Soft Electroactive Elastomer Bodies that Can Sense and Contract

by

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in

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Declaration of Authorship

I, AUTHOR NAME, declare that this thesis titled, 'THESIS TITLE' and the work presented in it are my own. I confirm that:

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Signed:

Date:

"When do you think you can submit your thesis?"

T. Giffney, April 2024

"Today."

R. Ellingham, August 2024

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Abstract

College of Engineering

Mechanical Engineering

Doctor of Philosophy

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The Thesis Abstract is written here (and usually kept to just this page). The page is kept centered vertically so can expand into the blank space above the title too...

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- Family - for being supportive.
- German people for giving me a home to type out words.

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Abbreviations

EIT	**E**lectrical **I**mpedance **T**omography
DEA	**D**ielectric **E**lastomer **A**ctuator
LAH	**L**ist **A**bbreviations **H**ere
LAH	**L**ist **A**bbreviations **H**ere
LAH	**L**ist **A**bbreviations **H**ere
LAH	**L**ist **A**bbreviations **H**ere

Physical Constants

Speed of Light $c = 2.997\ 924\ 58 \times 10^8\ \mathrm{ms}^{-\mathrm{S}}$ (exact)

Symbols

R	resistance	Ω
F	force	N
add	Roman symbols	here...
σ	stress	kPa
ε	strain	%
ρ	conductance	S
add	Greek symbols	here...

Dedicated to baked beans in all their glory...

Chapter 1

Introduction

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 tools for creating and characterising artificial pressure sensitive skin technology and then continues to explore the integration of this artificial skin technology into an artificial muscle technology.

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 in technology in various soft robotic actuators[**?** **?** **?** **?** **?**], sensors[**?**], and soft robotics control[**?** **?**]. 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 biodegrabale or recyclable materials, shelf life, and use of renewable resources[**?**]. 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[**?** **?** **?** **?**].

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)[**? ? ? ?**] and ionic polymer-metal composites (IPMCs)[**?**]. 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 of artificial muscle technology has been made a reality. This is seen in the self-sensing of DEAs in one dimension usually through capacitive measurement between the compliant electrodes during operations to obtain the magnitude of a contraction. However, the pressure mapping done by cutaneous mechanoreceptors on an artificial device has not been thoroughly explored as of the writing of this thesis.

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 a dielectric elastomer actuator. The composite type used throughout the thesis is simple 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 sensor characteristic. 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 transient phenomena seen in conductive particle composites.

2. Use additive manufacturing methods and design a mixer for FDM printing of conductive particle composites for soft sensors and actuators.
3. From the characterisation in objective 1 mitigate the effects of the transient phenomena.
4. Create a set of metrics for quantifying the performance of an electrical impedance tomography based artificial skin.
5. Integrate an electrical impedance tomography based artificial skin onto a dielectric elastomer actuator.

1.3 Chapter Contributions

Chapters 3 - 7 contain the core novel research contributions. Chapters 2 and 8 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 Material 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 - A Novel Mixing Method for 3D Printing Conductive Particle Elastomer Composites: This chapter discusses the place for advanced manufacturing for 3D printing for soft actuator and sensor technology and a new form of mixing for such manufacture.

Chapter 5 - 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 6 - 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 7 - Hardware for a DEA-EIT Sensor Actuator Hybrid Device:: 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: 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

To replace and supersede tasks that can currently only be performed by humans due to their dexterity, physical makeup, and intelligence; the skin and muscles completing these tasks must first be understood and quantified. Subsequently a review of various electrically driven artificial skin and muscle technologies was completed. Finally, background theory on two specific technologies soft sensing and actuating devices is given to setup a 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 the pressure-sensitive functions of skin. Two pressure-sensitive categories of skin and muscle tissue which allow for dexterous manipulation of objects are:

1. Proprioceptors: respond to 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. Cutaneous mechanoreceptors include Meissner's corpuscles, Merkel disks, Ruffini endings, and free nerve endings.

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.

