setup within the glass chamber.

To the best of my knowledge, this is the first report about ZnO/SWNT-based UV PDs at low temperatures. Moreover, when the UV irradiation is low (4 µW/mm²), the ZnO/SWNT-based
PD shows a clear response while the commercial PD (THORLABS FDS010) has no response at room temperature (Fig. 26 a)). The intensity vs. photocurrent relations at all tested temperatures and conditions are shown in Fig. 26 b).

![Fig. 26](image)

Fig. 26 (a) the photoresponse of wired ZnO/SWNT-based UV PD at low UV light intensity (the UV LED was supplied with 1.59 V, 1.60 V, 1.61 V, and 1.63 V); (b) correlation between photocurrent response from wired measurement and UV intensities for various conditions.

Low atmospheric pressure is another harsh environment often encountered in the exploration missions on Earth and in space such as stratosphere and Mars. Due to depletion or absence of ozone in those locations, UV irradiation poses strong threats to polymeric materials widely used in stratospheric balloons, Mars probes, and potentially the health of future human explorers [48] [55] [75] [76] [77]. Therefore, monitoring UV intensity under low atmospheric pressure is critical to the safety of instrument and explorers in those missions. The ZnO/SWNT-based UV PD shows an excellent stable and reproducible response to a cyclic UV light (370 nm) with increasing intensity from 172 to 525 µW/mm² under 6.7 kPa atmospheric pressure at room temperature (Fig. 24 f)). Evacuating the air from a glass chamber, which houses the whole measurement system, by a mechanical pump achieves an ambient pressure as low as 6.7 kPa, equivalent to an altitude of 18.7 km in stratosphere on Earth (Fig. 25 f)-g)). The results presented
here demonstrate that the ZnO/SWNT-based PDs can serve as UV sensing platforms in harsh environments such as low and high temperatures and low atmospheric pressure.

**Fig. 27 a)** shows the photoresponse of commercial PD under light irradiation from UV to visible region, showing promising photoresponse in these regions. Although pure SWNTs have strong absorption in the near-infrared region, they do not generate photocurrent under UV and visible light due to the short lifetime of their photocarriers [52]. As shown in **Fig. 27 b)**, the ZnO/SWNT-based PD shows excellent response to 370 nm but negligible response to visible light ranging from 405 nm to 630 nm, which is in consistent with previous report. **Table 1** summarizes the photoresponse of ZnO/SWNTs under different wavelength with various light intensity.

**Fig. 27 Photoresponse** of (a) commercial PD and (b) ZnO/SWNT-base PD under irradiation with different wavelength.
Table 1 Summary of photoresponse of commercial PD and ZnO/SWNT-based PD under light irradiation with different wavelength.

<table>
<thead>
<tr>
<th>Wavelength</th>
<th>370 nm</th>
<th>405 nm</th>
<th>430 nm</th>
<th>490 nm</th>
<th>550 nm</th>
<th>570 nm</th>
<th>630 nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Light intensity (uW/mm²)</td>
<td>108.7</td>
<td>224</td>
<td>178</td>
<td>78.8</td>
<td>274.5</td>
<td>12.3</td>
<td>138.3</td>
</tr>
<tr>
<td>Commercial PD (uA)</td>
<td>4.27</td>
<td>14.09</td>
<td>20.96</td>
<td>13</td>
<td>60.35</td>
<td>3.09</td>
<td>41.28</td>
</tr>
<tr>
<td>ZnO/SWNT (uA)</td>
<td>90</td>
<td>1</td>
<td>~0</td>
<td>~0</td>
<td>~0</td>
<td>~0</td>
<td>~0</td>
</tr>
</tbody>
</table>

Responsivity is an important characteristic to evaluate the performance of photodetectors. It is the ratio between photocurrent and power of incident light defined as follows: \((I_{\text{light}} - I_{\text{dark}})/P_{\text{light}}\), where \(I_{\text{light}}\), \(I_{\text{dark}}\), and \(P_{\text{light}}\) are the current under light irradiation, in dark, and the light power on the effective area, respectively [78]. Fig. 28 presents the dependence of responsivity on the UV intensity (370 nm) at various temperatures. Under the same UV intensity, the ZnO/SWNT-based UV PD shows an increasing responsivity with the increase of temperature. In the same environment, it exhibits a decreasing trend when UV intensity increases.

