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The Application of a Triboelectric Energy Harvester in the Packaged Product Vibration Environment

Andrew Lee Berry
Clemson University, albarry@g.clemson.edu

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Abstract

Smart packaging technology is growing every year, complemented by the development of micro-electronic devices. These two trends in innovation create unique capabilities for monitoring and tracking packaged products in transit. Developing in tandem with this momentum of invention and micro-scaling of technology is the need for innovative ways to power these devices. This paper details a novel system that harvests energy from the vibration inherent in the transportation of packaged products, stores it, and uses it to power sensors that measure the very same environment from which the energy is harvested. Also accomplished in this research is the exploration of the physical and electrical durability of the energy harvester, as well as its sensitivity to environmental relative humidity. A triboelectric energy harvester converts mechanical energy to electrical energy, which is then collected and used to charge a rechargeable energy cell. This energy cell may then be used to power small electronic devices for a myriad of applications, such as temperature and humidity sensors, accelerometers, or GPS tracking devices. This energy harvester is constructed in the form of a tier sheet to be used within a unit load, replacing a corrugate sheet with a device that achieves the same purpose, while enabling power generation. This research details a unique use of the triboelectric energy harvesting method in its application in packaged product distribution, as well as conditions, such as physical durability of the harvester and humidity of its immediate environment. The triboelectric energy harvester developed is experimentally validated for use in generating power sufficient to charge a coin cell battery capable of powering various field data recorders, the requirements of which are detailed in this manuscript.
Dedication

To my mother, Susan Barry: the gold standard (Of true Christian love, joy, peace, patience, kindness, goodness, gentleness, and self control). I was once told that you and I were the same person. It is the best compliment I will ever receive.
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I Thank my family: Susan; John, Hannah, Emma, and Caroline; David and Katie. You people get me (And also support me and all of the other good stuff).

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Chapter 1
Introduction

The current growth of smart packaging technology is staggering. “Intelligent packaging demand will record double-digit annual gains, reaching $1.3 billion in 2017,” according to a 2014 Packaging Digest report [1]. Another trend affecting packaging, reported by the FDA in 2006, is that “US businesses lose up to $250 billion of profit due to the counterfeit drug trade every year” [2]. Together, these two trends increase the use of small electronic devices in packaging today. Some examples of smart packaging include smart labeling, oxygen and moisture control, counterfeit prevention, and vibration and shock monitoring of unit loads in distribution. Small electronic devices used to prevent counterfeiting are RFID tags, designated product codes (“track-and-trace”), and GPS units. This increasing use of devices in packaging is expected to continue as capabilities increase and size and cost of these devices decrease. All of the technology advancements above have one major limitation in common: they use batteries as their power source. The necessity of replacing or recharging batteries results in limited run times and requires additional costs when batteries must be replaced. Also progressing at an incredible rate is the development and implementation of energy harvesters for real-world applications. Surprisingly, these trends have developed independently of one
another, and as yet, no energy harvesting methods have been applied to address the power needs of smart packages in packaged product distribution.

The research presented in this manuscript details the use of triboelectric energy harvesting in the packaged-product distribution environment. This energy harvester is built into the form of a tier sheet, and is designed for the purpose of converting mechanical energy (in the form of truck vibration) into electrical energy to be used in the charging of an energy cell. Multiple applications have been selected for the use of these charged batteries. Another focus of this research is the exploration of the physical durability and relative humidity sensitivity of the triboelectric energy harvester. The objective of this work is to prove this concept, and demonstrate that the system described above is capable of harvesting energy, and of charging rechargeable energy cells, or batteries.
Chapter 2

Review of Literature

2.1 Energy Harvesting

2.1.1 An Introduction to Energy Harvesting

A basic definition of energy harvesting is the conversion of one form of energy into another for a designated purpose. With this simplistic definition of energy harvesting, the mechanism used is usually harvesting mechanical energy in one location and using it for mechanical motion in another. An example of this is the windmill, which uses the force of the wind to turn its gears and mix or grind grain at its base. In a modern setting, energy harvesting can be much more complex. A modern definition of Energy Harvesting is “the conversion of ambient energy present in the environment into electrical energy [3].”

There are four main ambient energy sources available for energy harvesting in the environment. These are: mechanical energy (such as vibration, shock, deformation of materials, and the flow of wind and water); thermal energy sources (temperature gradients); radiant energy (such as solar and infrared radiation, as well as radio waves); and chemical energy (chemical reactions and biochemical processes [4]). There are energy harvesting methods that are used for every one of these sources, most of which require specialized materials and processes. The environment often limits the method of energy harvesting to be used for an application, as these ambient sources are not available in every location, or during every season of the year [4]. For the purpose of harvesting
ambient mechanical energy from the transport environment, the focus of this study, vibration energy harvesting methods are the most promising [5-7]. Reviews of the most common methods of vibration energy harvesting have been published [5-7]. These three methods are electrostatic [9-12], electromagnetic [13-17], and piezoelectric energy harvesting [18-29]. Recently, a fourth category, triboelectric energy harvesting, has emerged [30-61]. In the following section, each of these vibration energy harvesting methods is described, followed by an in-depth review of triboelectric energy harvesting, the focus of this study.

2.1.2 An Overview of Modern Vibration Energy Harvesting Methods

Electrostatic Energy Harvesting

Electrostatic energy harvesting generally uses structures that are composed of two metal capacitor plates that are isolated from one another by air, a vacuum, or some other type of insulator, often dielectric materials [7]. These two capacitor plates are electrically charged with equal, but opposite charges, which creates an electric field between the charged plates. Physical separation of these plates after charging generates current [8]. An input that would cause these two plates to constantly move relative to one another would generate a significant amount of electricity. Vibration is generally used to provide this motion [6]. Electrostatic energy harvesters designed to directly power MEMS devices have been developed [9-12]. Electrostatic energy harvesters are typically intricate metal structures that are attached to a battery for the purpose of charging the capacitor plates [7]. For this reason, traditional electrostatic energy harvesting, as described above, is not an ideal method for application in packaging distribution.
Electromagnetic Energy Harvesting

Electromagnetic energy harvesting is based on Faraday’s law, which states that when an electrical circuit (coil) is located in a magnetic field, movement of the coil or change in the magnetic field results in a generated charge [7]. Electromagnetic energy harvesting can be applied to multiple ambient energy sources, the most common of which are vibration and radio waves. In the case of vibration, the physical movement of a magnet or the coil causes a change in the electromagnetic field, a response that is consistent as long as the coil and magnetic field are consistent [6]. Using vibration or physical motion to cause this interactive motion of the magnet and coil to occur many times per second can generate large amounts of electricity over time. As is the case with electrostatic energy harvesting, electromagnetic energy harvesting uses many materials that are difficult to integrate into a packaging system, and the circuitry involved can be very complex [8]. In addition, the typical resultant power of electromagnetic energy harvesters is between 0.5 V and 2.0 V, which is lower than many small electronic devices need in order to be powered, which indicates that additional equipment, such as a step-up voltage conditioner, may be necessary [8].

Piezoelectric Energy Harvesting

The most common of these methods, and the method that has received the most attention in research is piezoelectric energy harvesting [6]. The piezoelectric effect is a phenomenon in which certain materials become electrically polarized in response to applied mechanical strain. Many materials exhibit this behavior, all of which fall into four main categories: single crystal (such as quartz); piezoceramics (such as Lead
Zirconate Titanate (PZT); thin film (such as sputtered zinc oxide); and polymeric materials (including Polyvinylidene Fluoride (PVDF) and many other polymers [8]). Recently, a fifth category has emerged, forced piezoelectric materials, or piezoelectric foams. This category uses mostly polymers that have been treated, usually with corona discharge treatment, causing them to exhibit strong piezoelectric responses. The most common materials used in piezoelectric systems are ceramic PZT and polymeric PVDF [29]. More recently, piezoelectric foam structures have been developed, due to their relative strength compared to traditional piezoelectric materials [26-29]. Many piezoelectric foams made from Polypropylene (PP) and Polyethylene Terephthalate (PET) [29].