The function of both kinds of receptors have been mimicked by certain device technologies. For example proprioceptors have been mimicked in wearables where joint motion has been estimated by the stretch of sensors placed over the joints to calculate pose estimates of different body parts. Examples of such devices are displayed in Figure 2.2

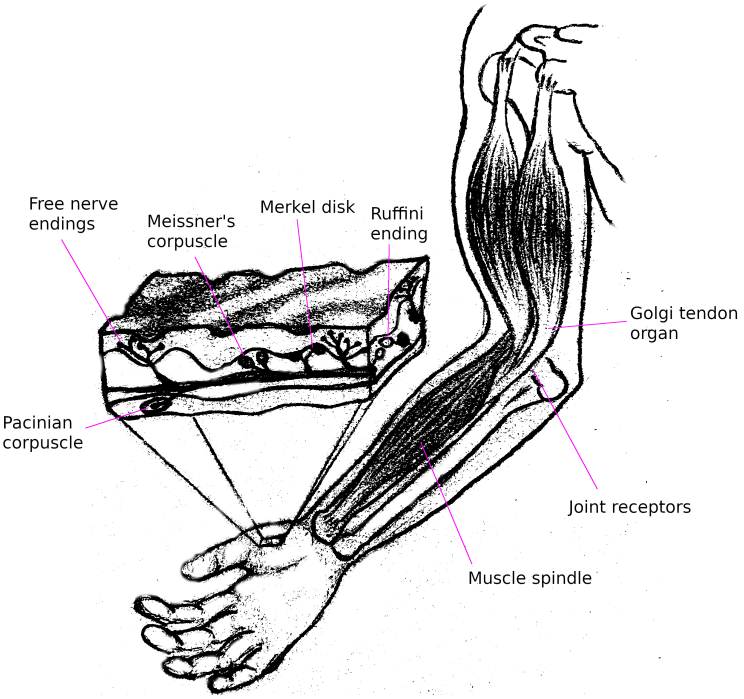


FIGURE 2.1: Examples of the locations of proprioceptors and cutaneous mechanoreceptors in the human body.

ADD -> picture of stretchsense glove and other wearables etc.pn

FIGURE 2.2: Caption

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. Examples of these sensors are shown in Figure 2.3.

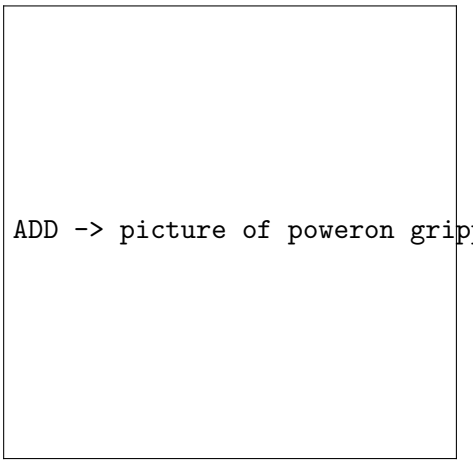


FIGURE 2.3: Caption

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

2.1.1 Skin Construction and Types

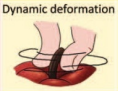
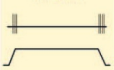
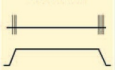
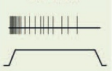
Skin is a laminate structure consisting of three main layers, the epidermis, dermis, and hypodermis. The top two layers the epidermis and dermis are a subset of the cutaneous layer which contain the majority of the pressure-sensitive mechanoreceptors [].

The skin contains can be categorised as glabrous/hairless or non-glabrous/hairy. Glabrous skin contains 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 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. 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.

Cutaneous mechanoreceptors and their functions are given in Table ,

TABLE 2.1: Comparison of typical mammalian mechanoreceptors characteristics [**?**].

