

Sensing and Actuation in Electroactive Elastomeric Bodies

by

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“When do you think you can submit your thesis?”

T. Giffney, April 2024

“Today.”

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Abstract

College of Engineering
Mechanical Engineering

Doctor of Philosophy

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Some of the world's most advanced technology is rigid due to various factors such as; manufacturability, miniaturisability, physical linearity, and more ideal physics in general. In parallel industries is also looking to use automation to improve and replace laborious tasks whether they be domestic, commercial, or industrially related tasks. There is a growing need for new innovations in technology to utilise the soft robotic solutions that mimic biological solutions seen in nature. This thesis is part of many to improve an understanding of the electroactive polymer subset of soft robotics and the limitations of specific implementations of artificial skin and artificial muscle technologies.

This thesis explores the integration of Electrical Impedance Tomography (EIT) with advanced soft sensing technologies, focusing on carbon black silicone rubber (CBSR) elastomer composites and Dielectric Elastomer Actuators (DEAs) to enhance pressure mapping, strain sensing, and actuation.

CBSR elastomer composites, noted for their high stretchability and biocompatibility, were investigated to understand their resistance relaxation behavior. This research contributes to optimizing the design of flexible dynamic strain sensors by modeling the response of resistance to transient strain inputs. The study developed an EIT-based pressure mapping system using a silicone CB nanoparticle sensing domain that mimics pressure mapping qualities human skin. This system was evaluated for its spatial and temporal resolution, showing potential for creating artificial pressure-sensitive skin with practical applications. Furthermore, the integration of EIT with DEAs was examined to improve the mapping of compressive forces across electrode surfaces. Despite some trade-offs in accuracy due to electrode compliance, this approach offers promising advancements for applications requiring precise actuation and pressure mapping. This work has majorly contributed towards filing a patent for an DEA-EIT actuator-sensor device. Additionally, the research uncovered unintentional power generation in DEAs, which could function as Dielectric Elastomer Generators (DEGs) due to mechanical strain. This finding highlights the dual functionality of DEAs and suggests opportunities for energy harvesting applications. Finally, a portable, low-cost EIT-based hardware system for pressure mapping was introduced. This system enables comprehensive characterization of various sensing domains and supports advancements in EIT-based soft sensor technology, with implications for biomedical devices, robotics, and energy harvesting.

Overall, this research advances the field of soft sensors by integrating EIT with innovative materials and technologies, providing new insights and applications in dynamic sensing and actuation.

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Abbreviations

| | |
|-------------|---|
| ADC | Analog-to-Digital Converter |
| CAD | Computer Aided Design |
| CB | Carbon Black |
| CFA | Cartesian Force Applicator |
| CE | Compliant Electrode |
| CoM | Center of Mass |
| DE | Dielectric Elastomer |
| DEA | Dielectric Elastomer Actuator |
| DEG | Dielectric Elastomer Generator |
| DUT | Domain Under Test |
| EIT | Electrical Impedance Tomography |
| ERT | Electrical Resistance Tomography |
| FEA | Finite Element Analysis |
| FEM | Finite Element Modelling |
| FPC | Flexible Printed Circuit |
| IDF | IoT Development Framework |
| MUX | Multiplexer |
| PCB | Printed Circuit Board |
| PCBA | Printed Circuit Board Assembly |
| PDMS | Polydimethylsiloxane (AKA silicone) |
| PNEC | Piezoresistive Nanoparticle Elastomer Composite |
| SMU | Source Measure Unit |
| SMD | Surface-Mount Device |
| SR | Silicone Rubber |
| THT | Through-Hole Technology |
| EAP | Electro-Active Polymer |

Symbols

| | | |
|-------------------|-----------------------------|---------------------|
| A | Area | [m ²] |
| C | Capacitance | [F] |
| ϵ_0 | Vacuum Permittivity | [Fm ⁻¹] |
| ϵ_r | Relative Permittivity | [a.u.] |
| K | Bulk Modulus | [Pa] |
| ν | Poisson's Ratio | [a.u.] |
| Q | Electrical Charge | [C] |
| U_E | Electrical Potential Energy | [J] |
| U_ϵ | Elastic Potential Energy | [J] |
| R | Resistance | [\Omega] |
| σ | Stress | [Pa] |
| S or ϵ | Strain | [a.u. or %] |
| $\dot{\epsilon}$ | Strain Rate | [%s ⁻¹] |
| V | Voltage | [V] |
| Y or E | Young's Modulus | [Pa] |
| z | Thickness | [m] |

Dedicated to tinned baked beans in all their glory... .

Chapter 1

Introduction and Motivation

Rigid robotic systems often have multiple rotary motors and various sensors integrated together for precise control of the robot, this is mirrored in biology with the animals having many actuator units in the form of muscles and a multitude of various receptors for sensing their environment. The rigidity of rotational motors is stifling creativity in the creation and development of devices amongst many other unforeseen future technology. Engineers are often constrained to solving problems and designing solutions using typical rigid sensors and actuators due to their current ubiquity and their evolved increased efficiency. With the rise of research into soft sensor and actuator devices, these such device need to follow suit of the traditional rigid sensors and actuators and become ubiquitous and viable option for general and specialised engineering design solutions.

This thesis has developed methods and tools for creating and characterising artificial pressure sensitive skin technology. The thesis then continues to explore the integration of this artificial skin technology into an artificial muscle technology. The work in this thesis has ultimately contributed towards a patent for DEA-EIT actuator-sensor technology in a quest to bring this work out of the academic realms into real-world applications.

1.1 Why Go Soft and Not Rigid?

The requirement for soft robotics in general has been driven by the limitations of current rigid robotic solutions to interact with natural organic material. Manipulation of natural organic objects such as animals, plants, fruit, vegetables, and meat have traditionally been handled by humans by hand due to our ability to use our dexterity and intelligent control systems to ensure minimal undesirable damage. With the advance in technology in various soft robotic actuators[16–20], sensors[21], and soft robotics control[22, 23]. The use of soft robotics in place of rigid alternatives, amongst other benefits, has the opportunity to be more sustainable by decreasing waste products during fabrication, using biodegradable or recyclable materials, shelf life, and use of renewable resources[18]. The use of soft robotics brings opportunity of creating devices with a reduced bill of materials size and less moving parts for maintenance. The use of soft robotics in biomedical and aerospace applications is especially desirable due to the difficulties experienced when designing with regular motors in the outer space and near sensitive biological tissue environments such as heat dissipation, lubrication, and mass[24–27].

The most common rigid actuator is the rotary electric motor and the global market was valued at USD 142.2 billion in 2020, with a predicted growth rate of 9.5% until 2032[?]. Although this

market is dominated by automobiles which currently require the traditional form of rotary electric motors, growing sectors of this market such as medical, factory automation, and aerospace have potential interest in adopting soft actuator alternatives for the reasons given above. In parallel, rigid strain sensors of types metallic foil and semiconductor, was given a global market value of USD 190.66 million in 2022 with a compound annual growth rate of 3.9% until 2029[?]. Adjacently the pressure mapping global market value, focused mainly on the health sector, was valued at USD 480 million in 2023 with an expected growth rate of 5.1%[? ?]. Many soft actuator technologies could be used in these growing medical, aerospace, factory automation, and agricultural sectors.

Soft robotic actuation can be achieved through various mechanisms including thermal, electrochemical, fluidic, magnetic, and electrostatic. Similarly soft stress-strain sensing can be achieved through various physical principles such as resistive, capacitive, magnetic, and optical sensing methods. Often the function of soft actuators can be inverted such that the deformation of the actuator can produce a signal used for self sensing, in electroactive polymer (EAP) technologies such as dielectric elastomer actuators (DEAs)[28–31] and ionic polymer-metal composites (IPMCs)[32]. EAPs have the benefit of electronic control over other soft actuator and sensor technologies controlled by fluids, heat, or light which contain the complexity of another energy transfer process.

Proprioception in artificial muscle technology has been made a reality. This is seen in the self-sensing of one dimensional strain of DEAs usually through capacitive measurement between the compliant electrodes during operations to obtain the magnitude of a contraction. However, the pressure mapping done similar to the mechanosensation performed by cutaneous mechanoreceptors on an artificial muscle device has not been explored as of writing this thesis.

Publications towards this thesis include three conference papers, one journal paper, and one provisional patent filed. This thesis has converged on the use of conductive particle based elastomer composites and their use in sensors and actuators, in particular an electrical impedance tomography (EIT) based artificial skin and it's integration into the artificial muscle technology, dielectric elastomer actuators. The composite type used throughout the thesis is simple to fabricate but not well understood in terms of its electromechanical transient and dynamic characteristics. The modelling of such conductive particle composites would elucidate the feasibility of inverting the model to create a responsive strain sensor. This composite has been characterised in one-dimension several times in literature already however, if a two dimensional sensing application of this composite is desired the characterisation of the sensor in two dimensions must be completed. A method to do such 2D sensing is using EIT. EIT has been used in the past for a huge range of applications, with few exploring the use of EIT as a pressure mapping sensor. Although EIT-based pressure mapping was first discovered 30 years ago, the technology is still in its infancy with several problems needing to be resolved before the technology can be used reliably in real-world applications.

1.2 Research Objectives

The research objectives and questions for this thesis are given below:

1. Quantify and analyse static, dynamic, and transient phenomena seen in conductive particle composites.
2. From the characterisation in objective 1 mitigate the effects of the transient phenomena.

3. Create a set of metrics for quantifying the performance of an electrical impedance tomography based artificial skin.
4. Simulate and integrate an electrical impedance tomography based artificial skin onto a dielectric elastomer actuator.
5. Investigate the energy harvesting of a device that is both a dielectric elastomer actuator and electrical impedance tomography device.

1.3 Chapter Contributions

Chapters 3 - ?? contain the core novel research contributions. Chapters 2 and ?? provide essential background knowledge and future research directions for the thesis respectively.

Chapter 2 - Literature Review: This chapter explores the nature of biological skin and muscle from an engineering perspective, quantifying necessary functions and properties desired to replicate or supersede for their artificial equivalents. The thesis then describes state-of-the-art soft sensors and actuators and their function.

Chapter 3 - A Simple Conductive Elastomer Composite with Complex Behaviour: This chapter uncovers the electromechanical tensile and compressive properties of carbon black silicone composites, in order to understand the material before it's use in sensors and actuators.

Chapter 4 - An Improved an Electrical Impedance Tomography Based Artificial Soft Skin Pressure Sensor: This chapter discusses the use of electrical impedance tomography to create a pressure mapping sensor and provides tools for analysing the suitability to various applications and choosing a suitable sensing domain.

Chapter 5 - Giving Artificial Muscles the Sense of Touch: This chapter describes the integration of the pressure mapping technology discussed in the previous chapter, how it can be integrated into dielectric elastomer actuators, and the trade-offs.

Chapter 6 - Unintentional Power Generation in a DEA-EIT Sensor-Actuator Device: This chapter discussed the unintended power generation of the simultaneous sensor actuator device discussed in the previous chapter.

Chapter 7 - A Portable Electrical Impedance Tomography Based Pressure Mapping Sensor and Validation System: This chapter discusses the small form factor, low-cost hardware design for a hybrid artificial muscle - artificial skin based device.

Chapter 8 - ?? Modelling of DEA-EIT Capacitively driven Hybird Sensing and Actuation Device: The is chapter models the a DE-EIT device in order to find an optimal range of parameters at which capacitive shunting can be used to improve the DE-EIT pressure mapping device responsiveness.

Chapter 9 - The Biomimetic Re-Evolution: This chapter discusses the future direction of the technology discussed in the thesis and acknowledges the future of the broad field of soft robotics.

Chapter 2

Literature Review

Humans can complete complex tasks due to their intelligence, dexterity, and physical make up. These complex tasks include agricultural picking, culinary preparation, factory goods processing, and biomedical practice. To complete these tasks with machines it is important to quantify these human qualities that the technology must match or supersede. The first part of this section is focused on understanding and quantifying human skin and muscle tissue often required for these complex human tasks. In parallel, artificial skin and artificial muscle state-of-the-art technology is reviewed. Finally, background theory on piezoresistive elastomer composites which will be utilised with specific sensor and actuator technology is given to setup foundational knowledge base and reference for the rest of the thesis.

2.1 Biological Skin form and function

Skin is the largest organ in the human body with many functions, however this thesis only aims to replicate some pressure-sensitive functions of skin. Two pressure-sensitive categories of skin and muscle tissue transducers which allow for dexterous manipulation of objects are:

1. Proprioceptors: respond to internal mechanical stimuli in a joint capsule, tendon, or muscle to give the sense of motion.
2. Cutaneous mechanoreceptors: respond to mechanical stimuli usually external to the body, including pressure and vibration, for the localisation of sensations.

Locations of both proprioceptors and cutaneous mechanoreceptors are shown diagrammatically in Figure 2.1. Proprioceptors aid in determining pose estimates of body parts in space, acting as sensors providing feedback closed-loop control for the neurological motion control of body parts. Whereas cutaneous mechanoreceptors have various roles including object recognition, manipulation control, as well as motion control.

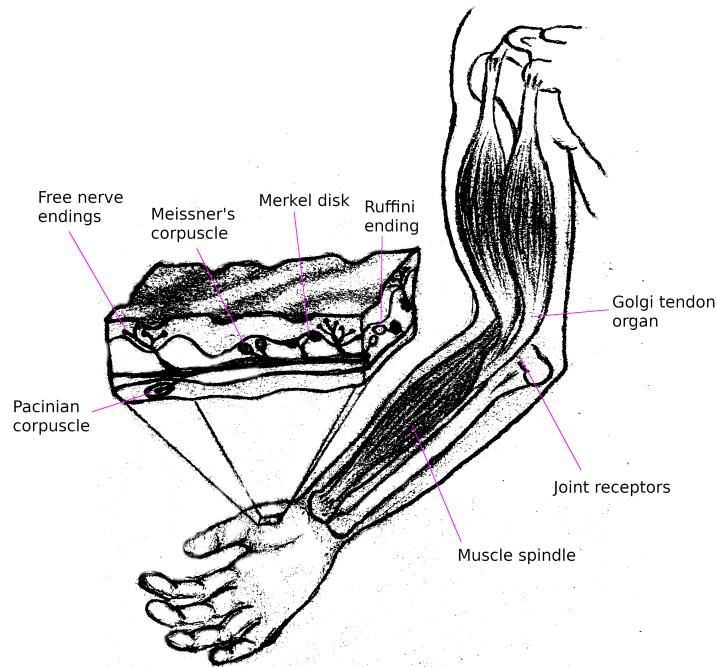


FIGURE 2.1: Examples of the locations of proprioceptors and cutaneous mechanoreceptors in the human body.

The function of both kinds of receptor have been mimicked by certain device technologies. For example proprioceptors have been mimicked in wearables and human assistive devices where joint motion has been estimated by sensors such as rotary/linear encoders, inertial measurement units (IMUs), and stretch sensors fixed adjacent to joints to calculate pose estimates of limbs[1–4]. Examples of such devices are displayed in Figure 2.2

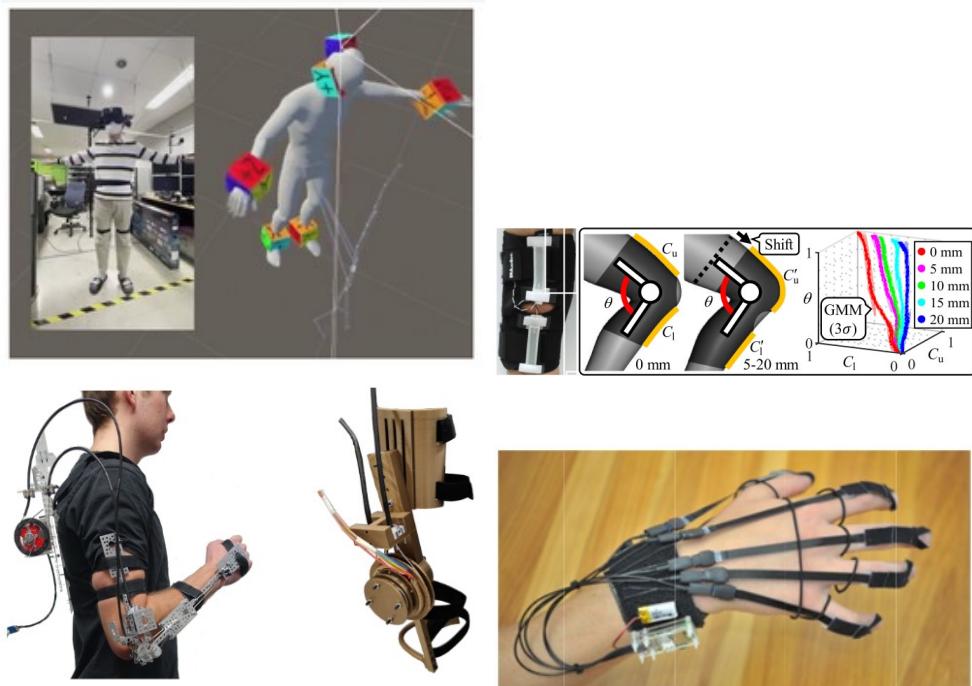


FIGURE 2.2: Clockwise from top left: IMU pose estimation[1] (© 2022 MDPI), stretch sensor knee joint pose estimation[2] (© 2020 IEEE), encoder elbow pose joint estimation[3], stretch sensor hand joint estimations[4].