**Triboelectric Energy Harvesting**

In recent years, triboelectric energy harvesting, another promising method of vibration energy harvesting, has piqued interest of many researchers and experienced significant advancement in research and practice [30, 31]. Triboelectric energy harvesting may be considered a subset of electrostatic energy harvesting, due to the nature of its operation, in which an electrical field is used for the collection of energy. Triboelectric energy harvesting is based on the phenomenon of the triboelectric effect, which is “a type of contact electrification in which certain materials become electrically charged after they come into frictive contact with a different material” [32]. A common name for this is “static electricity”. It is a type of contact electrification that results from the electrical output of a polymer electret, by physical contact with another material. Upon contact, these two materials transfer electrical charge. The triboelectric effect can be seen in many
materials common to packaging, and, in fact, nearly any material can participate in contact electrification. Though any material could be used, these harvesters commonly use materials such as Polydimethylsiloxane (PDMS), Glycol-modified Polyethylene Terephthalate (PETG), Polymethyl methacrylate (PMMA), or Polytetrafluoroethylene (PTFE), which are then paired with a conductor such as Silver (Ag), Aluminum (Al), Copper (Cu), Nylon, as these material combinations yield the best results [30, 31]. Using these materials, triboelectric energy harvesters can easily be built into a myriad of structures, and therefore may be used in a package system.

Triboelectric energy harvesters have been significantly improved in recent years, and have been applied to many applications. The majority of research on triboelectric energy harvesters is simply on the development and optimization of these harvesters in terms of power output, contact geometry, and energy sources from which they are able to generate electricity [30, 31]. In recent years, progress has been made in the development and advancement of triboelectric energy miniaturized triboelectric energy harvesters, called triboelectric nanogenerators (TENG) [30, 31]. A great number of materials found in the triboelectric series have been used in experimentation, as well as a number of surface morphologies, all in an effort to enhance the capabilities of these generators by maximizing charge. In addition to harvester design, advancements have enabled harvesting mechanical energy from human motion such as finger tapping or walking [33-41], the rotation of a wheel or tire [42-45], shock events [46], vibration, in multiple forms [47-51], bending and deformation of a material [52], wind [53-55], and flowing water [56, 57]. Triboelectric energy harvesting has also been combined with other methods of
energy harvesting in order to gain the benefits, while mitigating the downsides of both types. Examples of this include coupling piezoelectric and triboelectric vibration harvesting [58, 59], and the coupling of electromagnetic and triboelectric harvesting for self-powered sensors [60, 61].

2.1.3 Understanding the process of triboelectrification

*Contact electrification*

Triboelectricity is a type of contact electrification that results from the electrical output of one material when physically contacted with another material. Contact electrification is “A process that produces surface charges on two dissimilar materials when they are contacted and separated. During this contact, each material develops a charge of opposite polarity” [62]. Contact electrification can take place between nearly any combination of two materials, including metals (conductors), semiconductors, and insulators (non-ionic materials). The charge transfer mechanism of the first two material types, metals and semiconductors (ionic materials), is well understood [62-65]. The mechanism for metal-metal contact is described by McCarty [63]. The nature of the charge transfer in contact involving insulators has not yet been investigated by many, but has not yet been determined.

For ionic polymers, those that have a large, covalently bound ion with small, mobile counterions at the surface, it has been shown that the charge transfer is carried out by ions. When a second, oppositely charged surface contacts the initial surface, the small mobile ions are transferred to the second surface, resulting in a transfer of charge, or
contact electrification. Furthermore, the resulting charge that is left within each material corresponds to the charge of the large, covalently bound ion [62].

Many nonionic polymers (insulators) have been shown to experience contact electrification, including PMMA, PE, PS, and PTFE [62, 63]. These polymers do not possess mobile ions on their surface, but still transfer charge. As there are no mobile ions to transfer when contacted with other materials, it has been concluded that another charge transfer mechanism must be occurring. The three proposed mechanisms of charge transfer involving insulators are ion-transfer, electron-transfer, and material or mass transfer.

*Ion transfer mechanism*

Mobile ions are not present on the surface of insulators, resulting in the exclusion of this charge transfer mechanism from consideration for some time. However, many studies have suggested that ion transfer may still take place during contact electrification involving insulators [62-65]. In any non-vacuum environment, there is water present in the atmosphere, which accumulates on the surface of materials. This has been called ‘the water layer’ [66]. It has been suggested hydroxide and hydronium atoms within this water layer are the source of this charge transfer. This is called the ‘water-bridge theory’ [66]. However, it was later shown that charge can be generated and transfer in a vacuum in the absence of any moisture, suggesting that multiple charge transfer mechanisms may take place simultaneously [63, 65].
**Electron transfer mechanism**

Liu and Bard (68, 69) have shown that this electrification is caused by the transfer of energetic electrons located on the surface of these nonionic polymers. When insulators are physically agitated, the physical contact causes stress on the structure, allowing for these electrons to be released (69). Once free, they are attracted to the positively charged contacting surface, resulting in contact electrification. This theory has been confirmed using electrochemistry to show that electron-exchange does occur between insulators [69, 70].

**Material transfer mechanism**

Surface analysis methods such as x-ray photoelectron spectroscopy (XPS) have been used to show that the surfaces of both materials involved in contact electrification involving insulators are capable of transferring surface molecules to the opposite material surface [71]. These molecules transferred naturally carry a charge. By this same process, material transfer has been shown to be very localized on the surface of the material, resulting in localized groups of both positive and negative charges on both material surfaces [71, 72]. This has been called a ‘surface charge mosaic’, referring to the appearance of these localized charged material surfaces when using XPS [72]. This led to the conclusion, “Mass [material] transfer, therefore, cannot be ruled out as the mechanism of charge transfer in triboelectric phenomena…. Mass transfer must be considered in models of contact charging, simply because of the great degree to which it occurs [71].”
Evidence supporting each of these three charge mechanisms has been reported and confirmed. It has been suggested that the use of materials from different origins, and different contact mechanisms, contact pressures, and surface contact areas may be a cause of some of this disagreement [63]. It is possible, of course, that all three of these charge transfer mechanisms may be taking place at the same time.

**Polymer electrets**

Contact electrification with some materials results in the development of a positive or negative charge that is held within the body of the material, leaving it permanently or quasi-permanently charged. Materials that can maintain charge are called electrets. A definition of an electret is “a material that has a permanent, macroscopic electrical field at its surface” [63]. There are two classifications of electrets, dipolar electrets and space-charge electrets [63].

Dipolar electrets behave similarly to magnets in that they have a permanent, or quasi-permanent dipole across the bulk of the polymer. This means that the material has an electrically positive pole and an electrically negative pole. These may be fabricated by cooling a polymer from above its glass transition temperature (Tg) to below it, in the presence of a strong electrical field. When cooled, the material holds the electrical tendencies and has oppositely charged poles. Dipolar electrets are used in many applications, including electrostatic energy harvesting [63].

Electrets used in triboelectric energy harvesting applications are space-charge electrets [63]. Space charge electrets obtain their charges differently than dipolar: they do not have electric poles, but instead, they possess a net macroscopic electrical charge [63].
These are made from tribocharging of polymers, or the charging of polymers by means of contact electrification. The electrification occurs on contact with another material, as described in the previous section, and the electret material holds that charge. Both ionic and nonionic polymers can be space-charge electrets. Common nonionic polymers that can be space-charge electrets are Polyethylene (PE), Polystyrene (PS), and Polytetrafluoroethylene (PTFE) [63].

For a polymer to be either a dipole or a space-charge electret as described above, it must have the ability to withstand relatively large electrical potential without breaking down (high dielectric strength), and it must be able to hold that electrical potential within its structure for a long time, meaning that it must be a material with relatively low conductivity. Therefore, the stronger the dielectric strength and insulative properties of the electret, the higher the electrical potential of the contact electrification will be, and the stronger the ‘desire’ of the material to obtain or donate electrons, the higher the electrical potential of the contact electrification will be. These two concepts lead to the use of a triboelectric series.