	A - Glabrous skin			B- Both	
Receptor	**Meissner corpuscle** *A1*	**Ruffini corpuscle** *A2*	**Pacinian corpuscle** *A3*	**Merkel cell-neurite complex** *B1*	**Free nerve ending** *B2*
Perceptual sensory functions	Skin movement, handing objects	Skin stretch, movement direction, hand shape and finger position	Vibration when grasping an object	Fine tactile discrimination, form and texture perception	Pain, nociception
Skin stimulus	Dynamic deformation 	Skin stretch 	Vibration 	Indentation depth 	Injurious forces 
Localization	Dermal papillae	Dermis	Deeper dermis	Basal layer of the epidermis / around guard hair	Epidermis > Dermis
Afferent response / Stimulus	RAI-LTMR 	SAII-LTMR 	RAII-LTMR 	SAI-LTMR 	SA-HTMR 
Associated fibre	Aβ	Aβ	Aβ	Aβ	Aδ C-HTMR
Conduction velocity	35-70 m/s	35-70 m/s	35-70 m/s	35-70 m/s	5-30 m/s 0.5-2 m/s
Receptive field	22 mm^2	60 mm^2	finger, hand	9 mm^2	1-3 mm^2
Receptor / Hair density	150 / cm^2	10 / cm^2	20 / cm^2	100 / cm^2	4-25 / mm
Putative MS ion channels	β-ENaC/γ-ENaC/ ASIC2/ASIC3/TRPV4/ KCNQ4/Piezo 2	β-ENaC/ASIC3/ Aquaporine 1/ Piezo 2	β-ENaC/γ-ENaC/ ASIC1/ASIC2/ Piezo 2	ASIC2/ASIC3/ TRPV4/Piezo 2	TRPA1/TRPV1/ TRPV2/TRPV4/ Piezo 2

2.1.2 Characterising skin

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

1. Young's 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. Shear modulus - The relationship between the shear stress and shear strain in the linear region of the stress-strain characteristic curve [Pa]
4. Ultimate tensile stress (UTS) - The maximum tensile stress that a material can tolerate before breaking [Pa]
5. 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
6. Strain creep - All viscoelastic materials will experience strain creep to varying degrees depending on the viscoelastic properties of the material [$mm.s^{-1}$]
7. Stress relaxation - All viscoelastic materials will experience stress relaxation to varying degrees depending on the viscoelastic properties of the material [s]

8. Skin thicknesses - the thickness of all layers of skin the cutaneous epidermis and dermis and thickness of the hypodermis [mm]

9. Skin surface area - Biological skin has a large surface area and can also be regionalised to map skin function and sensitivity [m^2]

10. Isotropy/Anisotropy - The directionality of skin properties, also known as skin tension lines, give a topological map of the maximal stretch (i.e. minimal bulk 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 [**?** **?** **?** **?**]

2. Static force resolution - This is the detection resolution of static or slow-acting forces acting upon the skin [**?**]

3. Temporal resolution - This is the detection resolution of fast-acting forces acting upon the skin often required for texture recognition [**?** **?** **?**]

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

1. Young's modulus - varies largely depending on test method, test skin type, and subject. Values include 83.3 ± 34.9 MPa [**?**], 0.1 - 2.4 MPa [**?**], 10.4 - 89.4 kPa [**?**].

2. Storage and loss modulus - 141.9 ± 34.8 Pa and 473.9 ± 42.5 Pa at 0.8 Hz [**?**]. 473.9 ± 42.5 Pa and 32.3 ± 10.0 Pa at 205 Hz [**?**].

3. Shear modulus - Shear modulus has been reported to be 100 times that of elastic modulus for upper most layers of skin (epidermis and stratum corneum) [**?**]

4. Ultimate tensile stress - 21.6 ± 8.4 MPa [**?**]. 28.0 ± 5.7 MPa [**?**]

5. Life cycle - N/A. Complex to quantify for skin as it constantly regenerates over time

6. Strain creep - The strain creep was found to be 2.7 kPa.s for a 10 Pa step input on a dermis skin sample [**?**].

7. Stress relaxation -

8. Skin thicknesses - The thickness of human cutaneous skin ranges from 0.6 to 2.6 mm with an average skin thickness of 2 mm [**?**].

9. Skin surface area - The average surface area of skin in adult humans is 1.7 ± 0.1 m^2 [**?**].

10. Isotropy/Anisotropy - The tension lines in skin are determined by collagen fibre orientation and dynamic stretch events [**?** **?**]. 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 [**?**]. 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 [**?**].

    it has been shown that the receptive field area of SA1 and RA1 fibres increase linearly as the indentation depth increases with estimated minimum area of 5 mm2 for both and median areas of 11 mm2 for SA1 and 12.6 mm2 for RA1

11. Spatial resolution and touch acuity - The tactile field area increases with indentation depth for certain mechanoreceptors with a range of 5 - 12.6 mm^2 [**?**]. Two point discrimination is another metric for determining spatial resolution an has been determined as 3.7 ± 0.7 mm [**?**]. 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 [**?**].