Cutaneous mechanoreceptors have been mimicked by the development of pressure mapping of flexible surfaces. Examples of such technologies include, foot pressure based gait analysis, wheelchair seat pressure mapping. Commercially available examples of these sensors are shown in Figure 2.3.



FIGURE 2.3: Various pressure mapping devices. From top-left then clockwise: XSensor wheelchair pressure mapping sheet (© 2024 XSENSOR® Technology)[5], Pressure Profile Systems pressure sensors on a robotic hand (© 2023 PPS UK limited)[6], Soft pressure mapping gripper(© 2023 PowerON)[7], Tekscan thin pressure mapping platform[8](© 2024 Tekscan Inc.), Tactilus seat pressure mapping system[9](© 2024 Sensor Products Inc.)

Many of these pressure mapping technologies don't accurately mimic desirable qualities of regular biological skin and are specialised for their specific use cases. The following sections quantify characteristics of pressure sensitive skin.

2.1.1 Skin Construction and Types

Skin is a laminate structure consisting of three main layers, the epidermis, dermis, and hypodermis. The top two layers the epidermis and dermis are a subset of the cutaneous layer which contain the majority of the pressure-sensitive mechanoreceptors [33].

The skin can be categorised as glabrous/hairless or non-glabrous/hairy. Glabrous skin contains many of the mechanoreceptors given in Figure 2.1 whereas non-glabrous skin will also contain C-tactile afferent receptors for obtaining sensations through hair follicles. However this work is exploring simple monolithic/homogeneous-composite bodies so will not be replicating the sensor function of non-glabrous skin.

Depending on the region of skin different force resolution and spatial resolution will incur. Relevant cutaneous mechanoreceptors and their functions are given in Table 2.1. The tensile properties of skin is governed by skin tension lines, also called Lager's lines, which show the direction in which the maximal stretch can occur.

TABLE 2.1: Comparison of typical mammalian mechanoreceptor characteristics [15].

| Receptor | Meissner corpuscle A1 | Ruffini Corpuscle A2 | Pancian Corpuscle B1 |
|-------------------------------------|---------------------------------|---|--|
| Perceptual sensory functions | Skin movement, handling objects | Skin stretch, movement direction, hand shape, and finger position | Fine tactile discrimination, form and texture perception |
| Skin stimulus | Dynamic deformation | Skin stretch | Indentation depth |
| Localisation | Dermal papillae | Dermis | Basal layer of epidermis / around guard hair |
| Conduction velocity | 35 - 70 m/s | 35 - 70 m/s | 35 - 70 m/s |
| Receptive field | 22 mm ² | 60 mm ² | 9 mm ² |
| Receptor density | 150 / cm ² | 10 / cm ² | 100 / cm ² |

2.1.2 Characterising skin

The sensing qualities of skin is crucial for the sensory feedback in complex manipulation tasks. To aid the creation of technology that mimics qualities of biological pressure sensitive skin, the mechanical properties must be characterised. Biological human skin is highly variable in terms of its mechanical and sensing properties depending on the region of skin, giving large variation in skin characteristics. Skin can be characterised in terms of the following mechanical characteristics:

1. Elastic modulus - The static elastic properties determined by a linear region of stress and strain of the material. [Pa]
2. Storage and loss modulus - The dynamic elastic and viscoelastic properties determining the relationship between stress and strain. [Pa]
3. Ultimate tensile stress (UTS) - The maximum tensile stress that a material can tolerate before breaking [Pa]
4. Life cycle - The time or number of actuation cycles in which it takes for the actuator to degrade such that it cannot perform its intended purpose to specified standards.

5. Viscoelastic creep and relaxation - All viscoelastic materials will experience strain creep and stress relaxation to varying degrees depending on the viscoelastic properties of the material. [$\text{mm} \cdot \text{s}^{-1}$ and s]
6. Skin thicknesses - the thickness of all layers of skin the cutaneous epidermis and dermis and thickness of the hypodermis. [mm]
7. Skin surface area - Biological skin has a large surface area and can also be regionalised to map skin function and sensitivity. [m^2]
8. Isotropy/Anisotropy - The directionality of skin properties, also known as skin tension lines, give a topological map of the maximal stretch (i.e. minimal elastic modulus) direction of regions of skin.

Some of the functional properties in terms of pressure mapping include:

1. Spatial resolution and touch acuity - The spatial resolution of biological skin, which is mainly dependent on the innervation, mechanoreceptors density, and thickness of the cutaneous layers of skin [34–36].
2. Static force resolution - This is the detection resolution of static or slow-acting forces acting upon the skin [36].
3. Temporal resolution - This is the detection resolution of fast-acting forces acting upon the skin often required for texture recognition [34, 36].

A quantitative characterisation of mechanical and pressure sensing functional skin properties include:

1. Elastic modulus - varies largely depending on test method, test skin type, and subject. Values found in literature include 83.3 ± 34.9 MPa [37], 0.1 - 2.4 MPa [38], and 10.4 - 89.4 kPa [39].
2. Storage and loss modulus - varies largely depending on test method, test skin type, and subject. Values found in literature range include 141.9 ± 34.8 Pa and 473.9 ± 42.5 Pa at 0.8 Hz [40], 473.9 ± 42.5 Pa and 32.3 ± 10.0 Pa at 205 Hz [41].
3. Ultimate tensile stress - 21.6 ± 8.4 MPa [37]. 28.0 ± 5.7 MPa [42]
4. Life cycle - Skin cells are constantly growing, dying, and shedding. Skin is always actively remodelling based on external stimuli [33].
5. Strain creep - The strain creep was found to be 2.7 kPa.s for a 10 Pa step input on a dermis skin sample [40].
6. Skin thicknesses - The thickness of human cutaneous skin ranges from 0.6 to 2.6 mm with an average skin thickness of 2 mm [34].
7. Skin surface area - The average surface area of skin in adult humans is $1.7 \pm 0.1 \text{ m}^2$ [34].
8. Isotropy/Anisotropy - The tension lines in skin are determined by collagen fibre orientation and dynamic stretch events [43, 44]. The elastic modulus of human skin was reported to be 160.8 ± 53.2 MPa parallel to the skin tension lines and 70.6 ± 59.5 MPa perpendicular to the tension lines [42]. The UTS of human skin was reported to be 28.0 ± 5.7 MPa parallel to the tension lines and 15.6 ± 5.2 MPa perpendicular to the tension lines [42].

1. Spatial resolution and touch acuity - The tactile field area increases with indentation depth for certain mechanoreceptors with a range of $5 - 12.6 \text{ mm}^2$ [45]. Two point discrimination is another metric for determining spatial resolution and has been determined as $3.7 \pm 0.7 \text{ mm}$ [46]. The receptive field varies depending on the mechanoreceptors used so has been reported to be between 1 and 60 mm^2 as another methods of inferring spatial resolution [15].
2. Force resolution - Minimum force detection on various regions of human skin was found to be between $67 - 1007 \text{ mg}$ [47], and various mechanoreceptors $0.73 - 122.6 \text{ mN}$ [48].
3. Temporal resolution - Depending on the mechanoreceptor sensing the force input, a frequencies ranges of 0 to 800 Hz can be perceived by human skin [45]

2.1.3 Skin Modelling

Developing robust mechanical models for human skin is non-trivial for three main reasons:

1. High degree of viscoelasticity
2. Self-regeneration and healing
3. Constructed from various types of cells in a laminate structure

To solve the complexity of modelling such a material a review by Landry et al.[34] shows that many researchers have applied various non-linear mechanical models including Ogden, Mooney–Rivlin, Neo-Hookean, Yeoh, Humphrey, and Veronda–Westmann. When recreating an artificial muscle it is desirable to minimise the mechanical material model complexity so that the material can be more easily integrated into a control system with known behaviour. Similar modelling techniques can be used to model conductive particle elastomer composites due to the similar hyper-elastic and visco-elastic behaviours observed.

2.2 Pressure Mapping Artificial Skin Devices

This section outlines some of the main technologies which are flexible and/or soft and comparable softness to human skin tissue and can map force events throughout a surface. A particular focus on electro-active polymer (EAP) based sensing is present due to the potential of miniaturising the technology and the range of miniaturised electronics currently available. EAPs are essentially polymer materials which can be used as transducers which change electrical properties based on a mechanical input, vice versa.

2.2.1 Soft Pressure mapping technology

Pressure mapping devices can be categorised into their various sensing technology, such as resistive, capacitive, inductive, magnetic, optical, and acoustic. Transduction methods have been compared by Tiwana et al.[49], with recommendations to pursue ‘capacitive, resistive, piezoelectric, piezoresistive or a combination’ of methods to replicate mechanoreceptors in the human skin. However, additional optical and magnetic/inductive methods will also be considered in the following sections.

2.2.1.1 Resistive

Soft resistive pressure mapping has been commonly achieved in the past by using arrays of piezoresistive sensor elements, some of which are shown in Table 2.3. The resistive elements can be made using several different flexible piezoresistive materials, such as conductive particle polymer composites[50–52], intrinsically conductive polymers[51, 53?], microfluidic metals[54–56], hydrogel structures [57–59], and flexible piezoresistive semiconductors[60, 61].

TABLE 2.2: Comparison of different potential piezo-resistive sensor materials. Ranked 1 to 5, where 1 is desirable and 5 is undesirable. WIP - need to redo properly and find a reference for each box!

| Material: | Conductivity | Piezo-resistivity | Hardness | Manufacturability | Cost | Durability | Toxicity | Drift |
|-----------------------------|--------------|-------------------|----------|-------------------|------|------------|----------|-------|
| Conducting polymer | 5 | 3 | 3 | 2 | 2 | 3 | 2 | 3 |
| Electrolytic hydrogel | 1 | 2 | 4 | 3 | 4 | 2 | 2 | 2 |
| Conductive particle polymer | 2 | 4 | 4 | 4 | 4 | 4 | 2 | |
| Conductive particle paste | 3 | 2 | 4 | 3 | 4 | 2 | 3 | 2 |
| Conductive textile | 4 | 4 | 3 | 2 | 4 | 5 | 4 | 3 |

2.2.1.2 Capacitive

Similar to resistive pressure mapping, capacitive pressure mapping has more commonly been done using arrays of capacitive elements. Many capacitive touch sensors use the human body to shunt the electric field between the capacitor electrode(s) to a common ground. However, the operating principle of capacitive-based strain sensors rely on the deformation of the capacitor dielectric and/or the capacitor electrodes [62? , 63]

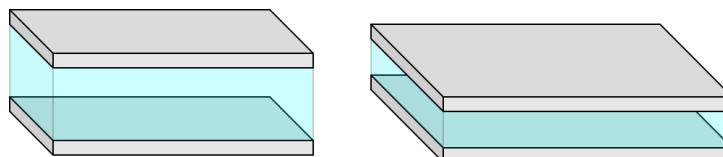


FIGURE 2.4: Two grey electrodes across a blue dielectric medium. Left: Uncompressed state. Right: Compressed state exhibiting larger electrode areas and a thinner dielectric thickness.

2.2.1.3 Magnetic

Magnetic strain mapping devices can be achieved using several methods. One method is to have a three layer stack with hall effect sensors [64]. The stack is made up of a the bottom layer full of rigidly connected three dimensional hall effect sensors, the second layer is made from an elastomer, and the top layer has a magnetic particle unit placed at a set distance above each of the hall effect sensors. The movement of the magnets alters the magnitude and direction of magnetic field sensed and data can be interpolated to create a map of strain deformation. The main advantages of this method is that each hall sensor can detect in three dimensions, hence normal and shear forces can be detected, and using magnetismfor sensing means less electrical noise in the system. The main disadvantages of this method of sensing is the added complexity in scaling the system and the electronics required and the rigid surface required.

2.2.1.4 Optical

There are various methods for making a optically driven artificial skins. A recent review has been curated by Lee et al. [65] all of the different methods of using optics for creating tactile sensors. The main advantages of optical sensors include the high speed sensor response, immunity to electrical noise, and their non-invasive nature. The main disadvantages include, the bulky hardware required for driving the optics and signal processing, the potential interference of external light sources, and the materials that can carry optical signals.

2.2.1.5 Acoustic

Acoustic soft tactile sensing has not been explored much compared to the other forms of sensing given. Park et al., Hughes and Correll [58, 66] have created a system which uses passive acoustic tomogrphy (PAT) to localise and and classify different types of touch. This form of tactile sensing is the most similar to the biological system of mechanoreceptors which are specialised to detect certain frequencies of vibration.

2.2.1.6 Soft Pressure mapping technology comparison

The softness of biological human skin has a large range as discussed in Section 2.1.2. There have been a range of works investigating sensors with a range of softness' and performance. A comparison of these start-of-the-art soft sensor works is given in Table 2.3.

TABLE 2.3: Comparison of soft sensor technologies.

| 1st Author | Sensing principle | Sensing region material | Sensing region elastic modulus or shore hardness | Electrodes per sensing position | Repeatability | Time series data shown | Spatial resolution | Temporal resolution |
|-----------------------|-------------------|-----------------------------------|--|--|-------------------------------------|--|----------------------------------|--------------------------------------|
| Gilanizadehdizaj [67] | Piezo-resistive | Ecoflex30-00 rGO composite sponge | 40 kPa | 2 sensels / electrode | 10 cycles for each stress | - | 10 x 10 mm | - |
| Fu[68] | Piezo-resistive | Carbon black silicone composite | 1.5 Mpa | 0.625 sensels / electrode | 50000 cycles | Yes. | 12 x 12 mm | 60 ms |
| Yang[69] | Piezo-resistive | Ecoflex graphene composite sponge | - | 2 sensels / electrode | 800 cycles | Yes. | 10 x 10 mm | 150 ms |
| Liang[63] | Capacitive | PDMS, PET, Si, Sio2, Cu laminate | 4000 Mpa | 1 sensel / electrode | - | Real-time use of sensor shown. No explicit time-series data. | 4 x 4 mm | - |
| Yan[64] | Magnetic | Ecoflex 00-50 | 83 kPa | 11 IC pins / sensel | 30,000 cycles | Yes. | 0.1 x 0.1 mm | 15 ms |
| Rossiter[70] | Optical | Polymer foam | - | 2 sensels / electrode | - | - | 10 x 10 mm | - |
| Shimdera[71] | Optical | Super clear silicone | 40 A | N/A. One fiber optic LASER and one camera. | Error increase of 1.7% over 30 days | Yes. | approx. 20 x 20 mm / 0 - 1100 um | Sample rate 1.6s. Training required. |
| Ramuz[72] | Optical | PDMS | - | N/A. Two arrays of OLEDs and Organic Photo Detectors used. | 900 cycles | Yes. | Not localised. | 300 ms |

2.3 Biological Muscle form and function

Biological muscles are a product of millions of years of evolution and the motion and other mechanical characteristics of biological structures is yet to be outperformed by artificial muscle technology. To determine how to quantify the performance of a biological muscle this section gives foundational knowledge about muscle function, structure, and how it can be characterised from an engineering perspective rather than the typical biological perspective, so that similar actuator devices with similar attributes can then be investigated.

Biological muscle is a naturally occurring tissue comprised of muscle fibres bundled together to apply a contractile force on connecting tissue or, in the case of smooth muscle, applying a force on itself. The base actuator units of muscle are proteins myosin and actin filaments, which effectively slide against each other to produce a contractile motion. The root cause of a muscle contraction is an electrochemical signal sent from the central nervous system to a motor neuron/s which travel to the muscle where electrochemical reactions take place for the contraction to occur[73]. The sliding motion of the myosin and actin filaments is due myosin

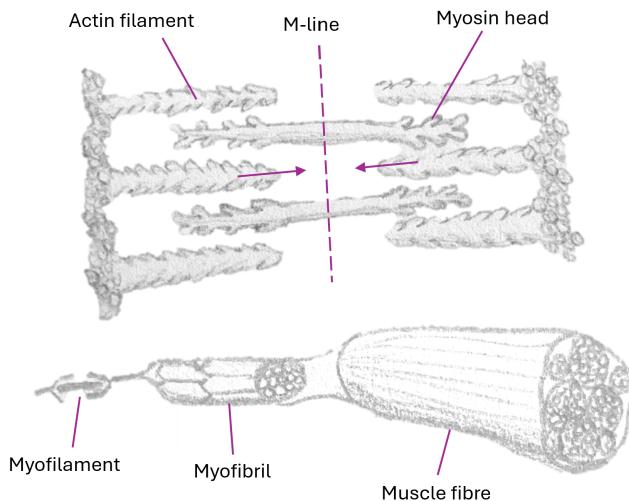


FIGURE 2.5: Components of a biological muscle contractile unit and meta-structure.

heads binding to the actin and pulling the actin towards a middle line (M-line) in multiple stroke actions. These filament actuators are stacked in three dimensions within a muscle fibre to amplify contractile stress and strain as shown in Figure 2.5.

On a macro scale, muscle is made up of bundles of fascicles connected together with a tissue called perimysium. Within the fascicles are many muscle fibres (i.e. muscle cells) which are surrounded by a connective tissue called endomysium. Within the muscle fibres there are many sacromeres stacked within a cylindrical-like structure called a myofibril. Each sacromere contains a contractile unit of myofilaments.