**Fig. 28** Photoresponsivity of the UV PD at various UV intensities from -80 °C to 100 °C, and under 6.7 kPa pressure (6.7 kPa).
Two factors dominate the photoresponses of ZnO/SWNT-based UV PDs. The first one is the absorption and desorption of oxygen molecules on ZnO nanoparticles [79] [80]. The ZnO nanoparticles absorb oxygen molecules that capture electrons from ZnO, forming O$_2^-$ on their surfaces and causing a depletion of electrons in the ZnO nanoparticles. Under UV irradiation, photogenerated holes in ZnO move to its surface and combine with O$_2^-$ molecules to release O$_2$, leading to the increase of conductivity. Moreover, the photogenerated electrons increase the quantity of electrons in ZnO, improving its conductivity as well [79] [81]. The second factor is the presence of rich, unique p-n heterojunctions in the ZnO/SWNT nanocomposites. The p-n heterojunctions at the interface of ZnO and SWNTs enhance the band bending in the conduction band due to the transfer of electrons from ZnO to SWNT, which can efficiently and quickly separate electron-hole pairs under UV irradiation. Therefore, ZnO/SWNT-based UV PDs show a quick response under UV light [52] [71]. After the UV light is off, both the reabsorption of O$_2$ molecules and the built-in potential at ZnO-SWNT p-n heterojunctions facilitate the recovery of photocurrent.

The difference in responsivity between high and low temperatures is mainly due to the change in absorption coefficient and bandgap of ZnO, and the barrier height between ZnO and SWNTs [82]. When temperature increases, the absorption coefficient increases [83]. Therefore, under the same light intensity, ZnO generates more electron-hole pairs at higher temperatures, leading to stronger photocurrent and higher responsivity. Additionally, the bandgap of ZnO decreases when temperature increases [83] [84]. Hence, it is easier to generate photocarriers in ZnO at higher temperatures since it needs less energy to excite the ZnO. Therefore, the ZnO/SWNT-based UV PD shows higher responsivity at higher temperature. The decreasing trend in responsivity at the same temperature is when UV irradiation increases is mainly due to the traps
in ZnO/SWNT nanocomposites [52].

Measuring the resistance of the integrated Cu temperature sensor enables the in-situ sensing of the ambient temperature of the ZnO/SWNT-based UV PD. Fig. 29 a) presents the change of resistance under cyclic temperature variation between 25 °C and 55 °C. The Cu temperature sensor exhibits a linear relationship between its resistance and temperature as shown in Fig. 29 b). The photoresponses of the ZnO/SWNT-based UV PD in Fig. 26 b), in combination with the temperature-resistance relation in Fig. 29 b), provide the means for calculating the UV intensity at unknown ambient temperatures based on measured photocurrent of UV PD and resistance of Cu temperature sensor.

4.2 Wireless, Battery-free ZnO/SWNT-based UV PDs

The following study is a continuation from the previous wired analysis of the fabricated UV PDs and temperature sensors. Near-field communication (NFC) module is introduced to the system to realize wireless data acquisition.
4.2.1 Characterizations of Wireless Sensing System

The ultrathin, ultra-flexible ZnO/SWNT-based UV PDs can be integrated with near-field communication (NFC) electronics to achieve wireless, battery-free power delivery and data acquisition (Fig. 30). The wireless system consists of an NFC microcontroller, interdigitated electrodes for the UV PD, a thermoresistive temperature sensor, a conformal spiral radio-frequency antenna, and other modulating resistors and capacitors (Fig. 30 a)). A flexible PI film (20 µm thick) serves as the supporting substrate for the electronics. Fig. 30 b) illustrates the circuit diagram of the wireless, battery-free UV PD system. NFC protocols serve as the basis for its operation [85] [86] [87]. The circuit includes an antenna for wireless power harvesting and data communication, a NFC microcontroller for data modulation, a reference system for calibration purpose, and a sensor system with two bridges respectively for UV and temperature sensing. An external reader wirelessly powers the system through electromagnetic coupling between their antennas at ~13.56 MHz and simultaneously receives data from the device.