12. Force resolution - Minimum force detection on various regions of human skin was found to be between 67 - 1007 mg [**?**]

13. Temporal resolution - Depending on the mechanoreceptor sensing the force input, a frequencies ranges of 0 to 800 Hz can be perceived by human skin [**?**]

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. constantly regenerates
3. made from various types of cells in a laminate structure

To solve the complexity of modelling such a material a review by Landry et al.[**?**] 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 behaviours observed.

2.2 Pressure Mapping Artificial Skin Devices

A range of devices that can emulate the pressure sensitive function of biological skin have been created for a range of purposes. This section will be outlining some of the main technologies which are flexible and/or soft and can map force events throughout a two dimensional 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 already available. Electroactive polymers are essentially polymer materials can be used as tranducers which change electrical properties based on a mechanical input, vice versa.

2.2.1 Pressure mapping devices

Pressure mapping devices can be categorised into their various sensing technology, such as resistive, capacitive, inductive, magnetic, optical, and acoustic. Examples have been gathered by [] showing the limits and trade-offs between each sensing technology.

2.3 Biological Muscle form and function

Note: This section was taken from an initial literature reviews from 3 years ago. Please re-review and update citations for latest 2021 - 2024 papers.

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 human-made bio-mimetic equivalent attempts. However, it must be noted that using bio-mimetic principles is not always the best method to design an actuator. Nature attempts to optimise structures through an iterative process called natural selection, but this optimisation is often only 'just good enough' for given environment[**?**]. Further optimisation of biological structures is often possible.

The concept of artificial muscles has brought much interest to the bio-medical industry, because of the potential applications with prosthesis, orthotics, and other medical assistive devices and instrumentation[**? ?**]. Applications of artificial muscles are not limited to the medical industry, but also many other fields, often where a compact, micron-scale actuator is required[**?**] or a device that mimics a biological actuator is desired.

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 form the central nervous system to a motor neuron/s which travel to the muscle where electrochemical reactions take place for the contraction to take place.

The sliding motion of the myosin and actin filaments is due myosin heads attaching 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 2.4.

The anatomy of a human skeletal muscle can be seen in Figure 2.5. The muscle is made up of bundles of fasicles connected together with a tissue called perimysium. Within the fasicles are many muscle fibres (i.e. muscle cells) which are surrounded by a connective tissue called endomysium. Endomysium is responsible for filling gaps in between muscle fibres as well as containing nerve axons and blood capillaries. Within the muscle fibres there are many sacromeres stacked within a cyclindrical-like structure called a myofibril.

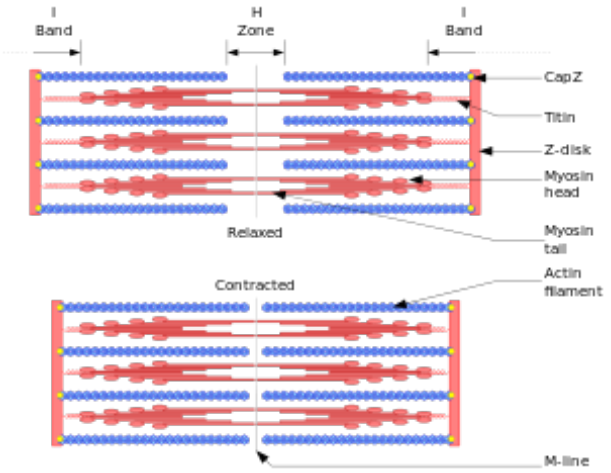


FIGURE 2.4: Diagram showing the components of a biological muscle contractile unit [**?**]

Each sacromere contains a contractile unit of myofilaments. There are tranverse tubules (t-tubules) travelling diametrically across the muscle fibre to maximise the reach of the muscle excitation signal from the nerve axon to as many myofibrils as possible.