2.3.1 Characterising a muscle

To quantify the performance of a biological muscle, certain metrics are compared. An artificial and biological muscle can be characterised using typical mechanical material parameters such as:

1. Stress - Force that is applied to the normal of the cross section of the muscle through various states of muscle excitation. $[Pa]$
2. Strain - The muscle change of length due to the stress applied through various states of muscle excitation. $[\%]$
3. Elastic modulus - The elasticity determining the relationship between stress and strain for the linear region of the stress strain characteristic curve. $[Pa]$
4. Energy density - The work done by the muscle per unit volume or mass. $[J.kg^{-1}]$
5. Power density - The work done by the muscle per unit volume or mass per unit time. $[W.kg^{-1}]$
6. Ultimate tensile strength - The maximum tensile stress that a material can tolerate before breaking. $[Pa]$
7. Efficiency - The work done by the muscle compared to the energy put into the system, known as metabolic cost in biological muscles. $[\%]$
8. Actuation frequency - The frequency range of actuation cycles using the system's method of excitation. $[Hz]$
9. Stroke - The maximum displacement an actuator can achieve $[m]$
10. Life cycle - The time or number of actuation cycles in which it takes for the actuator to degrade such that it cannot perform its intended purpose to specified standards.

As well as the commonly used medical/biological muscle metric:

11. Maximum isometric contraction force - the maximum force a muscle can apply without changing strain. This is also related to the ratchet-like mechanism and muscle locking where a muscle can apply a much larger force in a static state, as seen in the myosin binding[74].

Other qualities of muscle should be quantified on a case by case basis depending on the artificial muscle technology being investigated. For example, a major issue with dielectric elastomer actuators is the excitation voltage required for actuation is too large for many applications. Hence, excitation voltage could be another parameter considered for some electroactive artificial muscles.

Some of the biological muscle metrics have been quantified by previous research as seen below:

- Energy density - energy densities ranging from 0.4 - 40 $J.kg^{-1}$ [75].
- Power density - power densities ranging from 9 - 284 $W.kg^{-1}$ [76]
- Actuation frequency - natural actuation frequencies ranges 1 to 180 Hz [76].
- Strain - ranging from 5 - 30%[77].
- Efficiency - Thermodynamic efficiency of human muscle is typically between 20-35%[78]. However other biological muscle has been seen to reach efficiencies of up to 77%[78].

2.3.2 Muscle Mechanics

Before attempting to recreate a bio-mimetic actuator it is important to acknowledge the numerous simplified electro-mechanical system models of parts of the muscle actuation process. These models need to be understood to gain an understanding of the application of biomimetic actuators can be used in assistive soft robotic devices. From here we will present basics of the subject of bio-mechanics.

The stress and strain involved in muscle contraction is more complex than uniform materials and is non-linear. The stress and strain of a passive muscle (i.e. contractile units are not producing internal muscle tension) can be modelled with the following equation;

$$\frac{d\sigma}{d\varepsilon} = \alpha \cdot (\sigma + \beta) \quad (2.1)$$

Where ε & σ are strain and stress respectively. A solution for this is first order ODE is;

$$\sigma = \mu e^{\alpha\varepsilon} - \beta \quad (2.2)$$

Where μ is a free parameter determined empirically. The stress-strain of a passive muscle can be likened to tension being applied yarn. As more strands of the yarn are pulled into tension the stress increases, then as the last strands are brought into tension a maximum stress is reached, until the yield stress is reached. Linear approximations can still be made over regions of elongation depending on accuracy required for application. The stress-strain of an active muscle (i.e. when it is tetanised) is approximated to a piece-wise quadratic function or bell curve. It is important to note that the stress for both active and passive muscle is zero when the strain is less than 0.4, demonstrating the yarn-like nature of the muscle stress-strain as shown in Figure 2.6.

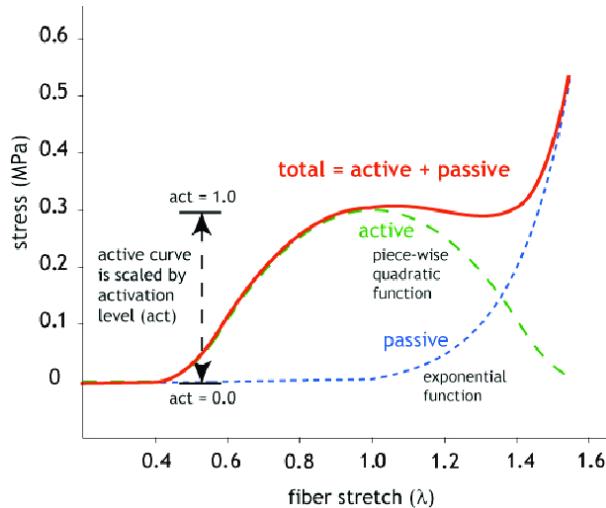


FIGURE 2.6: Stress and strain of active and passive muscles (© J. Teran | ACM 2003)[10]

Hill's muscle models commonly refer to a mechanical three element model [79] composed from, one parallel non-linear spring element, one series non-linear spring element, and a contractile unit.

2.3.3 Electrical Muscle Models

Similar to EAP-based artificial skin and artificial muscles, biological muscles also require electrical stimulation to function. The main method for providing an artificial electrical stimulation to a muscle, to simulate the signal a motor neuron would give to a muscle, is functional electrical stimulation (FES). Due to the biochemical nature of the motor neuron signal transport and the purely electrical stimulation provided by the FES device, the process isn't as efficient as the naturally occurring electro-chemical muscle activation, often resulting in increased muscle fatigue when compared to equivalent voluntary muscle contractions [80]. FES applies a voltage across between two electrodes on the user's skin above a specific muscle. The voltage simulates the signal form and frequency of action potentials between 4 - 12Hz[81]. The threshold for a muscle action potential to cause a muscle contraction is approximately 70 mV [82]. To artificially sense an intended muscle contraction electromyography (EMG) can be used. EMG also commonly uses two electrodes on the surface of the skin above a desired muscle. This EMG signal can be used as a sensor input for joint pose estimation. EMG senses the action potential impulses conducted along motor neurons to the muscle. There are many models for limb motion and EMG- and FES-based therapies [83–86].

2.4 Artificial Muscle Technology

There are many types of electrically actuated artificial muscles technology. Artificial muscle actuator technology that has gained particular interest in recent years include, the ionic polymer-metal composite (IPMC) actuator, the hydraulically amplified self-healing electrostatic (HASEL) actuator, magnetorheological elastomer (MRE) actuators, and dielectric elastomer actuators (DEAs). Each of these having qualities very similar to that of biological muscle usually with a trade-off in actuation response time, actuation force, and actuation strain for their various possible topologies. This section gives a brief overview of four state-of-art soft electromagnetically driven actuator technologies.

2.4.0.1 Ionic polymer–metal composite actuator

Ionic polymer-metal composite actuators (IPMCs) are soft actuators that can be actuated at a much lower excitation voltage than DEAs, commonly less than 10V. IPMCs are also desirable as artificial muscles they have shown large bending deformations, simple to fabricate, light weight and thin in design, and can have a fast actuation response time ($>15\text{Hz}$) at small displacements[87]. IPMCs also have a high work density and maintain a constant volume during actuation like biological muscles[88]. An IPMC is made up of an ionic polymer interlayer, two electrode conductive layers, and a voltage source. The ionic polymer interlayer allows for ionic transport and is typically made of treated Nafion or Flemion. These materials are typically used as ion exchange membranes so have the characteristics desired for the transporting ions during the actuation of the IPMC actuator. The two electrodes are made of a suitably conductive and flexible material. The interlayer is treated such that it is filled with water molecules and cations, with the chemical backbone of the interlayer being slightly negatively charged. When a voltage is applied across the electrodes the cations are repelled from the cathode and travel towards the anode while the water molecules are displaced in the opposite direction towards the cathode. The ionic polymer then swells as the cations repel each other along the anode side of the interlayer, while the polymer elements on the cathode side effectively shrink[89]. This swelling adjacent to the cathode provides the device's bending actuation.

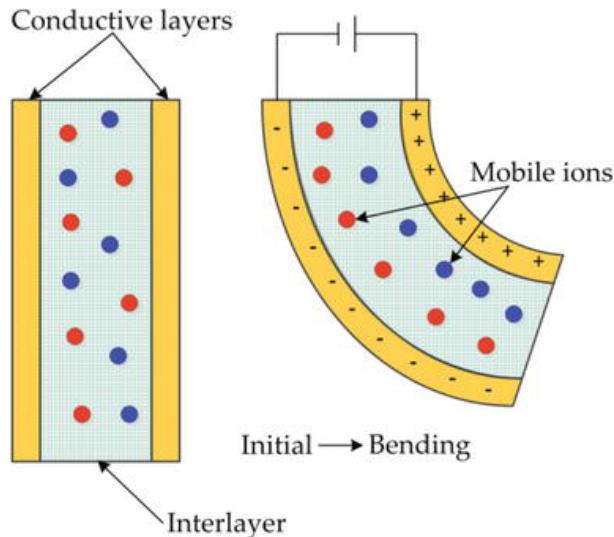


FIGURE 2.7: Diagram of the typical architecture of an IPMC actuator[11] (© 2018 Yanjie Wang and Takushi Sugino)

There are many variations of the design and manufacturing of IPMCs to optimise the actuator for an application as shown by [90]. Although the process of manufacturing IPMCs is simple, it takes a long amount of time (often >48 hours[87]) for the ionic polymer interlayer to absorb the necessary ions and undergo the necessary reactions. There has been much research into the optimal manufacturing of an IPMC [90–92]. The use of additive manufacturing has been used successfully to generate more complex geometries using fused filament deposition[93].

IPMCs can also be used as sensors. When an IPMC undergoes bending due to an external force there is a potential generated across the electrodes, which indicates bending direction and magnitude[94].

Two key deficiencies of current IPMC actuator technology are the maximum force output achievable and the life cycle of the actuator in a dry (non-aqueous) environment. The force output optimisation of IPMCs has been investigated by several researchers, all of which having a maximum actuation force in the milli-newton scale [94–96]. Because the IPMC actuators rely on hydrated ionic transport to actuate this means if the IPMCs are in a dry environment then over time they will decrease their maximum actuation force.

The applications of this actuator is limited to applications requiring a small actuation force and a wet environment. Current applications include flexible catheters [97], small biomimetic robotics [98, 99], aquatic robotics[100, 101], with many other applications yet to be discovered.

2.4.0.2 HASEL actuator

A hydraulically amplified self-healing electrostatic (HASEL) actuator is a recent soft actuator technology developed in 2018[12] which displays many qualities that are better than current artificial muscle technology. HASEL actuators are made up of three main components: electrodes, dielectric fluid, and an elastomeric shell. The electrodes need to be highly conductive, able to handle high electric potential, and can be solid or flexible. Hydrogel electrodes have been proven to be a good material for the electrodes because of their elasticity while still maintaining a high conductivity[102]. In one application the hydrogel material is bonded to a polydimethylsiloxane (PDMS) substrate for mechanical strength and for ease of bonding to the actuator biaxially-oriented polypropylene (BOPP) shell[12, 57]. HASEL actuators use high electric potential across

two electrodes to create an electrostatic force. This force induces a zipping effect which pulls the electrode together from one end to the other as the electric field strength increases. The zipping of the two electrodes pushes the dielectric fluid into the reservoir increasing the pressure which alters the shape of the reservoir bounds providing an actuation motion. When the electrodes have displaced all of the fluid between them the actuation displacement is at a maximum. The electrostatic zipping action allows a large force to be generated due to snap-through transition. Snap-through transition is an actuation instability which has been discussed in previous research as a means of amplifying DEA actuation strain[103]. Recorded efficiency values of HASEL actuators

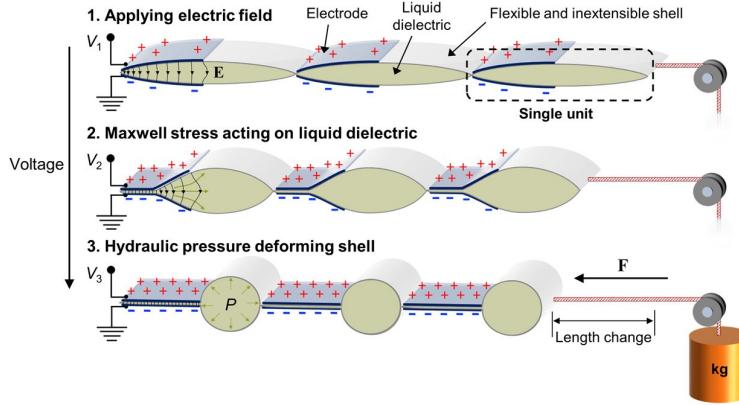


FIGURE 2.8: Diagram of the typical architecture and the contraction stages of a HASEL actuator[12]

actuators of 21% are comparable to that of human muscles of 20 - 35% [78]. The actuators have had a frequency response of up to 20Hz. Large strains of 124% have been recorded, but can only be achieved when actuating at a resonant frequency. Strains of up to 79% have been recorded using a linear planar HASEL actuator configuration and DC voltage stepping. Else, strains of only 10% have been recorded for static steady strain[12]. Because there is a relationship between the motion of the actuation and capacitance between the electrodes, this means self sensing can be achieved through the electrodes. Although due to the flexible and fluid nature of the device, modelling of the HASEL is difficult and limited in accuracy.

The simple and commonly used manufacturing process for HASEL actuators is completed in six steps as shown by the diagram below:

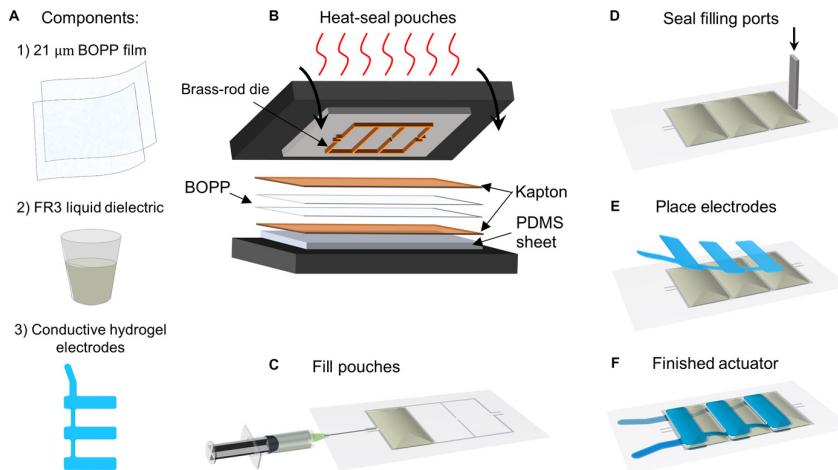


FIGURE 2.9: Diagram of the simplified stages of HASEL actuator production[12]

Other attempts have been made to use polyjet inkjet based additive manufacturing to make the whole HASEL actuator and have been successful with proof of concept, but are yet to be developed from prototype stage[104].

The cyclic life of HASEL actuators are high, because of their self-healing properties. When there is a dielectric breakdown through the liquid dielectric the damage caused is not permanent like when a DE breaks down. The liquid may form some small air bubbles, however these may not effect the operation of the actuator, instead this can increase the likelihood of another dielectric breakdown. The cycle life of the HASEL actuator was seen to be larger than one million with a given torus shaped HASEL actuator[102]. The HASEL technology is promising with a number topologies possible, some topologies include toroidal, planar linear[102], and scorpion metasoma(tail)[105].

2.4.0.3 Dielectric Elastomer Actuators

The dielectric elastomer actuator (DEAs) are often called artificial muscles because they share similar characteristics to biological muscle such as, the large strains achievable, the high elastic energy density, many topologies/configurations achievable, and constant volume during its contraction.

A DEA consists of a dielectric elastomer (DE) film sandwiched between two compliant electrodes. To excite the actuation, a high electric potential is applied to across the electrodes creating an electrostatic force between the two compliant electrodes. This force pulls the two electrodes together applying stress (known as Maxwell's stress) to the elastomer and hence strain parallel and perpendicular to direction of the electrostatic force. When the DEA is contracted the surface area of the electrodes increases and the thickness of the DE decreases causing a change in capacitance and Maxwell's stress. A dielectric elastomer actuator can be modelled

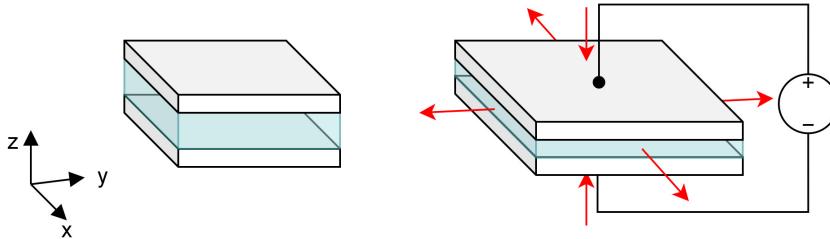


FIGURE 2.10: DEA with two compliant light-grey electrodes and a transparent light blue dielectric elastomer. Showing deformation without and with a voltage applied across the electrodes.

as a flexible parallel plate capacitor in its simplest form. Using this we can determine the electrostatic pressure to be:

$$\sigma_{es} = \epsilon_0 \epsilon_r \frac{V^2}{z^2} \quad (2.3)$$

Where σ_{es} is the electrostatic pressure, ϵ_0 and ϵ_r are the vacuum and relative permittivity constants, V is the voltage potential applied across the electrodes and z is the thickness of the DE. The electrodes used for a DEA need to be made of a conductive material, but require similar elasticity to the dielectric material. An ideal material for these electrodes would have high conductivity. This conductivity would change minimally and predictively under large strains. Many composites have been used in practice for these electrodes, with the most common in early development being a silicone rubber and carbon powder composite. However, the unpredictable nature of carbon powder elastomer composites has lead to research into many

other materials/silicone additives such as hydrogels, graphene sheets, metallic nanostructures, carbon nanotubes, liquid metal[106–109]. The ideal material for the dielectric elastomer should have a high elastic modulus and a high electric breakdown voltage. The elastic modulus needs to be sufficiently high so that less electrostatic pressure can create a larger strain. While the breakdown voltage of the material needs to be sufficiently high such that the material will not break down at the maximum desired strain. If a material can be found with a high enough electric breakdown strength at a smaller thickness than current research prototypes then a higher stress can be achieved giving a larger or equivalent actuation force at a lower voltage.