Two types of wireless UV sensing systems enable measurements of UV intensity in mild and harsh environments. An on-patch type system with sensors on the same substrate as the electronics (Fig. 30 c) and e) serves as the platform for UV sensing in mild environments. The operation temperature of the NFC microcontroller is between -20 °C and 70 °C. Therefore, an off-patch system with sensors on an extended, external cable serves as the platform for measuring UV intensity in harsh environments (Fig. 30 d) and f)). The UV and temperature sensors are exposed to harsh climates such as extreme temperatures and vacuum, while other electronics such as the NFC microcontroller are in mild environments. This architecture represents the typical scheme for monitoring harsh environments – the sensors need to be exposed, but other sensitive electronics can be housed in regulated mild environments such as in spacesuits and the body of a Mars rover.
Fig. 30 Wireless, battery-free, flexible optoelectronic systems integrated with ZnO/SWNT-based UV PDs. (a) Exploded view of the schematic for the wireless optoelectronic system consisting of off-the-shelf electronic components, a ZnO/SWNT-based UV PD, a temperature sensor, an NFC antenna, and a PI substrate; (b) Circuit diagram showing the operation principles of the system. (c-f) Schematics and optical images of on-patch and off-patch systems.

4.2.2 Characterizations of Wireless DAQ System

4.2.2.1 Fabrication of NFC Electronics

The fabrication of the NFC circuits follows the same process of processing Cu/PI laminate films. For on-patch NFC systems, an E-beam evaporation process deposited a 100 nm Cu thin film
on top of the previously defined Cu patterns. Photolithography and wet etching processes defined the electrodes for the UV PD and temperature sensor. Then, all other electronic components were bonded to the circuits using a rework station and a low-temperature solder paste (Chip Quick Inc.). The details of the electronic components are in Table 2. Drop-casting the ZnO/SWNT solution on the electrode region finished the preparation of the on-patch NFC ZnO/SWNT-based UV PDs. For the off-patch devices, an E-beam evaporation process deposited a 100 nm Cu thin film on a separate PI thin film (Dupont, 50 µm thick). Photolithography and wet etching processes defined the electrodes for the UV PD and temperature sensor on this separate PI substrate. Then, after the same bonding and drop-casting processes as the on-patch devices, an anisotropic conductive film (ACF) cable was thermally bonded to the UV and temperature sensors and the NFC circuits, connecting the sensors to the circuits.

4.2.2.2 Design of NFC Antenna

The dimension of the conformal antenna was designed according to the application note from STMicroelectronics. The antenna consists of 12 loops of circular traces. The external and internal diameters are 12.8 mm and 16.5 mm, respectively. The circuit diagram is in Fig. 31. The corresponding electronics are in Table 2. The resulting LC circuit, when connecting the antenna to NFC the microcontroller, obtained a resonance at 13.7 MHz.
**Fig. 31 Schematic of the off-patch wireless device design.** The red boxes define locations of the soldered electronic component (details shown in Table S2).

**Table 2** The components of wireless circuit (corresponding component location is shown in Fig. 31).

<table>
<thead>
<tr>
<th>Component</th>
<th>Specification</th>
<th>Detail</th>
</tr>
</thead>
<tbody>
<tr>
<td>R1</td>
<td>100 kΩ</td>
<td>RC0201FR-07100KL</td>
</tr>
<tr>
<td>R2</td>
<td>10 kΩ</td>
<td>RMCF0201FT10K0</td>
</tr>
<tr>
<td>R3</td>
<td>1 MΩ</td>
<td>RC0402FR-071ML</td>
</tr>
<tr>
<td>R4</td>
<td>0 Ω</td>
<td>RC0402JR-070RL</td>
</tr>
<tr>
<td>NFC Microcontroller</td>
<td>N/A</td>
<td>RF430FRL152H</td>
</tr>
<tr>
<td>C1</td>
<td>100 nF</td>
<td>GRM033R61A104ME15D</td>
</tr>
<tr>
<td>C2</td>
<td>10 nF</td>
<td>GRM033R61E103KA12D</td>
</tr>
<tr>
<td>C3</td>
<td>2.2 μF</td>
<td>GRM155R60J225KE95D</td>
</tr>
<tr>
<td>C4</td>
<td>1 μF</td>
<td>GRM155R70J105KA12J</td>
</tr>
</tbody>
</table>