*Material selection: the triboelectric series*

Every material responds differently and develops a level of charge following contact electrification. Many materials don’t develop a significant charge, while others develop and hold significant positive or negative charges following contact electrification. Though the exact nature of the charging may not be known in every case, there are very consistent trends across a range of materials that have been observed.
A triboelectric series is an arrangement of materials that are ranked according to the polarity of charge they develop following contact electrification [65]. In a triboelectric series, the materials listed towards the top are those that develop the most positive charge following contact electrification, while those at the bottom develop the most negative, with the middle of the series being relatively neutral in charge. Countless triboelectric series have been constructed and reported. Four of the most comprehensive series in literature have been compiled into one, noting the differences between each series [62]. A typical triboelectric series is reported in Fig. 2.1.

Questions have been raised regarding the validity of this tool, citing varying placement and charge response of certain materials in different triboelectric series across published literature. For example, Teflon® (PTFE) is known to be a non-polar polymer, but develops strong negative charges during contact electrification [62]. Another example of the limitation of the triboelectric series is the ability of two identical materials to experience contact electrification with one another, one developing a strong positive charge and the other a strong negative charge [64, 65]. Despite these concerns, triboelectric series are well established in literature and are used in material selection for many triboelectric energy harvesters, and a triboelectric series is the primary tool used for selection of experimental materials in this research.

Figure 2.1: Triboelectric Series [62]
Surface modification of electret materials: enhancing contact electrification

It is common to treat materials in order to raise the surface energy, for a variety of purposes. This is also common in the preparation of triboelectric energy harvesters, as it has been shown that raising the surface energy of the material can increase resultant triboelectric charge [75, 76]. Referring to this process, McCarty states, “Space-charge electrets result from adding charge to the surface or bulk of the material by bombarding it with an electron beam or ion beam, spraying it with ions from the corona discharge of a high-voltage electrode, contacting it directly with a charged electrode, or transferring [charge] to or from the material by other means [63].”

Tribocharging: Physical rubbing of polymer electrets

Space charge electrets, described in a previous section, are developed by tribocharging of polymers, which is the charging of polymers by means of contact electrification. Therefore, the act of contacting two surfaces in order to cause contact electrification is also the cause of the development of the electrical potential of the electret. It has been shown that repeated rubbing or contacting and separating of materials can cause the charge of the electret to become stronger, which then increases electrical output [65]. Tribocharging can be accomplished using a wide range of materials, and with a number of precise methods. The optimal method of tribocharging depends on the material being treated. Referring to tribocharging patterns, Galemback states, “The direction of charging in asymmetric contacts is materials dependent, an observation which is likely to play an important part in the eventual overall mechanistic understanding of triboelectricity. For Teflon the larger region charges positively but for
Nylon the larger region charges negatively [64].” It has been demonstrated that a PTFE sheet that has been physically rubbed causes a higher surface energy than PTFE that was not treated by physical rubbing [68].

Corona Discharge Treatment

A second method of charging polymer electrets is the use of corona discharge from a high-voltage electrode [63]. In the corona treatment process, an electrical field of very high voltage is created between a positive and a negative electrode. In the case of this research, this voltage is adjusted between 10,000 and 45,000 V at a high frequency of 4.5MHz [73]. In the area between these two electrodes, the air is subjected to this strong electrical field, causing a dielectric breakdown of the components of the air. This causes the separation of negative electrons from positive ions, and imparts a large amount of potential energy, resulting in high kinetic energy. The electrons and ions collide with others in the same area, causing the same event to occur with more electrons and ions. This chain reaction is called an “electron avalanche”. In the case of this research, the corona discharge is negative, meaning that high-energy free electrons are repelled outward from the electrodes and electrical field [74]. When this occurs close to a polymer surface, these electrons bombard the surface, causing lasting effects. The use of corona discharge to maximize charge of contact electrification is well documented, ranging a number of corona methods and materials [75-79].

In 2015, it was concluded that a combination of both of these treatment methods produces the most electrically and thermally stable electrets for use in contact electrification. For this, a two-step process is followed in which the material is first
tribocharged by physical rubbing, followed by a second step in which the material is exposed to corona discharge [79]. This process as described above was followed for the treatment of the PTFE film used in this research.

*Triboelectric energy harvesting: modes of excitation*

There are four modes of contact electrification used in triboelectric energy harvesting: vertical contact-separation, in-plane sliding, single-electrode, and freestanding triboelectric layer [31]. Each of these contact modes describes the nature of the interaction between the two triboelectric layers in its system, and therefore prescribes the basic structure and type of motion required to achieve it. For the vertical contact-separation mode considered in this study, a vertical stack design is necessary.

Vertical contact-separation mode was the first contact mode to be used with triboelectric nanogenerators (TENGs) [32]. With this vertical contact design, there must be two different triboelectric material layers with an electrode on each layer. These two surfaces need to contact and then separate for a maximum transfer of charge. Typical excitation methods for vertical contact-separation mode are vibration and shock events, brought about by finger tapping [35], human walking [33, 34], engine vibration, etc. [31].

In-plane sliding mode uses horizontal motion, rather than vertical, to achieve the charge transfer. In this mode, two triboelectric layers are placed in contact with one another, and then sliding is induced, causing relative frictive motion of the two surfaces [31]. The positive and negative charges on each surface of the two triboelectric layers in their initial positions are satisfied by the opposite charges on the opposite surface. When
lateral motion is induced, the charges are no longer perfectly satisfied, causing a transfer of charge between the two surfaces [31]. A device in which these two surfaces regularly slide back-and-forth is capable of generating a significant amount of charge. Devices using this mode of excitation typically are designed for harvesting planar or rotational mechanical energy, such as the rotation of a tire [42-46].

Single-electrode mode allows for more relative motion of the two surfaces, due to the necessity of only one electrode [31]. With this design, only one of the materials involved is attached to an electrode, allowing the other material to move more freely. This also allows for the possibility of different structural designs than are possible with both vertical-contact and in-plane sliding modes [31]. With more freedom of movement, triboelectric energy harvesters using this contact mode have been used to harvest mechanical energy from the flow of water and wind.

In freestanding triboelectric layer mode, the charge transfer takes place due to the relative motion of a previously charged triboelectric material and an electrode [31]. Oscillating motion closer and further away from the electrode causes a potential difference, resulting in the transfer of electrons from the triboelectric layer to the electrode. In this mode, there is no requirement of physical contact [31]. This mode of charge transfer can be accomplished after one of the above processes has taken place, or after charging of the polymer electret using tribocharging or corona treatment, as the material must have a charge in order for the free-standing triboelectric layer mode to be possible.
2.1.4 Conditions of Triboelectric Energy Harvesting in the Distribution Environment

Triboelectric energy harvesting is a versatile method of harvesting energy. The number of material combinations that can be used with one another, in addition to the multiple contact modes and possible structural designs highlight this versatility. There are, however, two conditions that must be met to obtain a maximum charge during energy harvesting. These are the frequency of vibration input and the relative humidity of the environment. Both of these conditions have an optimal point at which the maximum possible charge is produced, with the charge decreasing as the conditions change to other frequencies or humidities, above or below the optimal point [62, 63, 65, 80-89].

Excitation frequency

The frequency at which triboelectric energy harvesters respond best, from a charge generation standpoint, is between 15-40 Hz [80, 81]. As frequency increases above this range, the charge generation of the energy harvesting system decreases. The narrow operating bandwidth discussed by many in research is not unique to triboelectric energy harvesting, but is also a design challenge for electromagnetic and electrostatic energy harvesters [81]. Many have successfully combined energy harvesting methods or developed damping systems within their energy harvesting structures to expand this operating bandwidth [82-84].

The focus of this research is the application of an energy harvesting system to the packaged product distribution environment. For this reason, the frequency of vibration typical to this distribution environment is important. It is known that the non-stationary
random vibration and shock events that describe this distribution environment impart vibrations from 1-100 Hz. [85, 86]. Other modes, including rail, sea, and air transport, impart similar forces to packaged products in distribution [85, 86]. The frequency response of the triboelectric harvester used in this research is described in Fig. B-1, in Appendix B.