Many other topologies exist to generate different actuation motions using the same electrostatic pressure generation principle. These include actuator topologies such as stack[110, 111], helical[112], bending[113], lens[114], cylindrical, and rolled shaped actuators[115]. Each of which having a range of applications.

DEAs are often fabricated in a laboratory environment using a pre-strained elastomer. The pre-straining accomplishes four key qualities; stores elastic strain energy, ensures DE is planar within the bounds of the jig, controls the initial thickness of the DE, and puts the DE in an optimal stress-strain region, often taking advantage of elastomer hyper-elasticity. There is no standard practice for the fabrication of DEAs, other methods such as additive manufacturing have also been explored to generate more complex geometries and to increase production speed[116, 117].

As well as actuating, DEAs can also be used for sensing. DEAs can be used as sensitive capacitive sensors, where any strain applied to the DE will relate to the effective capacitance between the two electrodes[28, 118, 119].

Currently dielectric elastomer actuators all require voltages within the kilo-volt range to generate an adequate stress and strain for a range of applications. A key problem encountered by researchers designing DEAs is the trade-off between actuation force and strain magnitude [110]. This high voltage requirement may deem the technology dangerous for use where there is a possibility that a human may come into physical contact with the high voltage electrodes.

2.4.0.4 Magnetorheological Elastomer

Magnetorheological elastomer (MRE) actuators, also known as magnetoactive soft materials (MSMs), are a relatively new form of actuator however the theory reinforcing operating principle has been known since at least the 1980s [120]. The structure of an MRE actuator generally consists of a ferromagnetic elastic composite and a driving magnetic field. An example of this is a composite of iron-carbonyl powder and PDMS. The operating principle of MREs is that magnetic flux travelling through the MRE will change mechanical characteristics within the elastomer (i.e. stiffness or displacement of the body). The operation of a MRE actuator is similar to a DEA however instead of having an electric field cause a contraction it is a magnetic field causing a deformation. An MRE is typically made of silicone rubber containing magnetic ferrite based particles uniformly distributing throughout its volume. This kind of actuator is current controlled and can hence operate at a low voltage. This helps mitigate the risk of electric shock of a device in close proximity to humans (unlike HASEL actuators and DEAs). A key issue with using magnetorheological elastomers as soft actuators is that they require heavy gauge conductors for the high current they require for generating a magnetic field. The high current requirement means that actuators have only been created that have a solid electromagnet driving a soft MRE[121].

When manufacturing MREs, uncured liquid silicone rubber is mixed with magnetic (commonly carbonyl iron) particles to form a 3 dimensional matrix of crosslinks with the magnetic particles

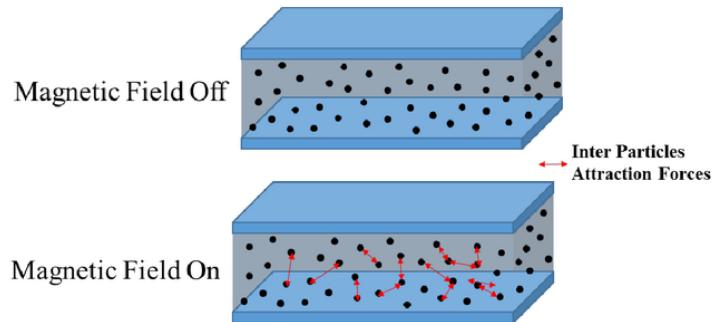


FIGURE 2.11: Diagram showing MRE contraction actuation when a magnetic field is applied[13]

fixed between the crosslinked polymers. A core issue when creating an MRE is the agglomeration and corrosion of magnetic particles due to residual water within the mixing operation. The magnetic particles can be processed to have a hydrophobic quality to mitigate this issue [122, 123]. During the curing process a magnetic field can be applied to align the particles within the elastomer to control the particle isotropy[123, 124].

There have been attempts to use additive manufacturing to make MREs[123, 125], however the method described has not optimised the structure of MRE for any application and the particle dispersion throughout the MRE has not been proven uniform throughout the print volume.

The current applications of MRE actuators are limited, however magnetorheological fluid (MRF), is a fluid which becomes more viscous with an applied magnetic field as currently has many modern applications. This fluid substance is largely used in applications where damping control is desired such as vehicle suspension[126], medical assistive devices[127] and helicopter seat damping [128]. Potential MRE actuator applications include fluid valve control[121] and active vibration control similar to that mentioned for MRFs[126].

2.5 Soft Conductive Particle Piezoresistive Composites

Soft sensors and actuators require low-stiffness materials for their active sensing/actuation domains. The requirement of softness is governed by the mechanical modulus values depend on the application requirements. The use of conductive particle elastomer composites is explored in this work due to the customisability of the electromechanical characteristics. A core part of this thesis is understanding the behaviour of conductive particle elastomer composites for their use as a range of EAP-based sensing and actuating devices. The characteristics that make conductive particle elastomer composites (CPECs) ideal for soft sensor and actuator devices often include, low stiffness, controllable conductivity, controllable piezoresistivity, mouldable, 3D printable, low toxicity, durable, inexpensive, easy to obtain, simple fabrication process, and sustainable[123, 129–131].

2.5.1 Fabricating Conductive Particle Elastomer Composites

Before exploring the known conduction and piezoresistive mechanisms and models for CPECs, it is important to understand how the fabrication process of a CPEC may affect its physical structure.

CPECs are made by dispersing conductive particles through a curable liquid elastomer matrix. To change the electromechanical properties of the material, the dispersion of the conductive

particles throughout the matrix can be optimised through various methods. To minimise the agglomerations of primary conductive particles often a sonication step is completed. This involves a mixture of the conductive particles and a liquid, usually in the form of a solvent, to be placed in a ultra-sonication bath.

The sonication bath performs a frequency sweep and it has been shown that sudden implosion cavitation near the agglomerates help cause the separation of the agglomerates into their primary particles[132, 133]. The degree of deagglomeration and dispersion is affected by various factors including sonication time, frequency of oscillations, oscillation intensity, particle wettability, and liquid matrix viscosity[133, 134].

This sonication usually occurs before the the particles are added to the elastomeric matrix due to the large viscous damping effects of liquid elastomers. The next step involves mixing the dispersed conductive particles throughout the liquid elastomer, this can be done using a variety of mixing methods, including a planetary mixer, magnetic mixer, screw mixer, static mixers, amongst others [131, 135–137]. During the mixing process often the liquid solvent used in the dispersion stage is evaporated, leaving only the curable elastomer and the conductive particles. Although often impurities and voids are a by-product of the previous processes which can give undesirable qualities.

When sufficient mixing of the liquid elastomer and conductive particles have been completed the material is formed into a desired final shape using advanced additive manufacturing methods [117, 125, 138–142] or traditional moulding [143] or film making techniques [144]. During the moulding process the material undergoes a form of curing, such as UV, catalysed, or moisture curing. If the composite material has not already been integrated into a device containing electrodes and other mechanical support structures these are integrated at the end of the process.

2.5.2 Conductive Particle Elastomer Conduction Mechanisms

Depending on the fabrication process stages stated in Section 2.5.1 for fabricating CPECs, the dispersion of conductive particles will always vary.

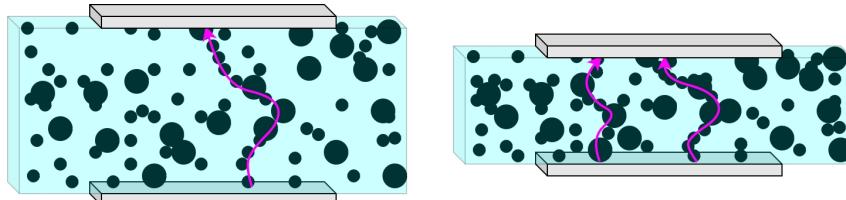


FIGURE 2.12: Two grey highly conductive electrodes across a CPEC cuboid showing enlarged black conductive particles within a blue polymer matrix. Left: An uncompressed CPEC. Right: A compressed CPEC.

Some of the physical features of these conductive percolation networks can be quantified and directly relate to the macro-level electromechanical properties of the material. Such characteristics of a conductive percolation network include, the type of conductive particle(s) used, particle dispersion, the elastomeric matrix, and any impurities or voids. The aspect ratio of a conductive particle filler can drastically change the conductivity and piezoresistivity of a CPEC. For example the aspect ratio of carbon nanotube particles (CNTs) is very large compared to that of regular carbon black (CB) particles, this has been shown to give improved conductivity for smaller weight volume percentages[145, 146], among other electromechanical property changes.

Also the inherent particle conductivity a core parameter to consider when choosing a conductive particle composite.

Conductive particle dispersion is an important characteristic of CPECs when optimising the electrical properties of a CPEC. Particle dispersion includes the inter-particle distance distribution[131], particle agglomeration distribution[135], particle isotropy/anisotropy [147], and sedimentation[148]. The filler elastomer matrix also contributes to the piezoresistive effect, through it's viscoelasticity, elastic modulus, and dielectric permittivity within the CPEC.

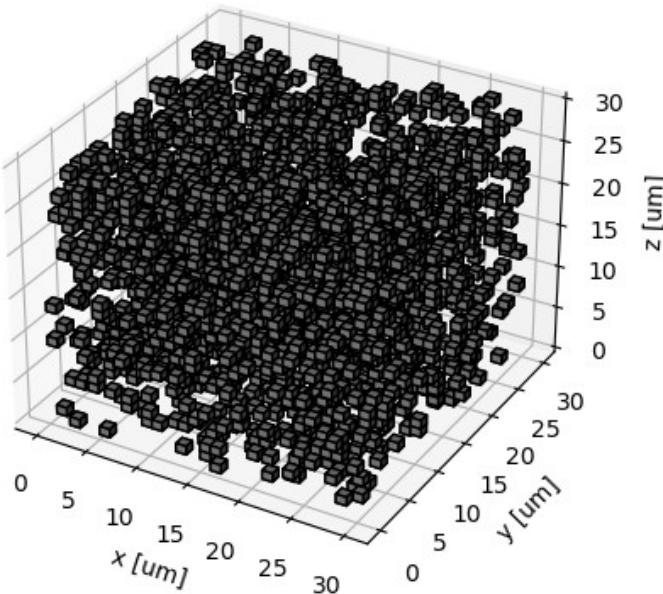


FIGURE 2.13: Example of a randomised cube percolation with a volume percentage of 8% of $1\mu\text{m}$ particles

Microscale models for CPECs and the relationship between particle and electric charge motion are often computationally heavy, overly idealised, and non-invertible [149]. A microscale model example can be seen in Figure 2.13. However, microscale modelling of CPECs may give insight into understanding complex physical phenomena that may relate to the macroscale models made for CPECs. An alternate method for modelling CPECs is the formation of macroscale models[150].

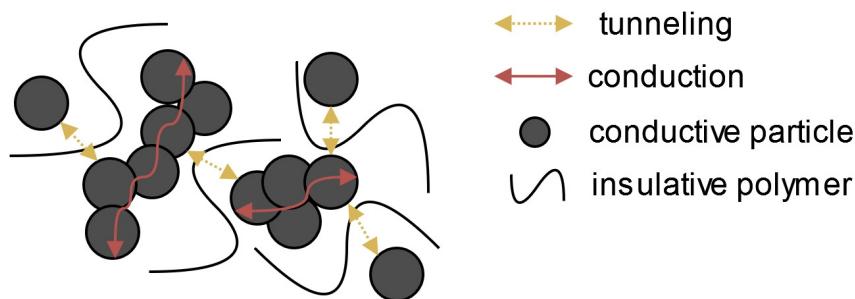


FIGURE 2.14: Electrical conduction and tunneling representation diagram.

Electrical DC conduction through a CPEC occurs via two main mechanisms, electrical conduction and quantum tunneling [151–154]. Electrical conduction uses the conduction band electrons

shared by adjacent atoms to allow movement of electrons throughout chains of cascading these conductive atoms. The second mechanism of conduction is through quantum tunneling which is stochastic in nature and allows for conduction through insulative boundaries between the percolative network of conductive particles [155, 156].

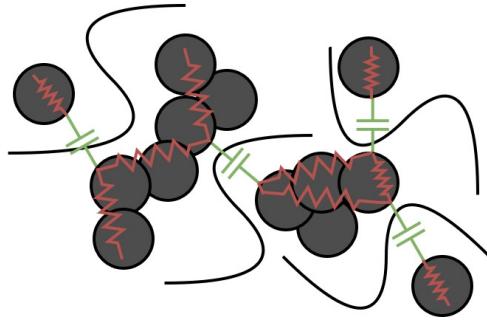


FIGURE 2.15: AC conduction RC network representation diagram.

The a CPEC can be modelled with an RC network as shown in Figure 2.15. Electrical AC conduction can occur through a CPEC through capacitive means depending of particle spacing with a decrease in reactance becoming more prominent for composites near the percolation threshold[130].

2.6 Literature Review Conclusions

The original purpose of this thesis was to develop novel sensor and actuator technology that mimics the pressure mapping capabilities of human skin and combine this with the actuation properties of human muscle. Through this review of current literature, several key conclusions can be drawn that will lay the foundational knowledge for the rest of this work.

The review of biological skin has revealed quantitative parameters that define its mechanical and sensory capabilities. This review highlighted mechanical characteristics such as the elastic modulus, viscoelastic creep, and surface area, as well as functional properties like spatial and temporal resolution. These factors provide a foundation for designing artificial skin that can replicate or even surpass the sensing functions of soft human skin. The review on pressure mapping technologies was then completed showing a range of different transduction methods for similarly soft sensing domains, showing that replicating human mechanoreceptor sensation is a multifaceted problem. Human skin uses various mechanoreceptors with different qualities and trade-offs and similarly different pressure mapping technologies use different pressure transduction methods each with different performance characteristics and limitations. The parallel review on biological and artificial muscles showed that DEAs and HASEL actuators are promising technologies for mimicking biological muscle quantitatively. Although characteristics common to both technologies such as high actuation voltage and limited device lifetime limit the applications of them.

The thesis has converged on using CPECs to fabricate EAP sensor and actuator devices, hence a brief literature review highlighting CPEC fabrication techniques and electromechanical characterisation has been given. These composites exhibit beneficial properties like flexibility, tunable electromechanical behavior, and ease of fabrication, which make them suitable for integrating into soft robotic systems. However, challenges such as achieving uniform particle dispersion, minimising agglomeration, and optimising the conductive network for stable long-term operation are still active areas of investigation.

This literature review has given a brief overview of some of the devices and theory related to the thesis, throughout the thesis there will be more background theory given on a need-to-know basis for each chapter.

Chapter 3

A Simple Conductive Elastomer Composite with Complex Behaviour

Abstract

3.1 Introduction

As discussed in the Literature Review chapter, conductive particle elastomer composites are desirable for soft sensor and actuator applications for a variety of reasons. However, it is crucial to understand the electromechanical behaviour of these composites if we wish to create complex control systems with such materials. Although conductive particle elastomer composites (CPECs) are simple in concept, consisting of dispersed particles through an elastomer matrix, the electromechanical behaviour is not well understood on a macro or micro-scale. This chapter will focus on only tensile stress of the specimen, and how it relates to the electrical resistive relaxation. Compressive characterisation and modelling is covered in the subsequent chapters. This work endeavours to understand the material behaviours of carbon black silicone rubber composites on a macro-scale to help create better inverse models so that the material can be used more accurately for stress/strain sensors.

3.1.1 Background

Carbon nanoparticle-silicone elastomer composites are stretchable conductive materials with diverse applications such as, highly elastic strain sensors [52, 143, 157], dielectric elastomer actuators [158, 159] and electromyography electrodes [143, 160, 161]. Understanding the dynamic resistance relaxation characteristics of carbon black (CB) silicone rubber (SR) elastomer composites would improve performance in fields which require high efficiency of space, power and accuracy, such as the devices used in biomedical and aerospace fields. Unlike many common strain gauges, CBSR composites can have strains of over 300% without yielding [162] depending on the type of SR and CB used and the method of fabrication. This strain and the strain used in this work is higher than commonly used constantan strain gauges, which typically have a maximum strain of $\pm 3\%$ [163], with traditional metal alloy based strain gauges often having significant plastic deformation after less than 10^4 cycles [163] at this 3% strain.

Some characteristics of CBSR composites which make it suitable for strain sensors include that, the material is relatively inexpensive, readily available, non-toxic, bio-compatible, and can have

a high gauge factor. Other alternative composite conductive particles, such as carbon nanotubes (CNTs) [164, 165] and metallic particles [109, 166], have been seen to be more carcinogenic than CB alternatives [167–169]. The fabrication of CBSR composites requires a degree of optimisation to ensure that the carbon particles are adequately dispersed to ensure high conductivity and high yield strength of the material. More importantly the homogeneous dispersion of CB particles means improved repeatability of experimental results and more accurate models for the eventual applications of CBSR composites. A comprehensive model of how the resistivity changes with strain has not yet been developed.