**4.2.2.3 Characterization of NFC Antenna and Operation of NFC Data Acquisition**

To measure the S11 transmission parameter of the NFC antenna, a miniVNA Tiny (mini Radio Solutions (Mrs)) was connected to a loop formed by a short wire (5 cm length) through an SMA cable. The NFC antenna with all modulating electronics was put at the center of the loop.
Sweeping from 10 MHz to 20 MHz obtained the attenuation of the NFC antenna around the targeted frequency (13.7 MHz). A negative attenuation in dB indicated a successful transmission of signals to the NFC microchip (Fig. 32). The wireless UV sensing systems based on NFC protocols can establish stable power transmission and data communication with the reader when it is bent to a large curvature, even though there is a shift in the reading frequency and a decrease in attenuation.

![NFC antenna frequency performance under bending](image)

**Fig. 32 NFC antenna frequency performance under bending** (black-blue-green shows frequency shifting with increasing bending curvature; black-red shows decreasing in performance with increasing in distance between the patch and the reader.

4.2.2.4 Characterization of UV PDs Using Wireless DAQ System

For wireless UV sensing system, the ZnO/SWNT-based PD was first calibrated using UV LED by finding the voltage change under different, known light intensity. **Fig. 33** presents the relationship between the light intensity and photovoltage in wireless system at room temperature. The light intensity can be obtained from the known output voltage.
The NFC microcontroller stores voltage readings from the three ADC ports (marked in Fig. 30 b) and can be accessed using the GUI software from Texas Instrument. The ADC0 channel measures the voltage across R4 which is half of the supply voltage (VDD), since R3 and R4 was chosen to be the same. Then the sensor resistance (R_{sensor}, either temperature sensor or UV photodetector) is calculated by using the following equation:

\[ \frac{R_{sensor}}{R_{1,2}} = \frac{V_{ADC1,ADC2}}{V_{DD} - V_{ADC1,ADC2}} \]  

(1)

where \( V_{ADC1,ADC2} \) refers to the voltage reading for port ADC1 or ADC2, and \( R_{1,2} \) refers to the known resistance for R1 or R2 (shown in Fig. 30 b)), VDD is the total voltage.

For the UV photodetector, the voltage reading is further converted to current through the sensor:

\[ I_{UV\ PD} = \frac{V_{ADC2}}{R_{UV\ PD}} \]  

(2)

The current change (\( \Delta I \)) is then calculated by subtracting the baseline (current when in dark) from the calculated current when the UV PD is under UV irradiation. For the temperature sensor, the resistance change is calculated by subtracting resistance of the temperature sensor at 25 °C from the resistance at desired temperature, where the resistance is obtained from equation 1.
4.2.2.5 Finite Element Mechanical Analysis

The bending of the NFC device in Fig. 34a) is simulated using Abaqus Standard/Explicit. Thin plate buckling was selected to model the device bending under compression from fingers. The composite instance in Abaqus served as a proper representation of the device, with materials assigned to designated areas. The initial condition of analysis included a pair of compressive forces (1 N/mm) acting on opposite edges of the patch. Linear static simulation was carried out using ABAQUS/Standard solver. The mesh was constituted of conventional elastic elements with reduced integration (S4R) with a uniform element size of 0.1 mm. The analyses were performed on a high-end Dell server with 32 cores (Intel Xeon Gold 6144), 1 NVIDIA Tesla P100 GPU, and 384 GB RAM for fast simulations. FEA simulations calculate the strains in the device under large bending, showing that the strains are well below the elastic limit of the Cu thin films (1%) (Fig. 34 b)) [66].

Fig. 34 (a) Optical image of a bended on-patch system; (b) FEA results of the strain distribution of the on-patch system under bending.
4.2.3 Wireless ZnO/SWNT-based PDs Serving as UV Sensing Platforms in Harsh Environments

The off-patch UV PD (Fig. 30 f) serves as the platform for wirelessly measuring UV intensity at temperatures from -80 °C to 100 °C and under low atmospheric pressure of 6.7 kPa. The UV PD and the temperature sensor are placed in harsh environments, and the other electronics are placed under room condition. Fig. 35 presents the change of current in the UV PD of the wireless system under cyclic UV irradiation at room temperature (25 °C), high and low temperatures (100 °C, 50 °C, -20 °C, -80 °C), and under low pressure, respectively. Fig. 25 c)-g) show the experimental setup for tests under various temperatures and low atmospheric pressure. A thermal chamber provides low temperatures from 25 °C to -80 °C via controlled liquid nitrogen flow, and an oven generates high temperatures from 25 °C to 100 °C. A waveform generator periodically powers a UV LED at a frequency of 16.6 mHz. By applying various voltages from 1.8 V to 2.6V on the UV LED, the UV light intensity changes from 65 µW/mm² to 525 µW/mm².