**Humidity Concerns**

Many have noted that humidity of the triboelectric energy harvester’s immediate environment has an effect on contact electrification, and that it is essential for contact electrification to take place [62, 63, 65, 87-89]. The extent to which it effects charge generation and transfer, however, is not specified. In fact, the ideal conditions for charge generation are debated in literature. One study shows that a relative humidity (RH) of 0% yields the maximum possible charge, and that charge generation steadily increases as the relative humidity in the immediate environment of the energy harvester decreases [88]. Another study demonstrates that there is an optimum relative humidity, between 20 and 40% RH, but that the charge decreases once the humidity increases above 40% [66]. A third study reports that contact electrification can take place in a vacuum in the complete absence of any humidity in the environment, also stating that when humidity is present, charge generation is limited more by low humidity than by high humidity [87].

In a study conducted by the International Safe Transit Association (ISTA) monitoring containers travelling by sea from Asia, through Europe, and eventually to North America, the humidity fluctuated from 32% and 96% RH [89]. For the use of triboelectric energy harvesters in a packaging application, these data may be of concern.
2.2 Vibration Simulation & Energy Harvester Excitation

2.2.1 Vibration Simulation of the Distribution Environment

It is known that packages and products are often damaged or otherwise negatively affected by the forces they experience while travelling through the distribution cycle. For this reason, laboratory techniques have been developed to simulate the forces acting on these packages and products, in order to use this information to develop more robust packages and products that are able to withstand these potentially damage-causing events. Techniques exist for the simulation of all of these major forces acting on packages during transport, including shock, vibration, compression, and environmental effects. International Safe Transit Association (ISTA) & ASTM International (ASTM) both publish industry-accepted test standards [90, 91]. As vibration is the sole input used in this research for the excitation of the triboelectric energy harvester in this study, this review focuses on the laboratory simulation of transport vibrations.

There are three main types of vibration tests used in laboratory simulation, all of which can be accomplished using a servo hydraulic vibration system (Lansmont Corporation). These are the ‘fixed-displacement test’, which is also called the ‘bounce’ test (ASTM D999a), the sinusoidal (sine) tests (ASTM D999b, c), and the random vibration test (ASTM D4728) [92-94]. The fixed displacement test is accomplished by the table oscillating with a set amplitude, at a set frequency, usually between 4.4 and 4.5 Hz [94]. This causes the product or dummy product on the table to repeatedly bounce with low amplitude. The sine test is accomplished by the movement of the vibration table
in sinusoidal motion. The two categories of sine tests are the sine sweep and the dwell test. Sine sweeps are useful for determining the resonant frequencies of products. The fixed displacement and sine tests cannot be accurately called simulations of real transport vibration [94]. The third category, the random vibration test, is accomplished when the vibration table “moves with a constantly-changing complex mixture of frequencies and amplitudes, generally similar to the way transport vehicles actually move [94].”

Random vibration tests can accurately simulate the transport environment, and are the only type of vibration test that can do so [94]. “Random vibration tests are typically described by power spectral density (PSD) plots, [which are] graphs of ‘average’ acceleration intensity in the frequency domain (PSD as a function of frequency) [94].” The use of PSD plots to characterize random vibration for laboratory simulation is widely accepted [95]. Two common test methods for package distribution in which random vibration PSDs are prescribed are ASTM D4169 [96] and ISTA 3E [97]. “ASTM D4169 Truck Assurance Level II random profile may be the most widely used general simulation vibration test in the world…. It’s a bit outdated now, and there are more up-to-date-spectra available [98].” The more up-to-date spectrum referred to in the previous statement is the ISTA Steel Spring Random Vibration Profile, prescribed in ISTA 3E, Fig. 2.2 [97]. This profile is an accelerated random vibration profile made using vibration data collected from a steel spring truck, but it is known that other modes of transport, such as air, rail, and sea, produce similar forces to what is shown in this profile [85, 94, 99].
Figure 2.2: ISTA Steel Spring Random Vibration Spectrum PSD
Chapter 3

The Application of a Triboelectric Energy Harvester in the Packaged Product Vibration Environment

3.1 Abstract

Smart packaging technology is growing every year, complemented by the development of micro-electronic devices. These two trends in innovation create unique capabilities for monitoring and tracking packaged products in transit. Developing in tandem with this momentum of invention and micro-scaling of technology is the need for innovative ways to power these devices. This paper details a novel system that harvests energy from the vibration inherent in the transportation of packaged products, stores it, and uses it to power sensors that measure the very same environment from which the energy is harvested. A triboelectric energy harvester converts mechanical energy to electrical energy, which is then collected and used to charge a rechargeable energy cell. This energy cell may then be used to power small electronic devices for a myriad of applications, such as temperature and humidity sensors, accelerometers, or GPS tracking devices. This energy harvester is constructed in the form of a tier sheet to be used within a unit load, replacing a simple corrugate sheet with a device that achieves the same purpose, while enabling power generation. Many developments have been made in the field of triboelectric energy harvesting in recent years, including design and input
optimizations. This research details a unique use of the triboelectric energy harvesting method in its application in packaged product distribution. In addition, the scale and design of this tier sheet device are novel. The triboelectric energy harvester developed is experimentally validated for use in generating power sufficient to charge a coin cell battery capable of powering various field data recorders.

3.2 Introduction

The current growth of smart packaging technology is staggering. “Intelligent packaging demand will record double-digit annual gains, reaching $1.3 billion in 2017,” according to a 2014 Packaging Digest report [1]. Another trend affecting packaging, reported by the FDA in 2006, is that “US businesses lose up to $250 billion of profit due to the counterfeit drug trade every year” [2]. Together, these two trends highlight the importance of small electronic devices use in packaging today. Some examples of smart packaging include smart labeling, oxygen and moisture control, counterfeit prevention, and vibration and shock monitoring of unit loads in distribution. Small electronic devices used to prevent counterfeiting are radio-frequency identification (RFID) tags, designated product codes (track-and-trace), and GPS units. This increasing use of electronic devices in packaging is expected to continue as capabilities increase and size and cost of these devices decrease. All of the technology advancements above have one major limitation in common: they use batteries as their power source. The necessity of replacing or recharging batteries results in limiting run times and requires additional costs when batteries must be replaced. Also progressing over the last few years, is the development and implementation of energy harvesters for real-world applications. Surprisingly, these
trends have developed independently of one another, and as yet, no energy harvesting methods have been applied to address the power needs of smart packages in packaged product distribution. This paper summarizes the findings of a study of an energy harvester applied to charging small energy cells for a myriad of applications, and validated in a package distribution application. A triboelectric energy harvester is developed, an energy harvesting battery charger is designed, and the system is validated using an industry-accepted vibration test.

There are a number of energy harvesting methods that may be used for harvesting mechanical energy. The three most common methods are electromagnetic, electrostatic, and piezoelectric energy harvesting [5, 6]. The most common of these methods, and the method that has received the most attention in research is piezoelectric energy harvesting [24]. In recent years, triboelectric energy harvesting, an subset of electrostatic energy harvesting, has been developed and applied to vibration energy harvesting [30, 31]. Electromagnetic and traditional electrostatic energy harvesting are both very effective in harvesting vibration energy, but use materials and structures that are difficult to use when designing the energy harvester for a packaging application. Both piezoelectric and triboelectric energy harvesting could potentially be used in the context of packaging due to the materials used to build them, and the flexibility of structure that is natural to both methods. This study uses triboelectric energy harvesting for three reasons: the required properties necessary for energy harvesting are inherent to the materials in triboelectric energy harvesting (i.e. corona treatment is not required), triboelectric energy harvesting has not been thoroughly explored by the scientific community, and the materials and
structure used for triboelectric energy harvesting are flexible, and therefore able to be used in packaging application.