While previous work from our research group [170, 171] has focused on the response to quasi-static and low-strain-rate behaviour, these materials show dynamic effects where a significant resistance strain-rate relationship is present. The main characterisation investigated in this work for CBSR sensing involves understanding the relationship between the mechanical stress relaxation, electrical resistance relaxation and strain in time. A difference in time constants between the stress and resistance relaxations have been noted before in literature [164, 165, 172, 173], but never accurately modelled with the physical theory explained. Mersch et al. [174] have classified several transient ‘shoulder’ events and their related deformation events, compressive, tensile, and bending. These transient peaks have been observed by several researchers using the similar CBSR materials, however there is no conclusive mathematical model relating these transient peaks to strain in time. An understanding of this resistance relaxation and other transient phenomena would mean an accurate model could be made to predict the relationship between stress, strain and resistance within a CBSR composite.

Determining a stress-strain-resistance model would also allow us to understand the limitations of using this composite in sensing and actuation applications, so that the material can be used simultaneously as an actuation excitation electrode and a strain sensor. Understanding these characteristics may give rise to new applications of the composites material, for example, if the resistive relaxation properties of the material were known, it could be used as a mechanically activated timing device. An oscillatory flexible dynamic circuit has been demonstrated when mimicking the motion of a caterpillar as shown by Henke et al. [158], where the resistance relaxation modelling is be useful for more accurate electrical circuit dynamics. The theory behind mechanical stress relaxation is widely known and has been modelled using a variety of mathematical models [14] depending on the material modelled.

It is known that silicone rubber composites are viscoelastic materials and clearly exhibit the three traits of a viscoelastic material [14]: stress relaxation, strain creep, and stress-strain hysteresis. Stress relaxation is an effect observed when a step input of strain is applied to a material and there is a transient stress decay response which converges to a steady state value. A commonly used model for viscoelasticity is the generalised Maxwell body model of order n shown in Figure 3.1.

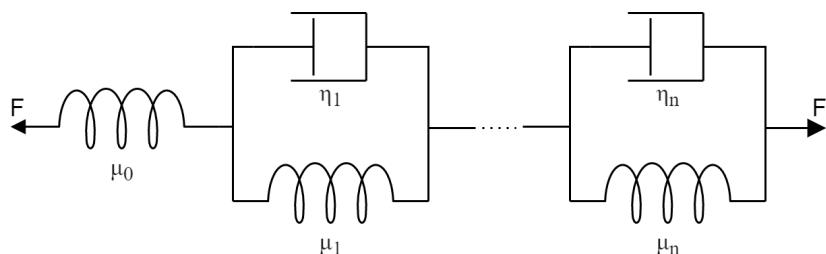


FIGURE 3.1: Mechanical spring dashpot diagram of the generalised Maxwell body model adapted from Fung et al. [14]

Where F is the force applied to the material, and μ and η values represent the spring and damping component constants, respectively. The stress relaxation function for this model is found in Equation 3.1, for, n , serial repeating units.

$$G(t) = a_0 + \sum_{i=1}^n a_i \cdot e^{-t/\tau_i} \quad (3.1)$$

Where a_0 , a_i are the magnitudes of relaxation and τ_i are the relaxation decay time constant components. All of the constants a_0 , a_i , and τ_i are functions of η and μ .

We initially assume that there is a relationship between the stress relaxation and resistance relaxation of the material. However, the generalised model can easily over-fit the data, if n is too high. To mitigate over-fitting, a generalised model with the minimum amount of repeating units while maintaining a high R^2 is used.

Few mathematical models describing the viscoelastic-resistance relationship have been formulated. Laaraibi et al. [175] developed a time-invariant model which uses a relaxation parameter to account for the viscoelastic effect. Mersch et al. [174] developed a model using transverse and longitudinal viscoelastic parameters to model a similar CBSR material. Both models show promise towards creating a more comprehensive physical model. However, the both models aren't tested for a randomised range of input strain signals.

3.2 Methods

The core experimental part of this chapter will be described from composite fabrication through to simultaneous strain tensile tests and resistance measurements. This is followed by the analysis data for resistance-strain phenomena, to quantitatively match the phenomena these to black-box and viscoelastic models.

3.2.1 Composite Fabrication

The CBSR composite was composed of Vulcan XC-72 CB powder (Fuel Cell Store, Bryan, USA) and two part Pt cured Dragon Skin 10 NV SR (SmoothOn, Macungie, USA). The CB powder has an average particle size of 50 nm and typical bulk density of 96 kg/m³. This grade of SR was chosen due to the following characteristics low elastic modulus, E , of 186 kPa tensile strength, σ_Y , of 2.75 MPa, and a low mixed viscosity, η , of 6,000 cps [176]. This E value is within the range of human skin tissue and the low η facilitates material processing in potential future applications with additive manufacturing.

The volume resistivity of pure CB powder itself is between 10^{-1} and $10^2 \Omega \cdot \text{cm}$ depending on how densely the particles are packed and the purity of the CB [52]. The ability of a CB matrix embedded within a highly insulative SR substrate to become conductive is determined mainly by the dispersion of the CB particles, and the tunneling that occurs between conductive CB and insulative SR bodies within the material volume [52, 165]. The composite being created must be highly conductive without compromising the elastic modulus and yield strength of the material. From percolation theory observed in literature [52] there is a threshold volume percentage of CB required to ensure that conductivity is maintained with certainty throughout the composite volume within the linear volume resistivity region. The percolation threshold for composite used in this work was difficult to predict due to the unknown configurations of agglomerations and dispersion of CB particles within the composite material. Empirically it was found that a

CB volume percentage of 7.5% or greater meant the composite material had a resistivity of less than $3.5 \text{ k}\Omega \cdot \text{cm}$ consistently with the fabrication method used.

The first fabrication step was to mix the CB nano-powder with part A of the liquid SR using a KK-50S planetary mixer (Kurabo, Osaka, Japan). A mixing function was used with specific rotational velocities and times for each axis, which was well suited towards de-aeration and viscous particle mixing. The composite mixture was then mixed with the cross-linker part B of the liquid SR using the same planetary mixing function to ensure adequate dispersion of the CB particles throughout the SR volume.

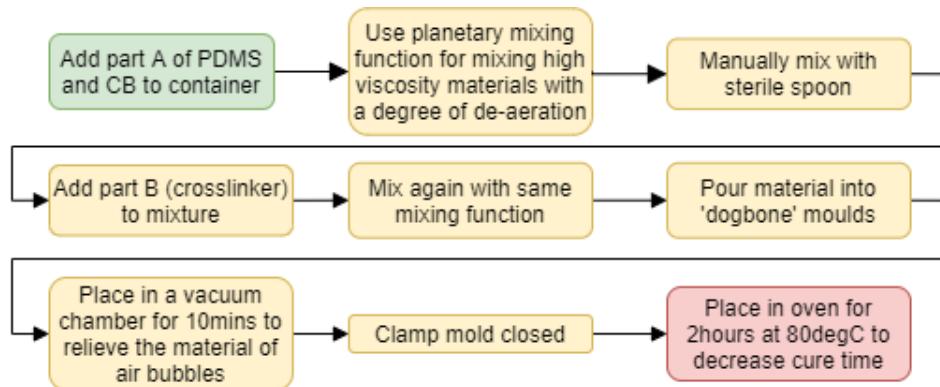


FIGURE 3.2: The steps involved in creating the CBSR composite material

For the fabrication of the CBSR specimens, a standard dog-bone shaped mould was developed for the mixed CBSR to cure in, based on ASTM standard D412 [177]. Before the mould was clamped shut the composite filled mould was immediately placed in a vacuum chamber for ten minutes to de-aerate the still liquid, curing CBSR mixture. The specimen was placed in a lab oven at a temperature of 80 °C for a two hours to accelerate curing. It has been shown that an increase in curing temperature for two part SR increases elastic moduli and decreases yield strength [178, 179].

3.2.2 Material Imaging

To determine how the microstructure may effect the macro-behaviour observed in the following electro-mechanical testing of the material, various optical imaging methods were used, including optical microscopy, scanning electron microscopy (SEM), and Raman spectroscopy.

The initial step was to observe the fabricated CBSR composite specimen using a stereoscopic microscope to view the internal structure of the CBSR composite specimens as shown in Figure 3.3.

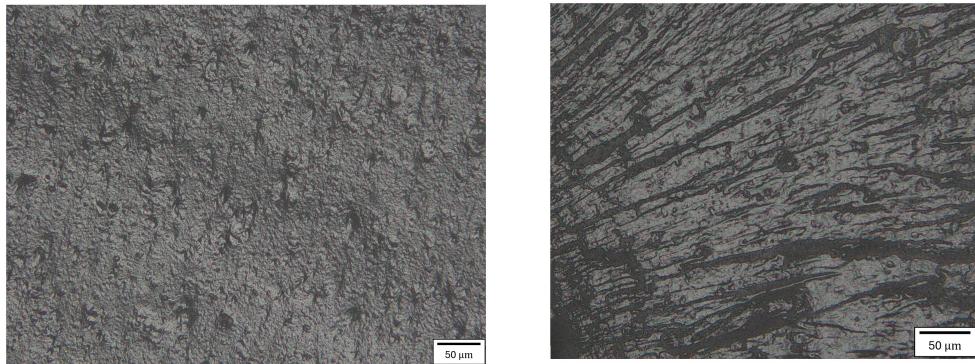


FIGURE 3.3: Microscope images at x20 magnification of a CBSR specimens with cross-sections made by; Left: Cutting with a scalpel. Right: Inducing an embrittlement fracture.

It is known in literature that preparing flat planar surfaces through elastomer composites for micro- and nano-scopic imaging is non-trivial [180]. Due to the silicone material elasticity and a low embrittlement point of -60 to -70 °C , traditional polymer composite specimen machining methods cannot be used if a flat surface is desired. Because of the lack of water content in the CBSR composite traditional biological specimen preparations methods are also not feasible.

Two methods were trialled to prepare the cross-sectional surface of the specimen for imaging, a scalpel cut and an induced embrittlement fracture. The scalpel cut was completed at room temperature conditions aligned vertically with the specimen. The induced embrittlement fracture used required extreme cooling by pouring liquid nitrogen over the specimen for 2 minutes, then snapping the specimen in two.

The induced embrittlement fracture shown in Figure 3.3 shows a very rough surface finish due to the many localised stress concentrations formed around voids and CB particles in the composite, and the non-crystallinity of the elastomer. The scalpel incision method showed an improved surface finish with a low amplitude undulating surface finish.

To observe smaller features such as voids and CB particle dispersion within the CBSR specimen the Apreo 2S FEG-SEM (Thermo Fisher Scientific, Waltham, USA) was used. Initially the CB particles were imaged validating their nominal primary particle size and displaying the agglomerated structure as shown in Figure 3.4. The nominal particle size was found to be 48 ± 20 nm randomly sampling and averaging over 30 particles observed via SEM images. The top surface edge of the CBSR specimens were imaged to highlight the difference in surface roughnesses. Finally the dispersion of the CB particles was attempted, however due to surface charging of the relatively highly insulative silicone matrix the image resolution was limited as shown in 3.4.

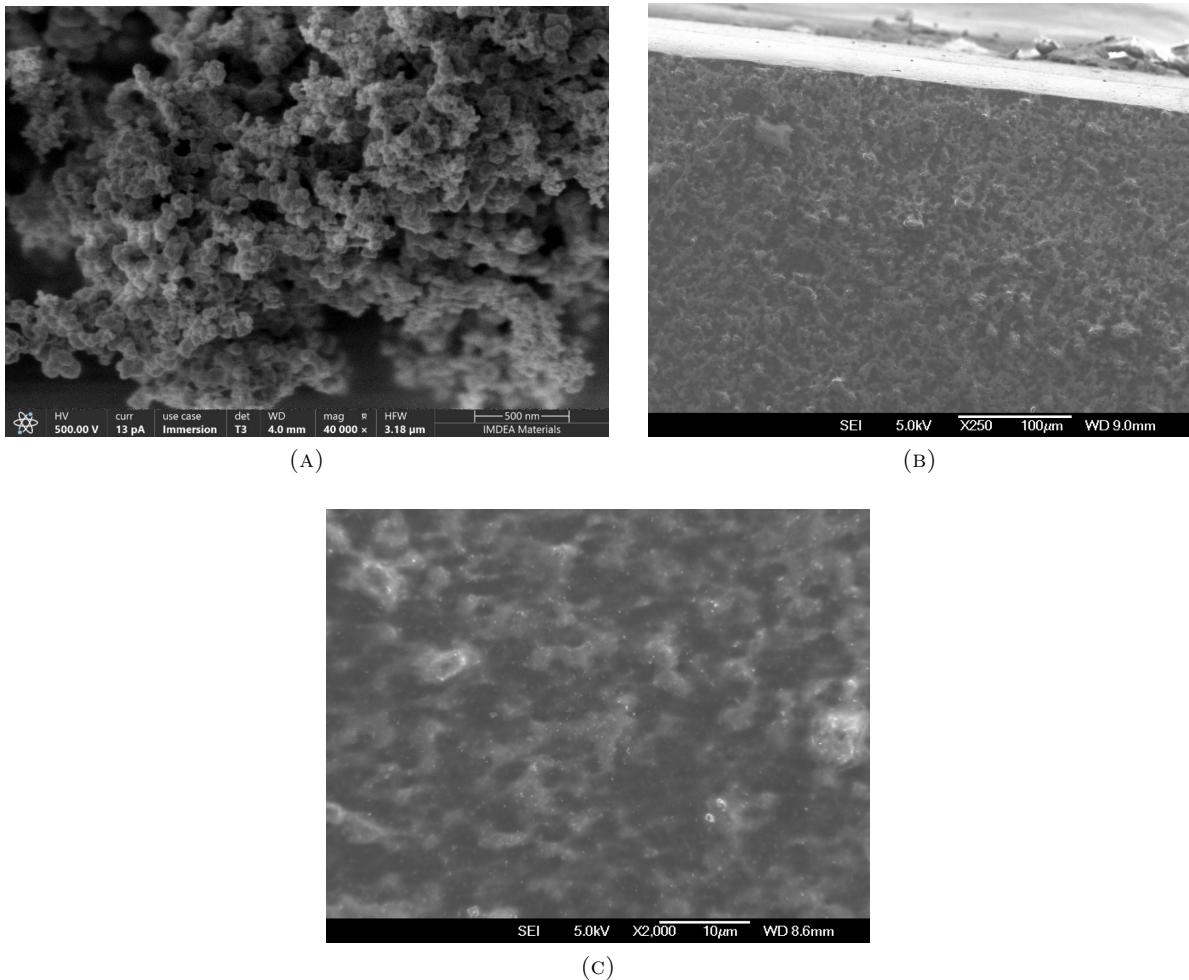


FIGURE 3.4: SEM images of; (A): CB agglomerated particles $\times 40,000$. (B): CBSR cross-section top surface edge site 1 $\times 250$. (C): CBSR cross-section site 1 $\times 2000$.

When measuring CBSR specimen for bulk electrical properties a reliable electrical connection to the material's conductive matrix is necessary. Obtaining high conductivity connection between the measurement electrodes and the material volume was investigated. The surface of the cured CBSR specimen is smooth and has proven highly insulative upon measurement, indicating that there is a thin insulative silicone film around the edges of the specimen as shown in Figure 3.4b. This surface insulation film existed in each side of the specimen, potentially indicating that the viscosity of the SR matrix is high enough to prevent the CB nanoparticles from settling on the bottom surface of the mould used. The white dots shown in Figure 3.4c represent carbon particles throughout the SR matrix. Resolution was limited due to the insulative SR matrix charging from the electron beam. This resolution discrepancy is apparent when comparing to the more conductive CB particles in Figure 3.4a.

To determine the presence and dispersion of CB particles on the surface of the CBSR specimens a inVia confocal Raman microscope (Renishaw, Wotton-under-Edge, United Kingdom) was used. A 532 nm laser with a was used in the Raman microscope. Several surface and cross-sectional images were taken showing a considerable difference in the CB-SR ratio as shown in Figure 3.5. The prominent 1350 cm^{-1} and 1600 cm^{-1} intensity peaks indicate the presence of CB and the prominent 490 cm^{-1} , 2906 cm^{-1} , and 2965 cm^{-1} peaks indicate the presence of SR [181, 182].