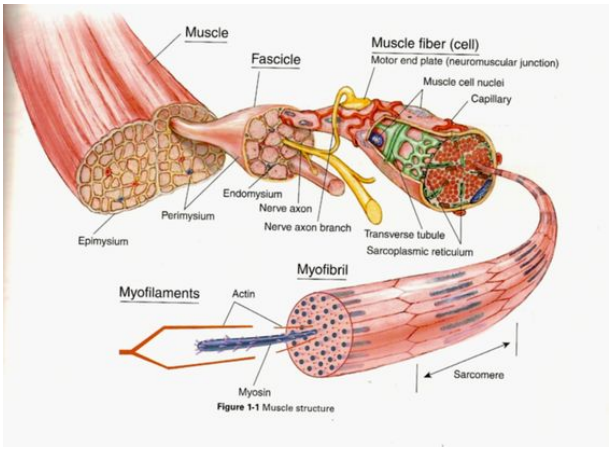


FIGURE 2.5: Diagram of the internal structures of a skeletal muscle[**?**]

2.3.1 Characterising a muscle

To be able to mimic a biological muscle there must be certain metrics characterising muscles such that both artificial and biological that can be compared. An artificial muscle can be characterised using typical mechanical material parameters such as:

1. Young's modulus - The elasticity determining the relationship between stress and strain for the linear region of the stress strain characteristic curve. $[Pa]$
2. Shear stress - Force applied parallel with a cross sectional area plane due to a state of muscle excitation. $[N.m^{-2} or Pa]$
3. Shear strain - The change in deformation perpendicular to the direction of loading to the due to a state of muscle excitation.

4. Shear modulus - The relationship between the shear stress and shear strain in the linear region of the stress strain characteristic curve. $[Pa]$
5. Energy density - The work done by the muscle per unit volume or mass. $[J.kg^{-1}]$
6. Power density - The work done by the muscle per unit volume (or mass) per unit time. $[J.kg^{-1}s^{-1} or W.kg^{-1}]$
7. Yield stress - Stress at which the stress strain curve of the muscle begins to become non-linear and the material strain may not return to it's resting (original) length. $[Pa]$
8. Ultimate tensile strength - The maximum tensile stress that a material can tolerate before breaking. $[Pa]$
9. Efficiency - The work done by the muscle compared to the energy put into the system, known as metabolic cost in biological muscles. [%]
10. Actuation frequency - The frequency range of actuation cycles using the system's method of excitation. $[Hz]$
11. Stroke - The maximum displacement an actuator can achieve $[m]$
12. Drift - Change in actuation displacement over time given the same excitation input value each actuation cycle. $[m]$
13. 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 commonly used medical/biology muscle metrics such as:

14. 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[**?**].
15. Muscle force direction and architecture - Biological muscles can have varying contraction force directions determined by pennation angle of the muscle and the muscle fibre configuration.
16. Fatigue - In a mechanical sense this is defined as the weakening of a material due to cyclic loading. In biological terms muscle fatigue defines a state where muscles are not performing in an optimal manner often due to cyclic contractions. The cause of biological muscle fatigue is caused by a lack of substances required during the muscle contraction process, or an imbalance of the substances required during the muscle contraction process[**? ?**].
17. Maximum contraction velocity/muscle bandwidth - Contraction velocity is the maximum velocity at which a muscle can contract due to an excitation. Which is important for artificial muscles where the and bandwidth of the muscle represents the range of contraction frequencies the muscle can operate.

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 this could be another parameter considered for some artificial muscles.

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

- Energy density - Biological muscle can have energy densities ranging from 0.4 - 40 $J.kg^{-1}$[**?**].
- Power density - Biological muscle can have energy densities ranging from 9 - 284$W.kg^{-1}$[**?**]
- Actuation frequency - The range of natural actuation frequencies for both vertebrate and invertebrate muscles ranges 1 - 180Hz[**?**].
- Strain - Biological muscle can have strains ranging from 5-30%[**?**].
- Efficiency - Thermodynamic efficiency of human muscle is typically between 20-35%[**?**]. However other biological muscle has been seen to reach efficiencies of up to 77%, such as in tortoises[**?**].

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;

$$\frac{d\sigma}{d\varepsilon} = \alpha.(\sigma + \beta) \tag{2.1}$$

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

$$\sigma = \mu e^{\alpha\varepsilon} - \beta \tag{2.2}$$

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.