Triboelectricity is a type of contact electrification between two different materials upon their physical interaction with one another. Contact electrification is “a process that produces surface charges on two dissimilar materials when they are contacted and separated. During this contact, each material develops a charge of opposite polarity” [62]. In the case of this research, the two materials are a sheet of aluminum (Al)-coated Polyethylene (PE) and a sheet of Teflon® Polytetrafluoroethylene (PTFE). An electrical charge develops when the aluminum surface contacts the PTFE surface. Upon contact, the charge transfers between the two sheets. The force of contact and number of contacts are both factors in the amount of charge that transfers over time. Therefore, a process that provides repetitive, forceful impacts between the two materials produces a maximum amount of charge between these materials.

A triboelectric energy harvester is a device that uses the principle of contact electrification between two oppositely charged materials and can capture the electrical charge transferred between them. In order to consistently capture energy, there must be consistent contact and separation of these triboelectric materials. While the various shock inputs encountered in the distribution environment excite these harvesters, the broad-spectrum, over-the-road truck vibration is the input of focus in this study.

In recent years, progress has been made in the development and advancement of triboelectric energy harvesters, called triboelectric nanogenerators (TENG) [31]. Many of the materials found in the triboelectric series have been used in experimentation, as well
as a number of surface morphologies, all in an effort to enhance the capabilities of these generators by maximizing charge. In addition to harvester design, advancements have enabled harvesting mechanical energy from vibration [47, 48], human motion [33, 34], the rotation of a tire [42, 43], and flowing water [56]. Future work is expected to be applied to the development of self-powered sensors that are able to detect mechanical, chemical, temperature, and flow (wind and water) events [31]. An example of this type of sensor is a cylindrical triboelectric energy harvester built into the structure of a capsule endoscope (a small capsule that travels through and examines a person’s digestive tract) removing the need for an invasive procedure. Creating a self-powered capsule endoscope that is not limited by battery power would overcome a serious hurdle for the current capsule endoscopes in use [100].

In this study, various materials and surface treatments are explored in their ability to generate triboelectric charge. Two different harvester configuration designs are evaluated for charging performance. A battery charging system is developed for energy storage. The optimized harvester design is then validated using an industry-accepted test method for simulation of the vibration encountered in truck transport.

Three tests are run in which three batteries are charged from an average of about 3.0 V to 3.4 V. This not only shows that the triboelectric energy harvester is capable of harvesting vibration energy from the package distribution environment, but also that the energy harvested from this environment has sufficient amperage and voltage to charge a battery capable of powering many small electronic devices.
3.3 Design of the Harvester

Triboelectric energy harvesters rely on contact electrification to generate charge between the layers that possess a large difference in charge polarity. When these layers come into contact, the charge is able to transfer to the opposite layer and be collected by electrodes built into the harvester. Triboelectric energy harvesters require a few basic components: at least two triboelectric material layers, physical separation of these layers, and electrodes for the collection of the energy that moves between these layers. Many different materials can be used in the structure of a triboelectric energy harvester, as long as they will develop charges of opposite polarity following physical contact with one another [63]. A triboelectric series is helpful in selecting two materials that will interact well with one another for triboelectric charge [62]. A triboelectric series is simply, “a list of materials empirically ordered according to their tendency to acquire positive or negative charges subsequent to mechanical contact [64], Fig. 3.1.”
The structure used for the harvester in this study is a single vertical stack design, meaning that the layers are simply stacked atop one another. The harvester designed and built is 55.88 cm x 35.56 cm x 8.50 mm, with six layers in total: B flute corrugate board, aluminum-coated PE with attached positive electrode, PTFE, cushion layer, aluminum-coated PE with attached negative electrode, and B flute corrugate board. There are four modes of contact electrification used in triboelectric energy harvesting: vertical contact-separation, in-plane sliding, single-electrode, and freestanding triboelectric layer [31]. Each of these contact modes describes the nature of the interaction between the two triboelectric layers in its system, and therefore prescribes the basic structure and type of motion required to achieve it. For the vertical contact-separation mode considered in this
study, a vertical stack design is necessary, Fig. 3.2. This contact mode also determines the type of mechanical input required to excite the harvester. In the case of vertical contact-separation, vibration is commonly the mechanical input, though any vertical mechanical input is sufficient, i.e., shock or compression forces.

The two triboelectric materials used in the structure of this harvester are aluminum-coated PE and PTFE. The aluminum-coated PE is a 0.125 mm thick film, coated on one side with 99.7% Al, sourced from Advent Research Materials, Ltd (Oxford, England). The PTFE used is a skived, virgin, 3.18 mm thick PTFE film sourced from CS Hyde Company (Lake Villa, Illinois). These materials have been used in combination for triboelectric charging in multiple studies, and have been shown to be very effective [34]. Aluminum has a tendency to develop a relatively positive charge after physical contact with most materials, while PTFE tends to develop a very negative charge. In the two harvester designs developed, Fig. 3.3a,b, there are two layers of aluminum-coated PE, with a PTFE sheet stacked between them.
The physical separation needed for the proper function of triboelectric energy harvesting is achieved in this design by a layer of foam cushioning. Two foam-cushioning designs are tested. The first, a foam cushion border around the edges of the harvester, Fig. 3.3a, is a proposed structure used in experimentation, but is not used in the final structure design of the energy harvester. This cushion-border design allows for contact to take place in the center of all materials, while achieving the separation that is required for triboelectric harvesters to function. This design works well for charge development, but allows unwanted horizontal motion between the layers of the harvester. This horizontal friction causes the aluminum coating on the top and bottom harvester layers to degrade over time, ultimately leading to a decrease in performance. These issues are not experienced by the final harvester design that uses a different cushioning structure, in which a number of circular foam cushions are affixed to one of the aluminum-coated PE layers, isolating it from the PTFE and aluminum-coated PE sheet on the other side of these cushions, Fig. 3.3b. These foam cushions separate the layers, enabling the development of a potential difference, without allowing the unwanted friction allowed by the initial design. Upon contact of these materials, the two layers attempt to reach equilibrium by transferring positive or negative charge or both in different surface locations, and the harvester’s electrodes can then harvest the mobile charge [64].
In addition to the structural development of these harvesters, a number of surface treatments are also explored. Tribocharging, the physical rubbing or friction of two different surfaces, is used as a material treatment in order to build up an initial charge both in the body and on the surface of the materials used in this study [63]. It has been demonstrated that the tribocharging of PTFE increases its surface energy [68]. The second material treatment used in this study is corona discharge. In the corona treatment process, a highly charged electric field is created, which causes the air to experience a dielectric breakdown, meaning that the components
of the air are highly energized and the bonds are broken. The result is high-energy, free electrons being repelled outward from the electric field, contacting nearby objects. When a polymer is introduced into this area, the surface of the polymer is bombarded by these electrons and typically develops a significantly higher surface energy. As a result of this, a polymer that has been corona treated can develop significantly higher charges when contacted by a second material. This allows for better performance of these treated materials in triboelectric energy harvesting [75]. The PTFE and aluminum sheets are treated both by tribocharging and corona treatment in development of the harvester in this study. The final design of the triboelectric energy harvester uses untreated AL-coated PE and tribocharged, then corona-treated PTFE, as described by Rychkov et al. [79].

The triboelectric energy harvester is designed to mimic a tier sheet to be used between product layers on a pallet load, Fig. 3.4. With this design, multiple triboelectric energy harvester tier sheets could be used on a single pallet of product. This configuration within the package system allows for the energy harvester to be located in a position that experiences a significant amount of mechanical energy in the form of pallet and product shock and vibration. Regardless of the transportation mode, there is a significant amount of vibration and mechanical energy input [85], but this study focuses specifically on the over-the-road truck vibration that is commonly experienced by packaged products in transit.

In this study, the tier sheet energy harvester design is paired with an energy harvesting battery charger, used to charge a rechargeable coin cell battery. The system described in this study is as follows: a triboelectric energy harvester built into a tier sheet
structure that generates and captures electrical energy from mechanical input, a battery charger that takes this harvested energy and uses it to charge a battery, and a rechargeable coin cell battery that is sufficient in voltage and capacity to power a number of small electronic devices for a myriad of applications.