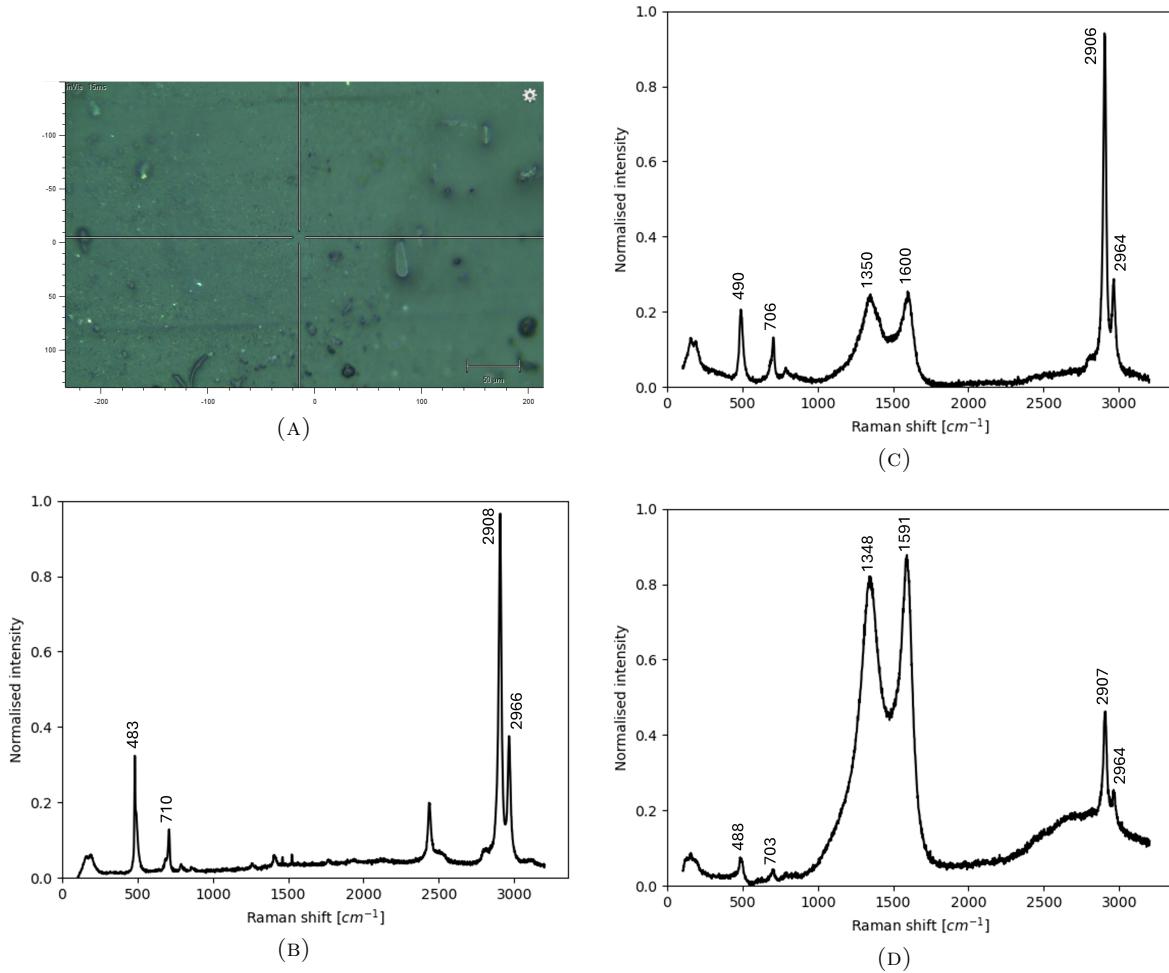


FIGURE 3.5: (A): Typical screenshot from the focused Raman microscope. Normalised Raman spectrum of (B) the plain SR. (C) the top surface of CBSR specimen. (D) the cross-sectional surface of CBSR specimen.

These plots in Figures 3.5b - 3.5d show that surface electrodes will have a smaller chance of obtaining a reliable connection to the conductive network to the internal the CBSR composite, due to the low CB particle content measured at the surfaces on the specimens.

3.2.3 Stress-Strain-Resistance Measurement

A custom test measurement device was made for measuring the desired characteristics of the CBSR material, so that parameters driving the data collection such as the current source and strain profile could be easily customised to suit the experiment. The strain, stress, and resistivity of the specimen were measured in parallel. The setup included the use of a 500 g TAL221 loadcell (HT Sensor Technology Co. Limited., Xi'an, China) in combination with a linear actuator stage driven by a NEMA23 stepper motor and a 2634B Keithley source measurement unit (SMU) (Tektronix, Beaverton, USA). The loadcell was found to have a resolution of ± 30 mN and the linear actuator stage used a G201x micro-stepper controller (Geckodrive, Santa Ana, USA) allowing for 40 μm steps.

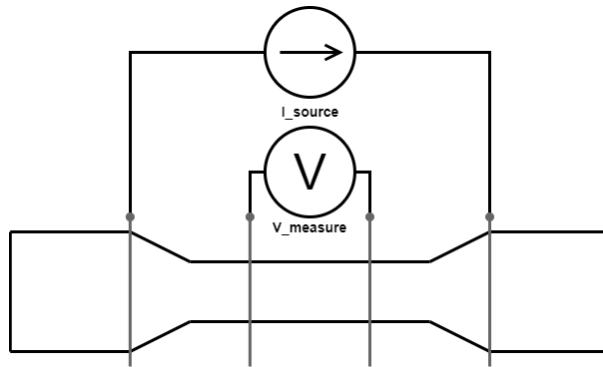


FIGURE 3.6: The composite dog-bone test specimen pierced by 4 metal pin electrodes. The outer and inner electrodes connected to an SMU current source and voltmeter respectively

The four-wire method uses four pin electrodes as seen in Figure 3.6. The four-wire method applies a constant current source through the outer electrodes and uses a voltmeter on the inner two electrode to determine the resistance of the specimen. The four-wire electrode configuration exhibited a larger signal-to-noise-ratio (SNR) compared to the two-wire alternative for resistance measurements. Four-wire pin electrodes were selected because they resulted in minimal specimen deformation, exhibited a high measurement SNR, no slip, and the electrode can pierce through the specimen which ensured a constant electrode impedance. The inner pin electrodes were symmetric about the centre and placed 20 mm apart with the outer pin electrodes being 40 mm apart as shown in Figure 3.7.

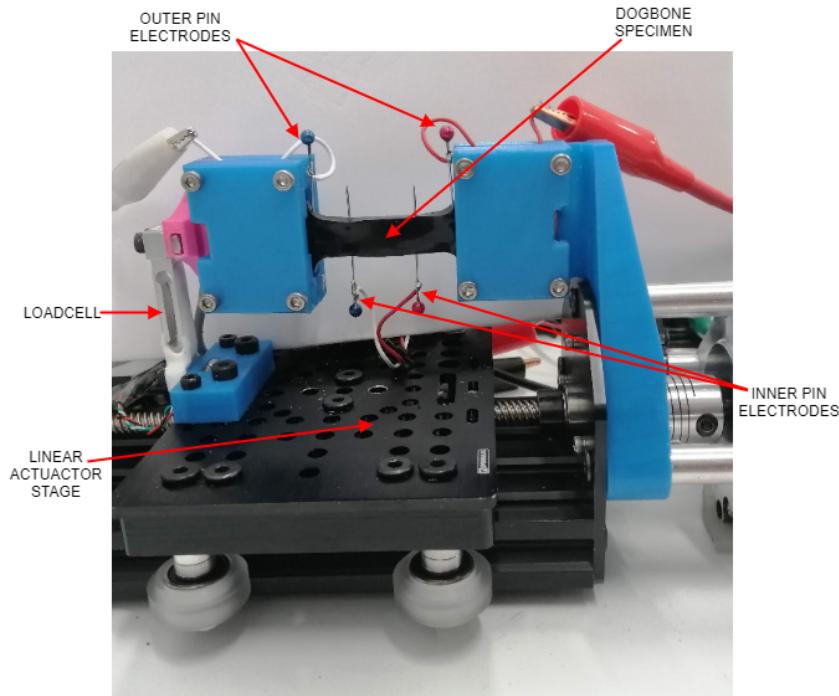


FIGURE 3.7: Photo of test measurement setup

The dogbone specimen was held in place using clamps with a 3D pyramidal array surface. The specimen clamp strain was calculated using the specimen Poisson's ratio and the maximum expected tensile experiment strain; to ensure the specimen would not slip, and ensure minimal deformation to the specimen.

The measurements were completed using controlled sinusoidal, saw-tooth, and pulse trains of strain to ensure repeatability of the models were consistent across varying experimental parameters. If this material is used as a sensor the model fitted to the stress relaxation must hold over many consecutive tensile strain events. As these materials are intended as large strain sensors, the main strains tested in this work were ranged between 0 and 30%, with a strain-rates ranging from 0 to $6.67 \% \cdot s^{-1}$.

All data was captured through serial interfaces and logged on a PC with a python-based program. An open source G-code platform GRBL was used to control the motion of the linear actuator. The PyVISA library was used to obtain all of the measurement data and to send commands to measurement devices. A calibration procedure ensured that the stress and strain of the specimen was always zero'd before each experiment. The functional architecture of the system is shown in Figure 3.8.

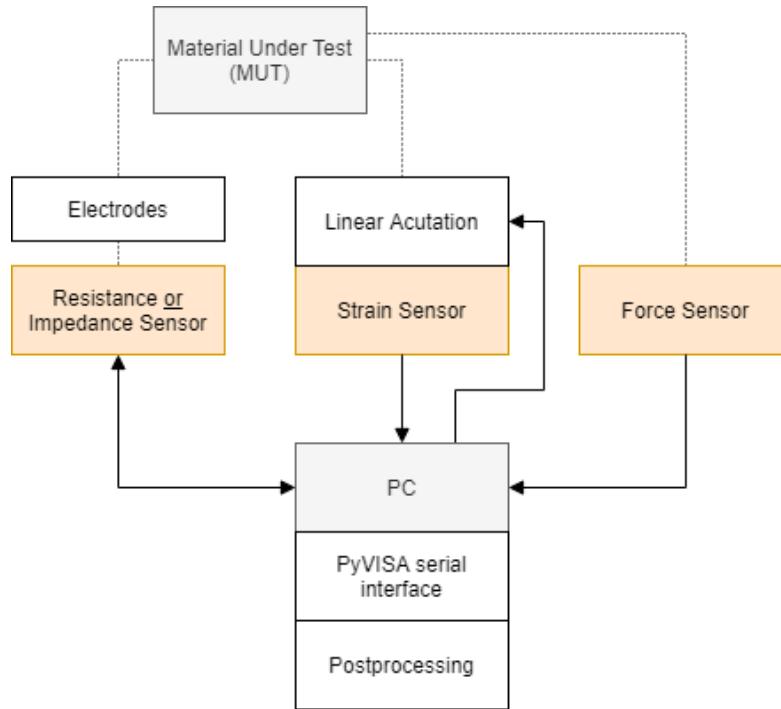


FIGURE 3.8: System architecture for the electromechanical tensile test rig showing data/physical connections

3.3 Results and Analysis

The CBSR composite material has shown complex nonlinear behaviour in previous literature as discussed in Section 3.1, hence a piece-wise approach to modelling the CBSR composite has been taken to describe the electromechanical dynamic behaviours of the material. To understand the transient behaviour of CPECs several dynamic repeatable electromechanical characteristics of CBSR have been classified and mathematical representations fitted. This section aims to provide repeatable mathematical relationships for various phenomena towards creating an overarching model which combines each of the phenomena.

3.3.1 Stress-Strain and Viscoelasticity

All of the specimens fabricated indicated a degree of viscoelasticity shown by the hysteresis seen when loading and unloading the material with 30% tensile strain in Figure 3.9. The 0, 7.5, and 10 w.t.% CB specimens have average elastic moduli, as measured in the loading phase, of 205.2 kPa, 321.4 kPa, and 342.1 kPa, respectively.

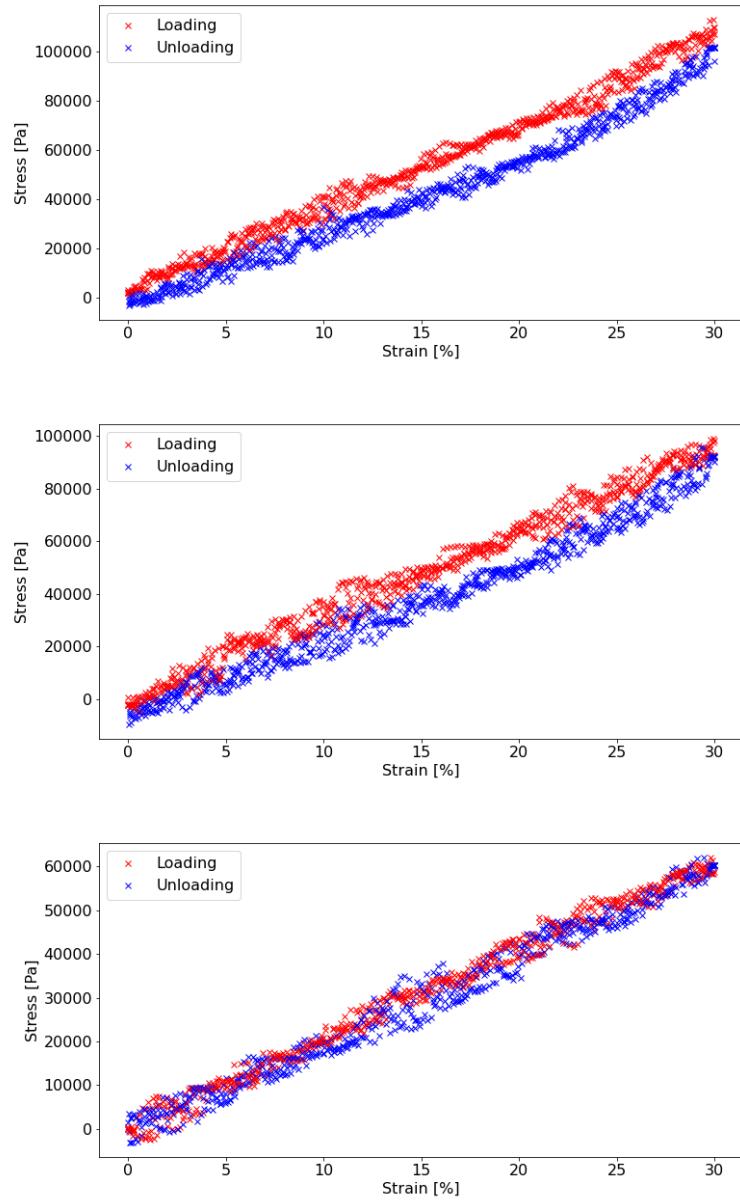


FIGURE 3.9: The loading and unloading of 30% strain on a composite test specimens with CB weight percentages from top to bottom of 10%, 7.5%, and 0% with data collected over five loading and unloading cycles

3.3.2 Quasi-Static Tensile Resistance-Strain

To understand how the material could be used for a low-frequency response application the underlying steady-state relationship between resistance and strain was found by producing a

quasi-static linear resistance-strain formula for strains between 0 to 30% strain. Data was gathered using steady-state measurements from four separate tensile strain pulses performed on the same CBSR specimen as shown in Figure 3.10.

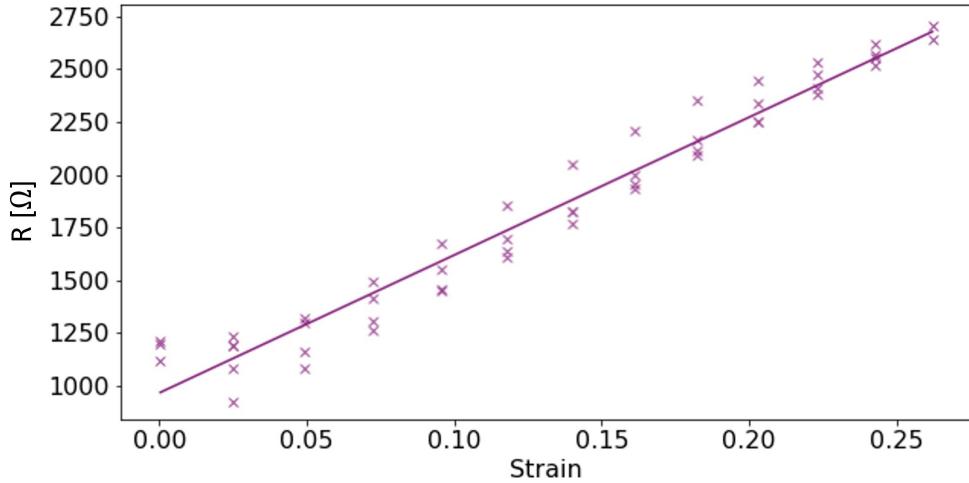


FIGURE 3.10: Linear quasi-static resistance-strain fit for 7.5 w.t.% CBSR specimen

The fitted parameters for the linear quasi-static model are given in Equation 3.2.

$$R = 6530 \cdot \varepsilon + 970 \quad (3.2)$$

3.3.3 Dynamic Resistance-Strain-Rate Response

When using this material as a tensile strain sensor many applications will require a strain-rate superseding the capabilities of the quasi-static model given in Section 3.3.2 due to the resistive relaxation in the material characterised in Section 3.3.5. The dynamic characteristics of the CBSR material is investigated to find a relationship between the strain-rate and resistance. Several saw-tooth strain signal experiments were completed using four different strain-rates of 1.67, 3.33, 5, and 6.67 $\text{%}\cdot\text{s}^{-1}$. To avoid measuring a time dependent relationship instead of the desired strain-rate dependent relationship, each set of different strain-rates were repeated as shown in Figure 3.11.

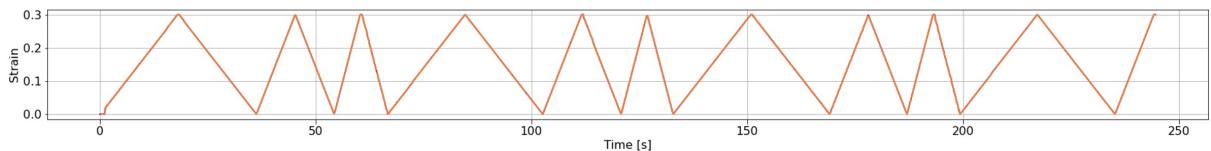


FIGURE 3.11: Strain input waveform for investigating strain-rate dependent resistance

The resulting resistance response exhibits a shoulder peak phenomena where a second peak is developed when suddenly decreasing the strain-rate. The resistance response is shown in Figure 3.12.