![Image](a)

![Image](b)

![Image](c)

![Image](d)

![Image](e)

![Image](f)

**Fig. 35 I-T responses of the wireless, battery-free ZnO/SWNT-based UV PD in room and harsh environments:** (a) room temperature; (b) 50 °C; (c) 100 °C; (d) -20 °C; (e) -80 °C; (f) low pressure. The data was collected with cyclic UV irradiation with a maximum intensity of 700 µW/mm².
The relationship between the change of current in the UV PD of the wireless system and the UV intensity in both mild and harsh environments is shown in Fig. 36 a). The Cu-based temperature sensor in the wireless system shows a linear curve dependent on temperature as shown in Fig. 36 b). Here, the resistance of the temperature sensor at room temperature (25 °C) works as the baseline. The result in Fig. 36 b) is calculated by subtracting the baseline-resistance (25 °C) from the resistance at desired temperatures. The results presented here show that these systems can serve as wireless, battery-free UV and temperature sensing platforms in harsh environments.

![Fig. 36](image)

Fig. 36 (a) Correlation between current response and UV intensities for various conditions; (b) the resistance change of temperature sensor under different temperature.

### 4.2.4 Wireless ZnO/SWNT-based PDs Serving as Wearable UV Sensors for Human Explorers

The flexibility, light weight, and thinness of the wireless ZnO/SWNT-based PDs bring great potential to wearable optoelectronics for monitoring UV exposure of human explorers. Fig. 37 a) shows an on-patch wireless ZnO/SWNT-based UV PD attached to a shirt. Fig. 37 b) presents an off-patch device with its UV and temperature sensors attached outside of a heavy-duty coat and other electronics shielded inside the coat. The wireless ZnO/SWNT-based UV PD is exposed under solar light in the morning, at noon, and in the afternoon in State College, PA (40.79° N,
77.86° W) in October 2019 (Fig. 37 c)). Fig. 37 d) includes the V-T response of the device to solar light at 1:00 pm. A black box repetitively covers the device so that the cyclic response can be obtained.

Fig. 37 Wireless, battery-free ZnO/SWNT-based UV PDs serving as wearable UV monitoring systems. (a) An on-patch type wireless system attached on a shirt for measuring UV intensity in solar light; (b) An off-patch type wireless system on a heavy-duty coat with the sensors exposed outside and other electronic shielded inside; (c-d) Location of measurements and the corresponding V-T response at 1:00 pm;

Fig. 38 a)-c) include the cyclic V-T responses at 11am, 3pm, and 4pm. The V-T responses are highly stable and repeatable. The UV light intensity in solar light can be calculated by calibration curve in Fig. 38 d).
Fig. 38 Direct measurement of voltage responses of wireless UV PD under solar light irradiation at (a) 11 am, (b) 3 pm, and (c) 4 pm; (d) linear fit curve of output voltage change in wireless device and light intensity.

Fig. 39 presents the voltage output and the calculated corresponding UV intensity in solar light from 11 am to 4 pm, respectively. Under solar light irradiation, the wireless device generates photovoltage, and its corresponding light intensity can be calculated based on the calibration method in Methods section. The UV irradiation under 370 nm in solar light reaches a maximum of 80 µW/mm² at 1 pm and then decreases to 30 µW/mm² at 4 pm.
4.2.5 Conformal Integration of ZnO/SWNT-based UV PDs on Complex and Foldable Structures

The ultrathin thickness and flexibility of the ZnO/SWNT-based PDs realize conformal integration to complex and foldable structures, introducing new routes to in-situ, integrated UV monitoring for demanding applications. The ZnO/SWNT-based UV PD can be wrapped on a small tube with outer diameter of 1.5 mm in Fig. 40 a), showing it can be used in a highly confined space. The wireless UV sensing system can also be attached to a non-planar surface such as the off-patch device on a 3 cm diameter cylinder (Fig. 40 b)).