3.4 Power Requirements for Applications

The battery charging system used in the harvester system is sufficient to charge a number of battery types, with voltages up to 5V. Almost all small, portable electronic devices use batteries in this range [31]. The battery used in this research is a 3.6 V, 40-mAh lithium-ion rechargeable coin-cell battery sourced from Dantona Industries, Inc. (Wantagh, NY). It is capable of providing the necessary power for the applications of this study for varying durations of time. Three specific applications chosen for this study are a HOBO UX-100 temperature / humidity data logger, a Copernicus II - 12 channel GPS module, and a Lansmont 3X90 field data recorder, listed in order of increasing power requirements.

The HOBO UX-100 Temperature / Humidity Data Logger is a small data logger that continuously records temperature and humidity, and is able to do so for long periods of time on a single battery charge. This device is typically used in warehouses, but could be used in a number of environments. It may be used in the context of packaging to continuously monitor the package’s environment throughout its distribution cycle. It has relatively low power requirements, only using a small 3V coin cell battery and drawing 1–3 mA continuously [101].
The Copernicus II - 12 channel GPS module is a simple GPS unit that requires very little power and may be used in a variety of applications. The Copernicus II requires 44 mA at 3.3V to be fully powered, which may be supplied by the battery charged in this study. The sampling frequency can be modified with these units so that the unit may survive for days before the battery must be recharged. In distribution, expensive medical, pharmaceutical, and electronic device packages are frequently tracked through the distribution environment, which is a need that may be easily met by imbedding this unit into the pallet or package system [102].

A Lansmont 3X90 Field Data Recorder is a field data recorder that has an internal triaxial accelerometer, temperature sensor, and humidity sensor. It is capable, when fully powered, to run for ninety days. The function of this unit requires a power supply of 9V, at approximately 1.1 mA continuous current. This unit is widely used in the distribution industry for characterization and monitoring of the distribution environment [103].

Table 3.1: Power Requirements for Devices

<table>
<thead>
<tr>
<th>Device</th>
<th>Battery Nominal Voltage (V)</th>
<th>Device Average Power Requirement (mA)</th>
<th>Continuous / Intermittent</th>
</tr>
</thead>
<tbody>
<tr>
<td>HOBO UX-100 Temperature / Humidity Data Logger</td>
<td>3.0</td>
<td>2.0</td>
<td>Continuous</td>
</tr>
<tr>
<td>Copernicus II GPS module</td>
<td>3.3</td>
<td>44.0</td>
<td>Continuous</td>
</tr>
<tr>
<td>Lansmont SAVER 3X90 Field Data Recorder</td>
<td>9.0</td>
<td>≈1.1</td>
<td>Continuous</td>
</tr>
</tbody>
</table>

The power requirements and battery specifications of each of the three applications are summarized for comparison in Tab. 3.1. The battery charged in this
research is capable of powering all three of these applications, based on their mA requirements. However, the third unit, the Lansmont Saver 3X90, uses a 9 V battery as its power supply. The battery charging system used in this study is only capable of charging up to a 5 V battery. With this in mind, the power requirements of the Lansmont Saver 3X90, though not currently supported by the batteries charged in this research, could be attained by charging a different battery of a sufficient voltage, using the same principles identified in this study.

3.5 Validation

A number of methods developed for the purpose of characterizing the performance of the triboelectric tier sheet and for measuring its ability to generate usable electrical energy are described in the following section. These include the use of an industry-accepted vibration simulation test method to provide the necessary physical excitation for the harvester to function, and a process for charging rechargeable coin cell batteries used to quantify harvester performance. With all of these key processes and test methods in place, it is possible to answer the question that drives this research: can triboelectric energy harvesters used in a packaging application generate enough to power field data recorders during vehicle transport?

The battery-charging device described above is the LTC 3331- Nanopower Buck-Boost DC/DC with Energy Harvesting Battery Charger, manufactured by Linear Technology (Milpitas, CA) [104]. The LTC 3331 has an input voltage range of 3.0–19.0 V, and has a very low minimum current requirement, making it a perfect choice for energy harvesting applications such as triboelectric energy harvesting, a process that is
known to produce high voltages with low amperage. Low-current producing energy harvesting methods are typically not used for the charging of batteries, as battery chargers must supply a constant current level to the battery in order to charge it. This battery charger was designed to combat this limitation, and is able to step-up the voltage and current provided to it in order to meet the battery’s charge requirements. It also uses a full-wave rectifier that allows for the entire energy pulse to be used, a feature that is very useful with triboelectric harvesting, which frequently generates nearly equal positively and negatively charged events.

This battery-charging device enables the achievement of a number of goals in this study. First, it charges the battery for use with the temperature and GPS loggers, Tab.3.1. Next, it is used for the conversion of low-current, high voltage electrical energy using a system of capacitors that allow the device to convert the input voltage to a specific required output voltage, an essential step in the use of this generated electricity. Lastly, by providing a consistent system of charge and measurement, the battery charger provides the ability to compare multiple experiments and thereby determine ideal experimental conditions for the optimization of charge development by the energy harvesting system.

The battery charger is able to charge the battery by providing two things: a constant supply of user-defined voltage that is above the current charge of the battery, and a consistent supply of current. The required voltage for charging the battery must exceed the voltage of the battery, as the voltage gradient must be higher on the battery charger side than the battery side in order for the charge to flow to the battery.
Though many types of mechanical input are capable of providing the physical excitation needed for this triboelectric energy harvester to function, this study focuses on an input that most resembles the physical forces experienced by packages in transit: random, broad-spectrum, over-the-road truck vibration.

It is common practice in industry to use vibration simulation equipment and test methods to simulate real-world vibration inputs in a laboratory setting. The International Safe Transit Association (ISTA) & ASTM International (ASTM) both publish industry-accepted vibration test standards [97, 96]. This research focuses on ISTA Steel Spring random vibration profile, Fig. 3.5, prescribed in ISTA test standards 3B, 3E, 3F, and 3H [97]. In this research, the testing is performed for four hours per test on a servo-hydraulic vibration system (Lansmont Corporation).

![Figure 3.5: ISTA Steel Spring vibration profile](image)

To simulate the packaged product distribution environment, a single stack of corrugate boxes containing automotive electrical assemblies is used. For all testing
performed in this study, the triboelectric energy harvesting tier sheet is located between the fifth and sixth (top) layer of this package assembly, Fig. 3.6. It is generally accepted that in the upper layers of a unit load (or single stack of boxes, in this case), the forces affecting the packages are amplified by the package system itself. This causes the mechanical potential energy available to the energy harvester when placed near the top of the unit load to be greater than when it is placed near the pallet [85].

Figure 3.6: Single Stack boxes with triboelectric tier sheet with layer details

With the experimental setup described in this section, including the triboelectric tier sheet, the LTC 3331 battery charger, and the ISTA Steel Spring Random Vibration Spectrum, the triboelectric energy harvester and battery charging system are validated. This is accomplished by three replicate tests of ISTA Steel Spring random vibration profile, for a duration of four hours each. A four second window of the voltage response of the harvester to the steel spring vibration input is illustrated in Fig. 3.7. The coin cell battery charging results from the three tests are reported in Tab. 3.2.
For all testing performed in this research, the battery charging unit is set with a UVLO (under voltage lockout) window of 5V-18V, meaning that any charge generated with a voltage level outside of this range could not be used by the charger, and would not be routed to the battery. For this reason, the charge generated in this research is described in terms of voltage, only. Though not quantified herein, the ability of this system to charge batteries demonstrates that sufficient levels of amperage (A) and power (W) are generated, in addition the required voltages that are recorded.