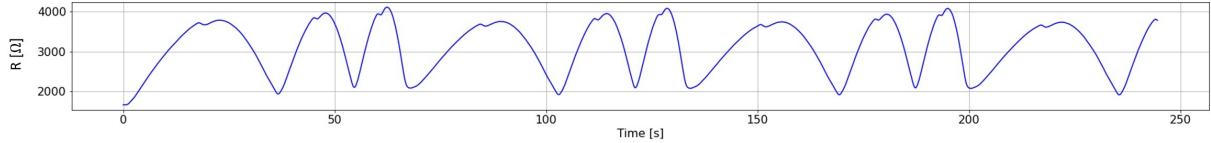


FIGURE 3.12: Resistance response from saw-tooth strain input Figure 3.11.

Hysteresis loops were used in Figure 3.13 to show resistance offset as a function of the strain-speed and simultaneously displaying the resistance hysteresis loops of different strain speeds.

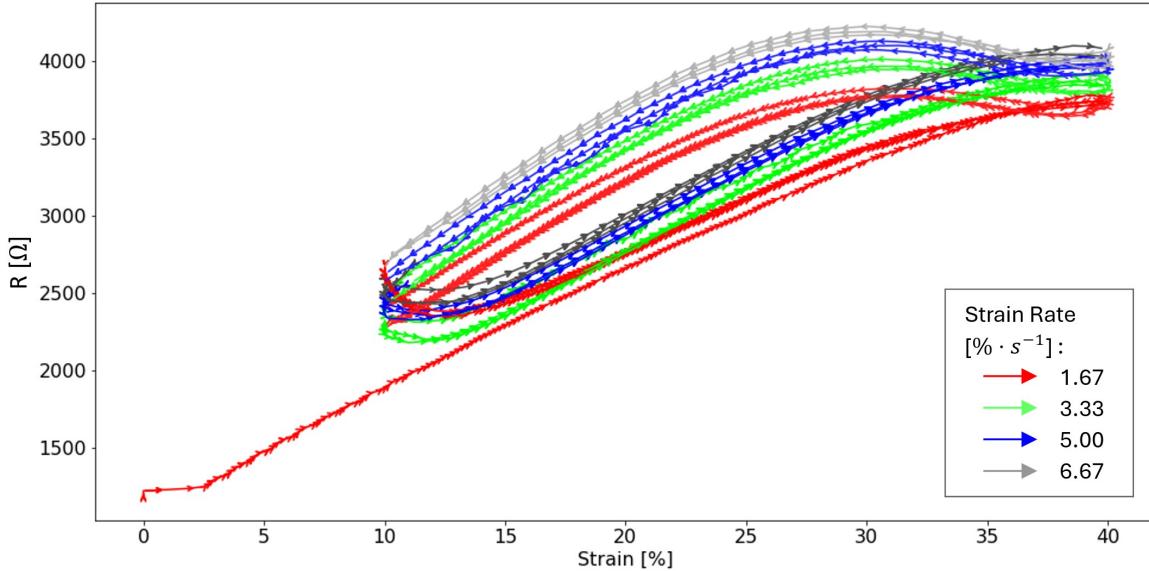


FIGURE 3.13: Resistance vs strain hysteresis loops from a saw-tooth strain input.

The material was pre-strained to mitigate non-linear effects that may arise near 0% strain. Using the $1.67 \text{ \%}\cdot\text{s}^{-1}$ strain-rate as a reference the average resistance offset of the other three strain-rates tested were calculated as shown in Table 3.1.

TABLE 3.1: Strain-rate dynamic resistance offset comparison. Using S1 as the reference resistance loop.

| Strain-rate # | S1 | S2 | S3 | S4 |
|---|------|------|------|-------|
| $\dot{\varepsilon} [\text{\%}\cdot\text{s}^{-1}]$ | 1.67 | 3.33 | 5.00 | 6.67 |
| $\Delta R_{S1} [\text{\%}]$ | 0 | +5.0 | +8.3 | +10.7 |

3.3.4 Falling Edge Strain-Rate-Resistance

A narrow peak in the measured resistance has been observed in the collected data when during a falling edge. This peak is not present in the stress plot shown in Figure 3.14, hence is a proposed characteristic of electrical behaviour only as a function of strain as the change in resistance is assumed to be primarily due to conductive particle motion.

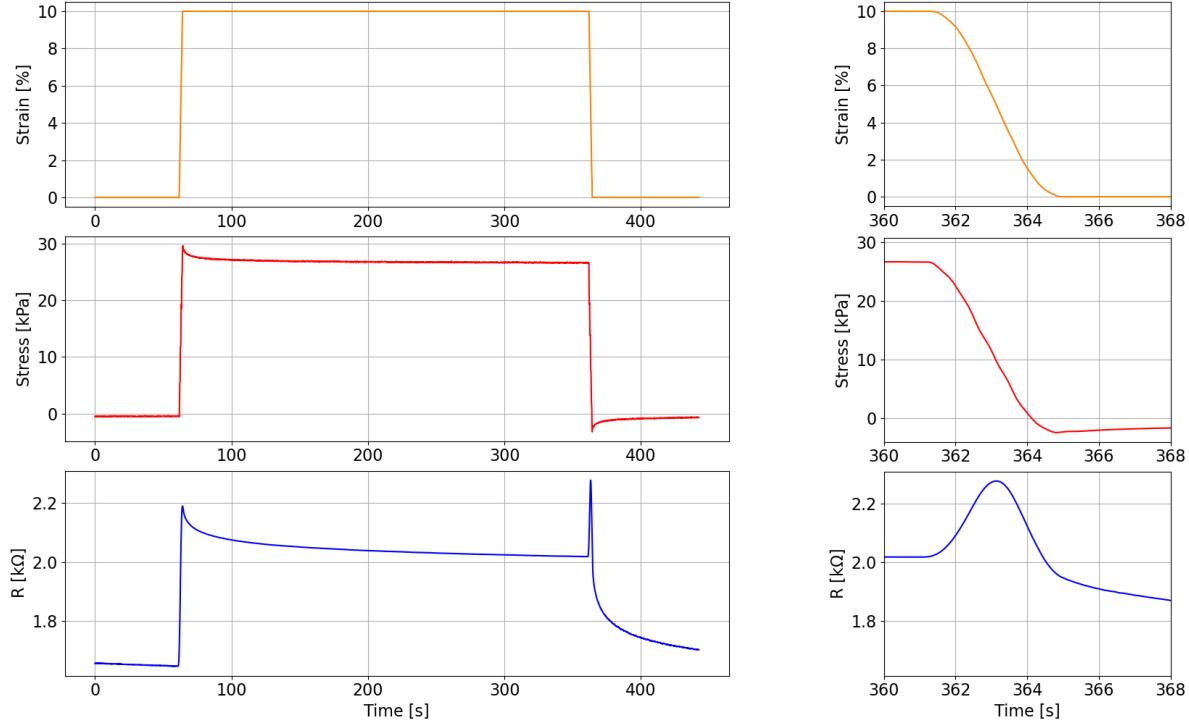


FIGURE 3.14: Left: Singular tensile strain pulse with resulting stress and resistance response.
Right: Zoomed to highlight falling edge shoulder phenomena.

A parameter fit study has been completed to determine the resistance peak amplitude can be determined with a range of strain-rates. We can see a repeated phenomena in Figure 3.15 whereby the derivative of the resistance signal seems to be equal to the strain curve. The resistance peak curve, R_p , can be modelled with a second order polynomial. When differentiated, this peak gives a linear function in a similar form of the linear strain curve seen in Figure 3.15. Hence we form Equation 3.3 which relates resistance to strain as a function of time.

$$\frac{dR_p}{dt} = J(\varepsilon) \cdot t + c \quad (3.3)$$

where J is a function of strain, $\varepsilon(t)$, and c is an offset bias determined by the initial strain condition.

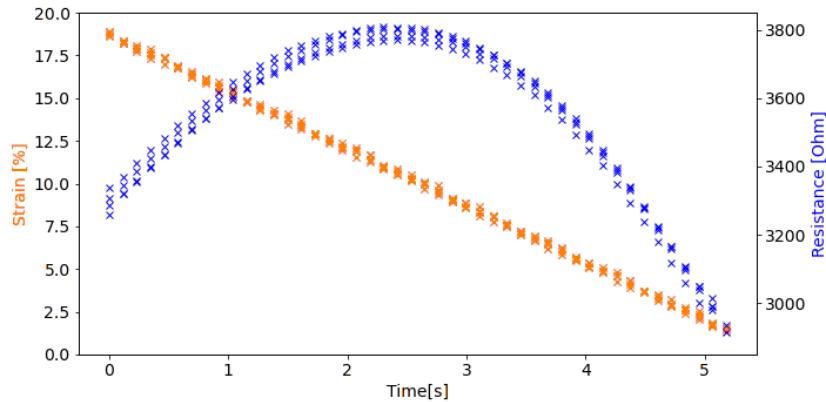


FIGURE 3.15: Strain-rate resistance relationship showing the specimen returning to a 0% tensile strain state from 10% at a strain-rate of $3.33\%\cdot s^{-1}$ from four tests for a 7.5% CBSR specimen

To show the strain-rate resistance relationship, more strain pulse train tests of 20% strain were completed. Using 20% strain allowed observation of sufficient data points to observe a trend. The pulses had four repetitions with a range of strain-rates, $\dot{\varepsilon}(t)$, of 1.67, 3.33, 5.00, and $6.67\% \cdot s^{-1}$. Using a 7.5 w.t.% CBSR specimen we obtain a relationship that agrees with the strain resistance component equation 3.3. As $\dot{\varepsilon}(t)$ increases through strain-rates in parallel with the magnitude of the resistance peak relative to the previous steady state of value resistance. Two formulae are fitting to the linear strain and resistance peak features given by Equations 3.4 and 3.5 respectively.

$$\varepsilon = A \cdot t + B \quad (3.4)$$

$$R_{peak} = C \cdot (t - D)^2 + E \quad (3.5)$$

The resulting plots fitting of the averaged data points for the strain and resistance peak curves are given in Figure 3.16.

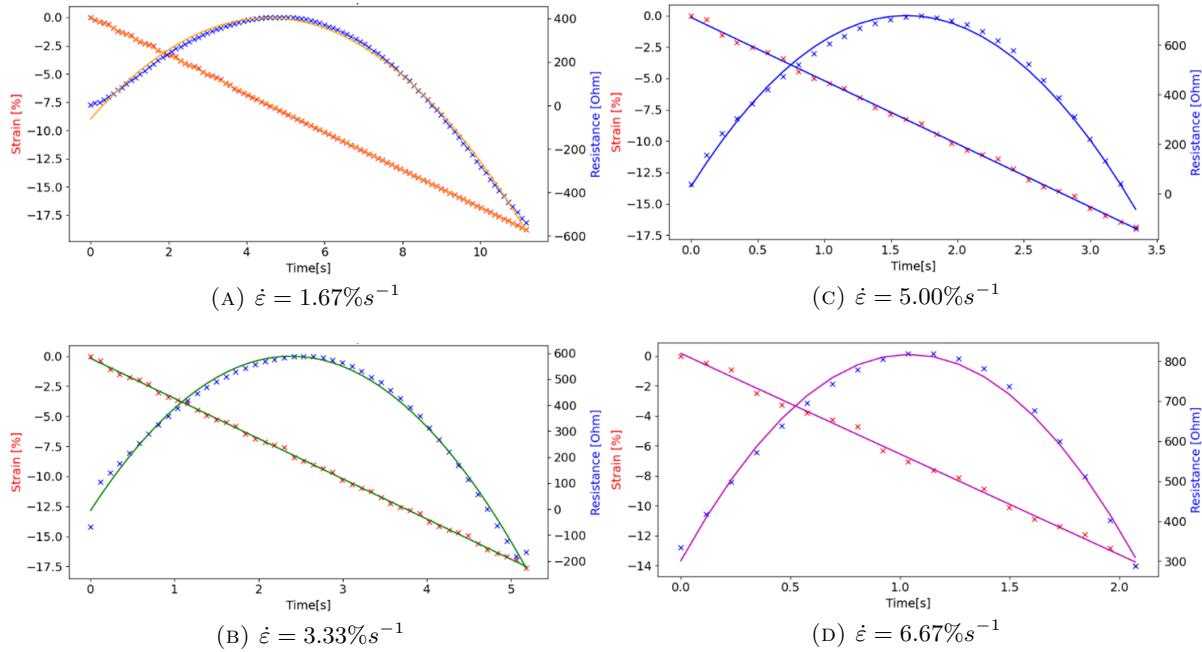


FIGURE 3.16: Fitting curves to a range of different tensile falling edge strain strain-rates.

TABLE 3.2: text

| Strain rate [%/s] | 1.67 | 3.33 | 5.00 | 6.67 |
|---------------------|--------------------|--------------------|--------------------|---------------------|
| A ($\times 10^3$) | -16.83 \pm 0.09 | -33.40 \pm 0.02 | -50.34 \pm 0.36 | -66.82 \pm 0.37 |
| B ($\times 10^3$) | -0.45 \pm 0.93 | -0.69 \pm 1.20 | -1.77 \pm 1.72 | -0.13 \pm 2.15 |
| C | -22.10 \pm 0.55 | -103.19 \pm 2.30 | -250.40 \pm 9.95 | -478.81 \pm 32.87 |
| D | 4.58 \pm 0.02 | 2.37 \pm 0.02 | 1.60 \pm 0.06 | 1.12 \pm 0.05 |
| E | 403.27 \pm 13.52 | 586.87 \pm 23.46 | 711.96 \pm 7.19 | 809.21 \pm 26.46 |

3.3.5 Resistance Relaxation Fitting

The initial model chosen to fit the stress and resistance relaxation data was the generalised Maxwell body model shown in Figure 3.1 with $n = 3$ cascading elements using Equation 3.6 to fit the model. Fitting the data using Levenberg–Marquardt non-linear least square algorithm over 30 data sets showed an instability with the algorithm using this model. When feeding

the previously fitted stress relaxation model parameters as initial conditions for the fitting of the next stress relaxation data set, the values of the parameters diverged exhibiting signs of overfitting. This divergence of the model parameters gave a large standard deviation showing the model was changing significantly each iteration of fitting. Hence a more simple model using Equation 3.1 with $n = 2$ was used to fit the stress relaxation data to Equation 3.7 with lower standard deviation of the model constants. Conversely when the resistance relaxation model analogous to stress relaxation model, shown in Equation 3.8, was fitted to the resistance relaxation data there was a stable fit with a better goodness of fit.

The decay time constants of the two models are different with the resistance having an longer overall decay which can clearly be seen in Figure 3.17. Below in stress relaxation models $G_{1,2}(t)$, shown in Equation 3.6 and 3.7, the constants a_{0-3} and τ_{S1-S3} represent the components of magnitude and time decay of the stress relaxation, respectively.

$$G_1(t) = a_0 + a_1 e^{-t/\tau_{S1}} + a_2 \cdot e^{-t/\tau_{S2}} + a_3 \cdot e^{-t/\tau_{S3}} \quad (3.6)$$

$$G_2(t) = a_0 + a_1 \cdot e^{-t/\tau_{S1}} + a_2 \cdot e^{-t/\tau_{S2}} \quad (3.7)$$

Analogously for the resistance relaxation function $H(t)$, the constants b_{0-3} and τ_{R1-R3} represent the components of magnitude and time decay of the resistance relaxation, respectively.

$$H(t) = b_0 + b_1 \cdot e^{-t/\tau_{R1}} + b_2 \cdot e^{-t/\tau_{R2}} + b_3 \cdot e^{-t/\tau_{R3}} \quad (3.8)$$

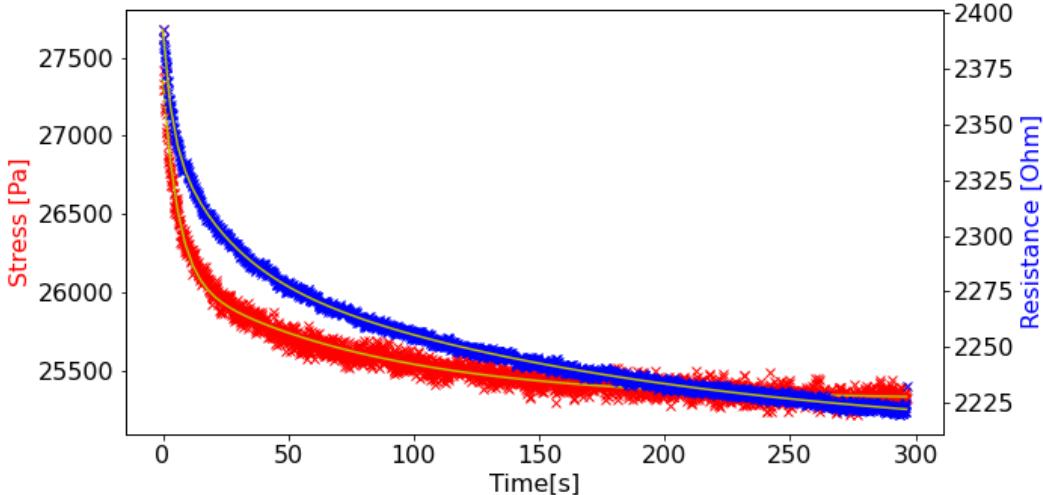


FIGURE 3.17: Comparing the relaxation decay time constants of stress and resistance for a 7.5 w.t.% CBSR composite after a 10% strain step input and fitting generalised maxwell body models to each.

The mean magnitude and decay time constants for the resistance and stress relaxations using 30 relaxation periods to fit the models to are given in table 3.5.