Space systems extensively employ foldable and deployable ultralight structures due to the extreme limits on their size and weight. Typical examples include the PI membranes of the sunshields of James Webb Space Telescope, the tape-spring booms of Hubble Space Telescope for deploying solar blankets, and widely used thermal blankets of spacecraft [88]. Those structures are under strong UV irradiation that can degrade their material and structural integrity. Fig. 40 c)-i) illustrate the feasibility of integrating the ZnO/SWNT-based UV PD on foldable thin membranes and shells for in-situ UV monitoring. Fig. 40 c)-d) present the stowage and deployment of a roll
of PI thin film (50 µm thick) with an on-patch wireless system. Fig. 40 e)-f) show a foldable and self-deployable tape-spring boom with an attached off-patch wireless UV PD. Fig. 40 g)-i) show the ZnO/SWNT-based UV PDs and temperature sensors attached on a radially retractable origami model. This model resembles the deployable shade of the NASA “Starshade” mission that blocks starlight for direct searching for and observation of exoplanets [89]. The results presented here, as well as the sensing capability of ZnO/SWNT-based UV PDs in harsh environments, further demonstrate their potential in serving as an active skin for in-situ UV monitoring in highly critical space systems.
Fig. 40 Conformal integration of ultrathin, flexible ZnO/SWNT-based PDs on complex and foldable structures.

(a) ZnO/SWNT-based UV PDs attached on a tube with the diameter of 1.5 mm; (b) An off-patch type wireless device attached on a cylinder with a diameter of 3 cm; (c-d) A wireless ZnO/SWNT-based UV PD adhered on a PI film in the unfolded and folded status; (e-f) A wireless ZnO/SWNT-based UV PDs attached on the highly deformable region of a foldable and self-deployable tape-spring boom; (g-i) ZnO/SWNT-based UV PDs directly fabricated on a radially retractable origami “Starshade” in deployed and folded configuration.
Chapter 5

Conclusions

This thesis focused on two potential applications of flexible electronics in the area of aerospace engineering. A fabrication method is first developed for a flexible structural e-skin, dedicated to measuring the performance of a tape-spring hinge. The fabricated devices have shown stable and promising responses in measuring structural vibration after a dynamic release, structure rotation angle during a quasi-static release, and strain development in the deformed location. The first three vibrational natural frequencies of the tape-spring hinge are successfully determined and verified by an FE frequency analysis. Both equal-sense folding and opposite-sense folding of the tape-spring hinge are studied. Although the shown e-skin is only applied to a tape-spring hinge, it is possible to adapt the design to other structures and surfaces. It is also possible to include other sensors, such as thin-film temperature sensors, gas sensors or MEMS-based sensors. This study suggests several future study directions. Firstly, further stabilizing the rGO/SWNT strain sensor can improve the reliability of the test result. The current calibration process requires an individual experiment with each strain sensor. Refinement of the fabrication method may achieve a uniform performance for each sensor. Secondly, integrating the fabrication method with additive manufacturing of metal or composite hinges to embed the e-skin to the surface of the hinges may yield more stable and accurate responses against structure motions.

This thesis also demonstrated that the capabilities for realizing ultrathin, ultra-flexible UV PDs based on ZnO/SWNT thin-film nanocomposites create opportunities in sensing UV irradiation in harsh environments. The method of material synthesis introduces a pathway for low-temperature, simple fabrication of ultrathin ZnO/SWNT nanocomposites on flexible substrates. The UV PDs show stable, repeatable, and high signal-to-noise-ratio responses to UV light in room conditions,
in harsh temperatures (-80 °C to 100 °C), and at low atmospheric pressure (6.7 kPa, corresponding to an altitude of 18.7 km in stratosphere on Earth). Flexible NFC electronics realize wireless, battery-free power delivery and data acquisition, further expanding the potential applications of the ZnO/SWNT-based UV PDs. The additional ability to integrate these systems onto intricate surfaces and foldable thin structures foreshadows further opportunities for in-situ UV sensing in highly demanding applications such as space telescopes, health monitoring of astronauts, aircraft and spacecraft structures, Mars rovers, and long-duration stratospheric balloons.
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