![Graph](image)

Figure 3.7: Vibration response of triboelectric harvester to ISTA Steel Spring profile

<table>
<thead>
<tr>
<th>Battery Nominal Voltage (V)</th>
<th>Battery Charge Initial (V)</th>
<th>Battery Charge Final (V)</th>
<th>Battery Charge (V)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.6</td>
<td>3.05</td>
<td>3.42</td>
<td>0.37</td>
</tr>
<tr>
<td>3.6</td>
<td>3.00</td>
<td>3.25</td>
<td>0.25</td>
</tr>
<tr>
<td>3.6</td>
<td>3.02</td>
<td>3.41</td>
<td>0.39</td>
</tr>
<tr>
<td><strong>Mean</strong></td>
<td><strong>3.02</strong></td>
<td><strong>3.36</strong></td>
<td><strong>0.34</strong></td>
</tr>
</tbody>
</table>
3.6 Conclusions

In this research, a triboelectric energy harvester is designed and constructed to generate electrical energy from a vibration common to the package distribution environment. This harvester is designed specifically to mimic a tier sheet, a common component in many unit load systems. ISTA Steel Spring random vibration profile is used to simulate truck vibration, and a unit load is simulated using a vertical column stack of corrugate boxes containing automotive electrical components in thermoformed trays. A battery charging system is used to provide a consistent method of battery charging and measurement, providing a reliable comparison of all vibration experiments to one another. Using this same battery charging system, the generated electricity is used to charge a battery, which is then applied to one of three chosen applications for this study. The following conclusions are made:

- It is possible to use triboelectric energy harvesting to charge a battery using the system described in this manuscript. Three 3.6 V (nominal charge) lithium-ion coin cell batteries were charged from a discharged state at 3.02V to an average charge level of 3.36V.
- By harvesting mechanical energy natural from the packaged product distribution environment, it is possible to generate sufficient levels of electricity to fully power many types of field data recorders, including a HOBO UX-100 Temperature/Humidity Logger and a Copernicus II GPS Logger Module.
Chapter 4

Exploration of the durability and relative humidity sensitivity of triboelectric energy harvesters in the distribution environment

4.1 Introduction

It is known that the charge generation of triboelectric contact electrification can be affected by certain environmental and mechanical factors, such as frequency of mechanical excitation and relative humidity of the environment. Another factor discovered in this research, and described herein, is the condition, or physical durability, of materials used in harvester construction, potentially a limitation due to physical durability constraints. Physical durability of the materials used to build the harvester is a concern: the surfaces of the materials may be affected by dust and other particles during vibration, the material surface could be affected by abrasion, or the cushion materials could degrade over time. These concerns stem from the nature of triboelectric harvesting, as physical contact is essential to the process. Initial testing with the triboelectric tier sheet design showed a potential weakness of the aluminum-coated PE material. Further work done to test these concerns is described in this research.

A review of literature on contact electrification and triboelectric charge generation revealed that humidity may affect charge generation. This is due to the nature of contact electrification, as it involves physical contact and a transfer of mobile electrical charge from one material, through the environment between materials (usually air), and to the
surface of the second material. The dependence of triboelectric energy harvesting on relative humidity of the immediate environment of the harvester has been demonstrated in a number of studies [62, 63, 66]. The optimal humidity for charge generation, however, is still disputed [66, 87, 88]. The extent to which humidity affects this process, and the optimal humidity for the triboelectric tier sheet design are both explored in this research. The effect of humidity and physical durability of materials used in triboelectric energy harvesting, with the specific application of the triboelectric tier sheet, are described in the following sections.

4.2 Durability Testing

The triboelectric tier sheet is a five-layer triboelectric energy harvester that uses the vertical contact separation mode of harvester excitation, and vibration as the mechanical energy source. This energy source and contact mode cause the harvester to have some potential physical weaknesses. These potential weaknesses are damage due to friction and the entry of foreign particles that can cause unnatural wear on the layers. Despite the use of vertical contact-separation mode, there is still some horizontal motion, or friction, of the layers with one another. This friction between the Teflon® and the aluminum-coated PE can cause removal of the aluminum coating, as seen in initial testing. In addition, the current design of the harvester does not encapsulate the structure, or prevent outside materials from entering the structure of the harvester between the layers. In initial testing, foreign particles, such as dust, dirt, or sand, entered between the layers during vibration and removed the aluminum coating from the PE film, significantly decreasing performance. In addition, this energy harvester is designed for
use in the packaged-product distribution environment, in which it could experience vibration forces for long periods of time. The ability of the harvester to generate charge for the entire duration of this extended vibration is key to the concept of the triboelectric tier sheet. To determine the effect of both of these physical concerns, and to test the ability of the energy harvester to generate charge, using forces common to the distribution environment, further durability testing is performed.

*Durability testing details*

Random vibration profiles used for package and distribution testing are typically time-compressed profiles, meaning that the data that was recorded and used to construct these profiles undergoes a transformation in which the intensity of the vibration is increased, while the time domain is decreased. In this process, the shape of the PSD profile is not changed, but the intensity is increased. This appears as the PSD level on the profile ‘moving up’ on the profile, Fig. 4a, b. This allows for testing to be done in an accelerated time frame, while maintaining the integrity of the vibration simulation test [94]. A comparison of the ISTA Steel Spring random vibration profile and the random vibration profile used for this 12-hour testing is provided in Fig. 4a, b.

The vibration test used to determine the physical durability of the triboelectric tier sheet energy harvester is a 12-hour random vibration profile that is not time compressed. The purpose of using un-compressed vibration data is to simulate actual road conditions, over a longer period of time than most vibration tests prescribed in testing standards. This is done to determine the ability of the energy harvester to withstand actual road conditions, or more simply, the physical durability of the energy harvester.
Though foreign particles such as dirt and sand caused damage in initial testing, no damage was seen in later validation due to this cause. Though it is likely that the dust and foreign particles were still present between the layers during this testing, due to the nature of corrugate board, no damage to the triboelectric material layers was seen, though it was clearly an issue in initial testing. This can be explained by a design change to the structure of the triboelectric tier sheet after initial testing, which significantly reduced the horizontal motion of the layers, leaving almost all contact and force to be experienced in the vertical axis.

Two other sources of damage were identified as a result of this testing, heat-induced degradation of the aluminum-coated PE and the permanent deformation of cushioning materials, Fig 4.2. After the 12-hour durability test, it is clear that a significant amount of heat develops between the layers of the triboelectric energy harvester as a result of contact electrification. This takes place where the most contact/friction likely
occurs, which is localized where the cushion circles meet the material opposite them in the structure. Once a significant amount of friction induced-heat has developed, the surface of the aluminum-coated PE material becomes significantly more susceptible to damage, Fig. 4.2.

![Damage on aluminum coated PE surface caused by friction and heat](image)

Figure 4.2: Damage on aluminum coated PE surface caused by friction and heat

A second type of damage found in this testing is the permanent deformation of the cushioning materials designed to separate the layers from one another, Fig. 4.3a,b. Over time, as these cushions are repeatedly compressed and released from compression, they begin to degrade, losing their cushioning ability. After the 12-hour vibration test was performed, all of these cushions were deformed, Fig 4.3b, losing approximately half of their initial thickness. This raises concern, as the ability of the layers of the energy
harvester to repeatedly contact and separate from one another is essential to its function. Without this separation, the energy harvester’s charge generation significantly decreases.

Figure 4.3a (Left): Cushion before 12-hour durability test; Figure 4.3b (Right): Permanent deformation of cushion after 12-hour durability test

4.3 Humidity Testing

Multiple studies have shown that there is a humidity range at which charge generation or transfer due to contact electrification is at its optimal level. These studies do not generally agree on this optimal range. One study shows that a relative humidity of 0% RH is best for contact electrification [88]. A second study shows that higher humidities are best for contact electrification [87]. A third study shows that there is an intermediate humidity range at which contact electrification is optimal, between 20% and 40% RH, and that charge generation/transfer decreases as humidity rises above this range [66]. It is known that packages travelling through distribution can be exposed to a wide humidity range [89]. For this reason, the triboelectric tier sheet energy harvester is tested at three humidity levels: 15% RH, 35% RH, and 65-70% RH.
Humidity testing details

The tests used to determine the ability of the triboelectric energy harvester to generate charge at various humidity levels are a series of 4-hour vibration tests using ISTA Steel Spring Random Vibration Spectrum, each test at a specific relative humidity. It is important to note that this test uses the actual ISTA random vibration profile, meaning that this is time-compressed vibration data, unlike the vibration profile used for durability testing, which was non time-compressed. Five total tests were performed, one at a low humidity level, three at the proposed optimal humidity level [66], and one at a high humidity level. Table 4.1 summarizes the tests and conditions of each test performed for humidity testing.