TABLE 3.3: Fitted constants and their mean, μ , standard deviation values for 0%, 7.5%, and 10% CBSR composite specimens using Equation 3.7.

Stress Model Parameters

| 0 % CB Specimen | |
|-----------------------|-----------------|
| Parameter | μ |
| a_0 | 20344 ± 42 |
| a_1 | 387 ± 60 |
| a_2 | 527 ± 58 |
| τ_{S1} | 72.1 ± 23.5 |
| τ_{S2} | 5.8 ± 1.5 |
| 7.5 w.t.% CB Specimen | |
| Parameter | μ |
| a_0 | 25363 ± 33 |
| a_1 | 802 ± 43 |
| a_2 | 1242 ± 52 |
| τ_{S1} | 71.0 ± 9.5 |
| τ_{S2} | 5.8 ± 0.7 |
| 10 w.t.% CB Specimen | |
| Parameter | μ |
| a_0 | 32303 ± 165 |
| a_1 | 1071 ± 54 |
| a_2 | 1649 ± 47 |
| τ_{S1} | 84.1 ± 10.6 |
| τ_{S2} | 6.5 ± 0.7 |

TABLE 3.4: Fitted constants and their mean, μ , and standard deviation values for 0%, 7.5%, and 10% CBSR composite specimens using Equation 3.8.

| Resistance Model Parameters | |
|------------------------------------|------------------|
| 7.5 w.t.% CB Specimen | |
| Parameter | μ |
| b_0 | 2154 ± 52 |
| b_1 | 81.1 ± 5.4 |
| b_2 | 56.4 ± 3.7 |
| b_3 | 42.2 ± 3.4 |
| τ_{R1} | 181.1 ± 33.6 |
| τ_{R2} | 22.8 ± 3.8 |
| τ_{R3} | 3.5 ± 0.6 |
| 10 w.t.% CB Specimen | |
| Parameter | μ |
| b_0 | 1650 ± 97 |
| b_1 | 55.2 ± 8.9 |
| b_2 | 77.4 ± 12.2 |
| b_3 | 38.4 ± 9.5 |
| τ_{R1} | 169.6 ± 61.7 |
| τ_{R2} | 21.9 ± 9.7 |
| τ_{R3} | 3.0 ± 1.6 |

3.3.6 Resistance-Stress Relationship

To display the non-linear relationship between the stress and calculated resistance within the material they are plotted against each other over 30 sequential relaxation periods of 300s. The non-linear relationship between stress and resistance changes over time for each relaxation as shown in Figure 3.18, where the data for the first relaxation is displayed in green and the last relaxation in blue.

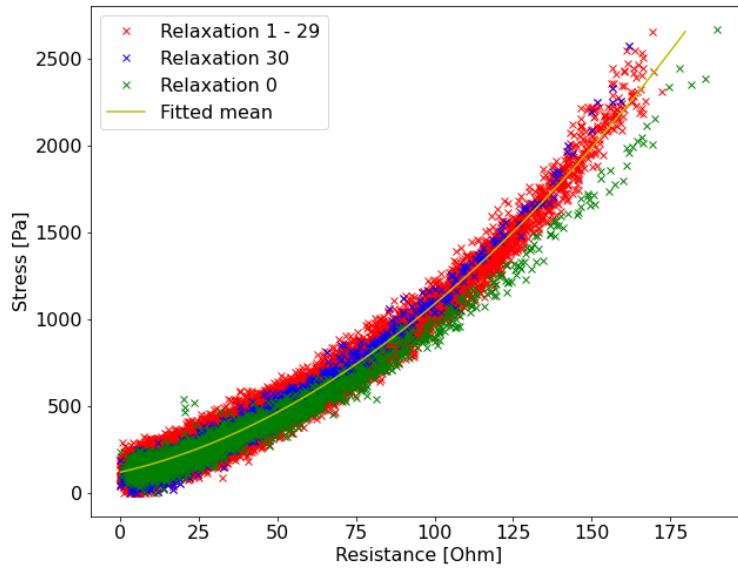


FIGURE 3.18: Comparing resistance and stress relaxation data against each other occurring during 30 pulses of a 10% strain step input for a 7.5 w.t.% CBSR composite

The stress-resistance relaxation data was fitted to a generic second order polynomial of the form,

$$\sigma(R) = aR^2 + bR + c \quad (3.9)$$

where σ is stress, R is the calculated resistance. When fit to the latter 15 cycles of a 30 cycle 10% strain pulse train of stress relaxation data we get the constant values for a , b and c .

TABLE 3.5: Fitted constants and their mean, μ , standard deviation, σ , and coefficient of variation, CV , values for 7.5%, and 10% CBSR composite specimens using Equation 3.9

| 7.5 w.t.% CB Specimen | | |
|-----------------------|-------|-------|
| Parameter | μ | |
| a | 0.055 | 0.006 |
| b | 4.15 | 1.06 |
| c | 121.8 | 16.3 |
| 10 w.t.% CB Specimen | | |
| Parameter | μ | |
| a | 0.098 | 0.007 |
| b | 6.37 | 0.76 |
| c | 155.8 | 38.8 |

3.4 Discussion

3.4.1 Fabrication

In this work, mixing has been performed using a planetary mixer. It has been shown in other works [52, 183] that other mixing methods, such as using a sonication bath and the addition of solvents, can yield better particle dispersion. A higher degree of CB particle dispersion has also been shown to alter the viscoelastic creep properties [183], and is therefore likely to affect the time constant of resistance.

Composite imaging and experimentation showed the existence of a thin insulative film on the composite surface inhibiting the use surface electrodes to give a good electrical connection to the percolated CB network. The imaging also showed the existence of voids in the micron scale which contribute towards the material viscoelasticity. The optimisation of particle dispersion and investigation into the effects of void dispersion and sizing could give insight into maximising the material gauge factor.

When characterising the reference pure SR material, the elastic modulus was larger than the 186.2 kPa elastic modulus specified by the manufacturer, likely due to the heat assisted curing process used [178].

3.4.2 Quasi-static Tensile Resistance-Strain

The quasi-static linear model given show promise for using the composite CBSR material for applications requiring a low frequency response sensor. This also gives a good steady-state part of a more complex model which includes the non-linear and time dependent dynamics.

3.4.3 Dynamic Resistance-Strain-Rate Response

Increasing strain-rates were shown to increase the offsets in resistance over a range of strains as shown in Figure 3.13. Showing the existence of a repeatable relationship between strain-rate and resistance, for constant strain rates. For strain acceleration different phenomena arose, such as the shoulder phenomena.

3.4.4 Falling Edge Strain-Rate-Resistance

A 'falling edge' resistance shoulder phenomena was observed and is clearly seen in Figures 3.12 and 3.14. This is a result of a change in strain rate. The amplitude of this peak was shown to increase with an increasing change in strain-rate from a steady state strain. The shoulder phenomena may exist on the rising edge of the strain adding to the resistive relaxation response however this was not easily distinguishable. In previous literature, the effects of the strain-rate and sudden change in strain-rate on apparent resistance of the CBSR material has not been modelled or shown.

3.4.5 Resistance Relaxation Fitting

The generalised Maxwell model has been applied to predict the stress relaxation of the CBSR composite and analogously the resistive relaxation seen, which successfully explains a significant

fraction of the resistance relaxation seen for a positive strain step input. However, a sudden peak of resistance when changing from one constant strain-rate to another is not yet explained, and consideration of temperature and strain history [14] could be used in further research to characterise a CBSR-based strain sensor for a range of application environments.

The data gathered show that the stress relaxation time constant values decrease with an increasing CB percentage, indicating that all constants in Equations 3.8 and 3.7 are also functions of CB percentage.

3.4.6 Resistance-Stress Relationship

A common hypothesis for the resistive relaxation phenomena is that it may be correlated to the resistance relaxation. This work found that the relationship between the two variables, resistance and stress, during relaxation events was non-linear and time-dependent. This non-linearity was found to have a good fit to a second order polynomial as shown in Figure 3.18. The Mullin's effect was evident in the results and a time dependence where the concavity of the polynomial increased a small amount per pulse.

3.4.7 Viscoelasticity

The hysteresis loop seen in the 10 w.t.% CB sample has a larger hysteresis loop showing that there is increased viscous/damping compared with the other two specimens percentages of CB. The pure SR specimen had no discernible hysteresis from the data as shown in Figure 3.9. The difference in hysteresis and hence viscoelastic properties, across the specimens will lead to different stress relaxation properties across the three composite materials.

3.4.8 Repeatability

The resistance relaxation model must be predictable over many strain cycles for use within many high stretch strain sensor application. If the resistance relaxation changes over time this needs to be modelled. Each test sequence showed that there was a downward trend in the calculated magnitude of resistance for each pulse over time. This downward trend is hypothesised to be due to the accumulation of charge within material over time generated by current source [], and was mitigated by using an alternating polarity measurement technique. The reversible current source helped to mitigate the capacitive effects seen, but a general downward trend in resistance was still observed as shown in Figure 3.19.

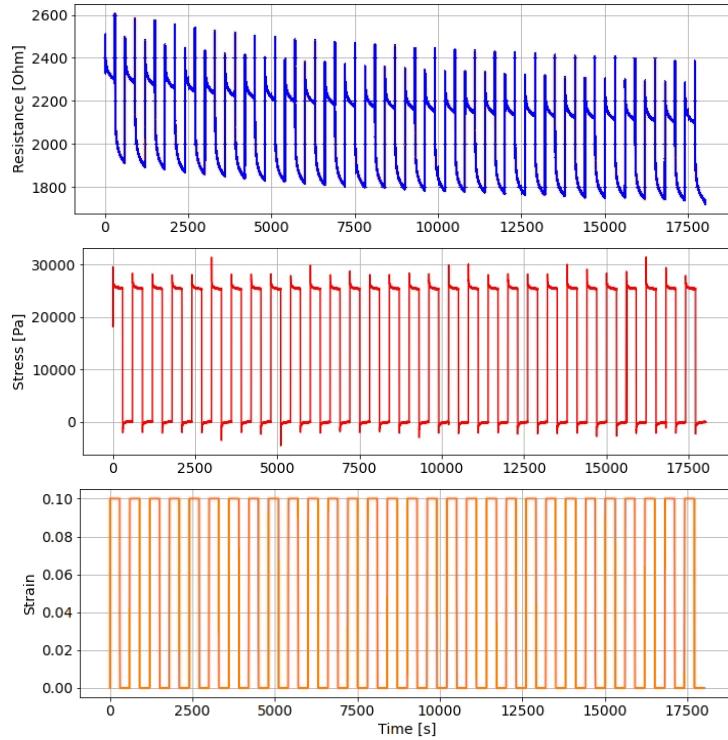


FIGURE 3.19: A typical test sequence of a 30 pulse strain train recording the calculated resistance and stress of a 7.5 w.t.% CBSR composite

For every sufficiently long test sequence the material reaches a steady state, after a finite amount of time. The capacitance read across the inner pin electrodes of the material decreased with increasing strain as shown in Table 3.6.

TABLE 3.6: Average inner electrode capacitances, C_{in} , measured for various strain, ε , values using a 7.5 w.t.% CBSR composite, measured using an LCR meter at 1kHz and 10kHz

| $\varepsilon[\%]$ | 0 | 10 | 20 | 30 |
|-------------------|----|----|----|----|
| $C_{in}[pF]$ | 53 | 32 | 24 | 20 |

A sufficiently high current source value was required to ensure a SNR that would not hinder data analysis. There seemed to be a wetting current threshold where the SNR dropped as shown by the 20 μ A resistance pulse response in Figure 3.20.

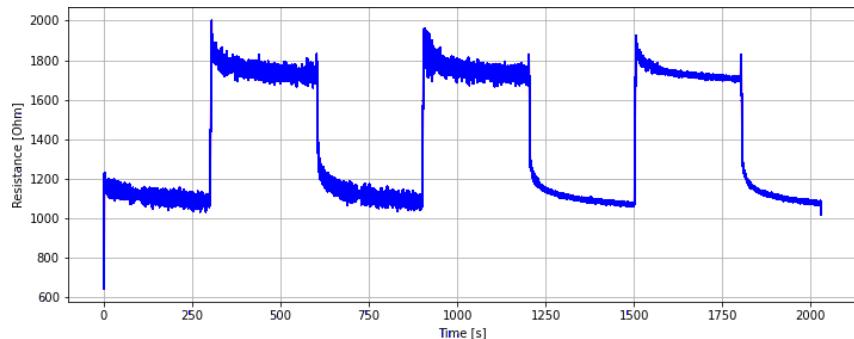


FIGURE 3.20: Three identical 15% strain pulses at three different current values ,in order, 10 μ A, 15 μ A, and 20 μ A.

A limitation of using this material as a strain sensor is the non-linearity of the material above a certain strain value, at which the composite's resistivity diverges towards a highly insulative value within the giga-ohms range. This non-linear behaviour of CBSR can be used as a mechanically activated switching device [158]. If modelled, this non-linearity could extend the range of strains that can be measured.

One aim of this work was to prove the hypothesis that the stress relaxation time constant is different to that of the observed resistance relaxation and able to be modelled mathematically. The apparent difference in time constants and the fitting of the data to two different equations show that the stress relaxation is not linearly related to the resistance relaxation shown clearly in Figure 3.17.

3.5 Conclusions

The relationships discussed in this work could be used to define a CBSR-based strain sensor models and show the expected behaviours in the composite material for different strain application scenarios.

In order to improve the accuracy of dynamic strain measurements with CBSR composites a stress and analogous resistance relaxation model was formed. The generalised Maxwell model, Equation 3.7 was used to fit to the stress relaxation data for three specimen with CB weight percentages of 0, 7.5% and 10%. The CV of the stress relaxation magnitude constants $a_0 - a_2$ were found to be consistently smaller than the CV of the stress relaxation decay time constants τ_{S1} and τ_{S2} , with maximum CV values of 15.45% and 32.54% respectively. All of the stress relaxation model constants increased with increasing weight percentage of CB.

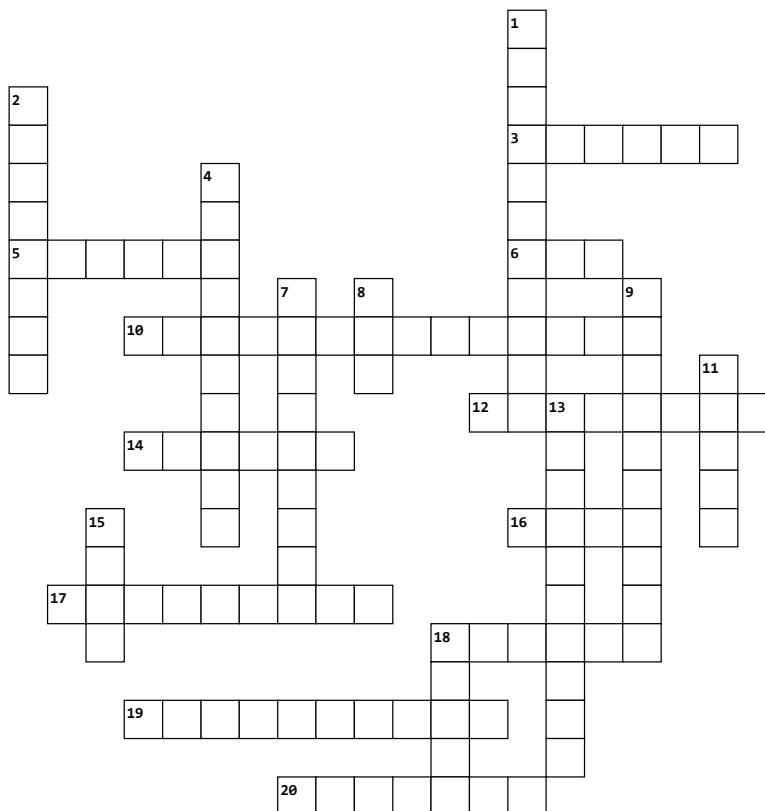
After modelling the stress relaxation, an analogous resistance relaxation model, Equation 3.8 was formed and fitted to, displaying similar attributes to the stress relaxation model fit with all of the model constants increasing with increased w.t.% CB. The CV of the analogous resistance relaxation magnitude constants $b_0 - b_3$ were found to be consistently smaller than the CV of the stress relaxation decay time constants $\tau_{R1} - \tau_{R3}$, with maximum CV values of 16.04% and 44.21% respectively.

A model relating the resistance and stress relaxation has been developed using a second order polynomial with all of the constants a , b , and c increasing with increased weight percentage of carbon black. With the models developed we have shown that the apparent resistance relaxation can be modelled, which will enable more accurate estimation of dynamic strain when these materials are applied as sensors.

Appendix A

Thesis Crossword

Tired of Marking? CROSSWORD



Across

- 3. Motivating emotion of a PhD student
- 5. This thesis is provided almost free of ...
- 6. Time constant symbol
- 10. Touch and change it's electrical properties
- 12. Motor-like
- 14. Soot
- 16. Texture
- 17. Curvy. Difficult to solve.
- 18. Epsilon is for permittivity and ...
- 19. You often ... while wearing a lab coat
- 20. Scottish Physicist who loves waves.

Down

- 1. Linearised model that mitigates time
- 2. A great elastomer
- 4. Meat of a capacitor sandwich
- 7. Mixed material
- 8. Want to solve a mesh? Use ...
- 9. Viscoelastic phenomena and massage type
- 11. A long book
- 13. Image slice
- 15. A couch potato would ... out
- 18. Some people have a sixth one

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