Table 4.1: Summary of prescribed humidity tests

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Relative Humidity (% RH)</th>
<th>Test Duration (Hr.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>15</td>
<td>4</td>
</tr>
<tr>
<td>2</td>
<td>35</td>
<td>4</td>
</tr>
<tr>
<td>3</td>
<td>35</td>
<td>4</td>
</tr>
<tr>
<td>4</td>
<td>35</td>
<td>4</td>
</tr>
<tr>
<td>5</td>
<td>65-70</td>
<td>4</td>
</tr>
</tbody>
</table>

The relative success of each test is evaluated in terms of relative battery charge. The system used to charge these batteries is the LTC 3331 (Linear Technologies), described in the previous chapter, or in full detail in Appendix A. For each test, a 3.6 V (nominal voltage) coin cell battery is discharged to 3.0 V. In the process of the vibration and charge generation, the battery is charged, and the extent to which it is charged
demonstrates the efficacy of the triboelectric energy harvester at each set of test conditions. Table 4.2 summarizes the results of these five tests.

Table 4.2: Summary of humidity test results

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Humidity (% RH)</th>
<th>Battery Charge Initial (V)</th>
<th>Battery Charge Final (V)</th>
<th>Battery Charge (ΔV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>15</td>
<td>3.00</td>
<td>3.40</td>
<td>0.40</td>
</tr>
<tr>
<td>2</td>
<td>35</td>
<td>3.05</td>
<td>3.42</td>
<td>0.37</td>
</tr>
<tr>
<td>3</td>
<td>35</td>
<td>3.00</td>
<td>3.25</td>
<td>0.25</td>
</tr>
<tr>
<td>4</td>
<td>35</td>
<td>3.02</td>
<td>3.41</td>
<td>0.39</td>
</tr>
<tr>
<td>5</td>
<td>65 - 70</td>
<td>3.00</td>
<td>3.00</td>
<td>0.00*</td>
</tr>
</tbody>
</table>

The data shown in Table 4.2 show that there is not a clear difference between the levels of battery charge between the lower humidity level, 15% RH, and the middle humidity level, 35% RH. There is a clear difference between the charge levels of both of these humidity levels the battery charge of the high humidity, 65-70% RH. In fact, the testing done at 65-70% RH did not yield any battery charge. It is important to note that in test 5, at 65-70% RH, there was charge generated. The nature of the battery charging process required that the voltage exceed 5.0 V in order for the battery charger to process this power and route it toward charging the battery. The charge generation simply did not exceed this 5.0 V limit, and therefore the battery was not charged. A more thorough description of the battery charging process is detailed in Appendix A. Though differences may be observed in the data shown in Table 4.2, additional testing should be performed using more replicates at each humidity level.
Chapter 5

Conclusions

A triboelectric energy harvester is designed and constructed to generate electrical energy from a vibration common to the package distribution environment. This harvester is designed specifically to mimic a tier sheet, a common component in many unit load systems. ISTA Steel Spring random vibration profile is used to simulate truck vibration, and a unit load is simulated using a vertical column stack of corrugate boxes containing automotive electrical components in thermoformed trays. A battery charging system is used to provide a consistent method of battery charging and measurement, providing a reliable comparison of all vibration experiments to one another. Using this same battery charging system, the generated electricity is used to charge a battery, which could then be applied to one of three chosen applications for this study. The physical durability and relative humidity sensitivity of triboelectric energy harvesters are also explored. Triboelectric energy harvesters used in a packaging application have been proven to generate sufficient electricity to power field data recorders during vehicle transport. This concept is the focus of this manuscript, and has been proven herein. The following conclusions are made:

• It is possible to use triboelectric energy harvesting to charge a battery using the system described herein, a significant feat due to the low-current nature of the individual charge-generating events that are inherent to a contact electrification-based energy harvesting method. Three 3.6 V
(nominal charge) lithium-ion coin cell batteries were charged from a discharged state at 3.02V to an average charge level of 3.36V.

- By harvesting mechanical energy that naturally occurs from the packaged product distribution environment, it is possible to generate sufficient levels of electricity to power many types of field data recorders, including a HOBO UX-100 Temperature/Humidity Logger and a Copernicus II GPS Logger Module.

- Physical durability testing shows that dust and foreign particles do not pose a significant threat to the continued performance of the triboelectric energy harvester.

- Multiple minor material damage sources were identified in durability testing, including heat-abrasion and cushion deformation. Further testing, or an alternative cushion design may be required in later designs of this harvester.

- Testing at both 15% and 35% RH produces similar levels of battery charge. This is contrary to the expected result, which is that low humidity severely limits triboelectric charge generation. Further testing at these humidity levels is needed to verify these conclusions, as a small sample set is tested in this study.

- Humidity testing shows that high humidity (65-70% RH) does limit contact electrification / charge generation of the triboelectric energy harvester used in this study.
Appendices
All battery charging performed in this study is done by the use of the LTC 3331, Nanopower Buck-Boost DC/DC with Energy Harvesting Battery Charger, manufactured by Linear Technologies, Fig. A-1. The LTC 3331 has many features that are essential to the charging of batteries with the type of power that typically comes from energy harvesting sources. A few of these include a duel-input full-wave rectifier, a high-voltage buck DC/DC converter, an input protective shunt, and an ultra-low quiescent current.
The duel-input full wave rectifier allows for two power sources to be used simultaneously. It functions to process an AC supply or DC supply that alternates between positive and negative voltage, and is used by the input pins AC1 and AC2, indicated by point A, Fig. A-1. This unit also has a Vin pin, indicated by point B in Figure A-1, that allows for the bypass of these two rectifier inputs if the supply does not require this function.

The high voltage buck DC/DC converter provides a window for input voltage, providing the ability to step down high voltage input to a usable, programmable output voltage level for either charging the internal battery of the unit or for powering and external unit. This function only operates while input supply is present, which reduces the overall power requirement for the operation of the LTC 3331.

The input protective shunt simply protects the LTC 3331 unit from high voltage or amperage spikes, which could potentially be harmful to the unit. If these spikes are supplied by the input, the protective shunt essentially temporarily shuts down the function of the unit until input levels are ‘safe’.

The determination and use of the correct under voltage lockout (UVLO) settings are essential to the effective use of this battery charging unit. The UVLO settings determine the window of accepted voltage by the battery-charging unit. It is important to characterize the behavior of the energy harvester, as the typical current and voltage levels that it supplies are important to know in order to match the settings and inputs for optimum energy harvesting and storage. For all testing using triboelectric energy
harvesters in this study, a UVLO window of 5V-18V is used. UVLO rising and falling parameters can be set using the pins indicated by point, Fig. A-1.

The output voltage pins, seen in point D in Figure A-1, are where the output voltage level of the battery charger is determined. For all testing using the triboelectric tier sheet energy harvester, an output voltage of 4.5V was selected. This level simply needs to be greater than, or equal to, the charging voltage of the battery to be charged. In this case, the battery’s charging voltage was 4.2V. As energy harvesting continues, the voltage accumulates on the Vout pin, point F in Figure A-1, until the desired output voltage level is reached on that pin. When this occurs, the power is then redirected to charge the battery.

Also essential for optimal battery charging are the float voltage settings and LBSEL setting, point E in Figure A-1. Float voltage refers to the voltage that is directed to the battery, once the set Vout has been reached on the Vout pin. The set float voltage should be the charging voltage of the battery used, found in the battery’s spec sheet. For this testing, a float voltage of 4.2 V is used, as this is the battery’s designated charging voltage.

The last setting that is key to the effective use of this unit is the OFF/ CHARGE/ FAST CHARGE pin, point G in Figure A-1. For this testing, the charge pin is used in every case, and for every test.

* For a full description of this unit, as well as a collection of other specialized units, visit http://www.linear.com
Appendix B

Additional Data

Figure B-1: Frequency response of triboelectric harvester to ISTA Steel Spring profile

Figure B-2: Shock response of triboelectric harvester to 18-inch drop of 18lb.box
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