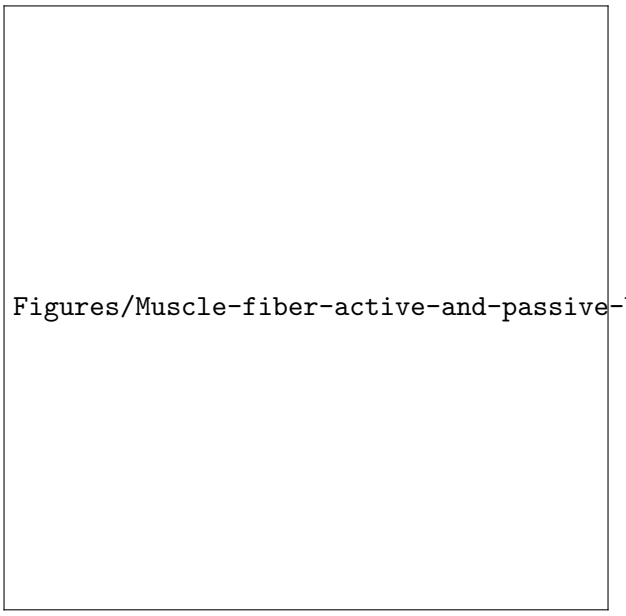


FIGURE 2.6: Plot showing the stress and strain of active and passive muscles [**?**]

Hill's muscle models commonly refer to the equation for Tetanic muscle contraction and a mechanical 3 element model published in the works of physiologist Archibald Hill [**?**]. The equation for Tetanic muscle for skeletal muscle is:

$$(v + b)(F + a) = (a.v_0/F_0).(F_0 + a) \tag{2.3}$$

Where

- F : tension in the muscle
- F_0 : maximum isometric tension generated in the muscle
- v : contraction velocity
- v_0 : maximum velocity (when F = 0)
- a : coefficient of shortening heat

F and v have a hyperbolic relationship, meaning that higher muscle loading will cause lower contraction velocity. The higher the contraction velocity the lower the tension in the muscle. This is thought to be caused by two factors. The first is the loss in tension as the contractile unit cross-bridges between the actin and myosin then reforms in a shortened condition. The second, but lesser cause of a decrease in tension during increased contraction velocity is the contractile element and connective tissue acting as a fluid damper due to their fluid viscosity[**?**].

The three element muscle model involve three main components. One parallel non-linear spring spring, one series non-linear spring element, and a contractile unit, displayed as mechanical free body diagram in figure 11. Where F^T is the tendon force; F^M is the muscle force; the l^T, l^M, l^{MT} are muscle length, tendon length and their combined lengths respectively; α is the pennation angle (i.e. zero if parallel muscle); The left and right non-linear spring elements represent a tendon and muscle spring characteristic

respectively; The 'CE' box represents the contractile element, which generates contractile force. There are many other variations of this model involving damping within the CE

FIGURE 2.7: Diagram of a Hill muscle model[**?**]

and/or damping parallel with the length of the muscle[**?**]. However to keep it simple we will just consider the model basic model shown. From this model we can obtain an equation of force elements;

$$F^T = F^{KT} + (F^{CE} + F^{KM})cos(\alpha) \tag{2.4}$$

Where F^{KT} and F^{KM} are the spring forces of the tendon and muscle respectively, which are a function of extension length. F^CE is the contractile force and F^T is the total contractile force as observed at the end of each tendon either end of the muscle.

2.3.3 Electrical Muscle Models

There are two major instruments for the actuating the muscle artificially and sensing muscle activity. These are FES (Functional Electrical Stimulation) and EMG (Electromyography). FES involves providing an artificial electrical signal to a muscle, essentially attempting to simulate the signal a motor neuron would give to a muscle. 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 muscle activation, often resulting in increased muscle fatigue when compared to equivalent voluntary muscle contractions [**?**]. 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[**?**]). EMG also commonly uses two electrodes on the surface of the skin above a desired muscle. EMG senses the nerve impulses sent to the muscle and propagated through action potential. The electrical models within the muscle actuation process can be broken down into small sub groups such as neuron action potential modelling, saltatory conduction modelling, muscle cell impedance modelling. These models become very complex due to the non

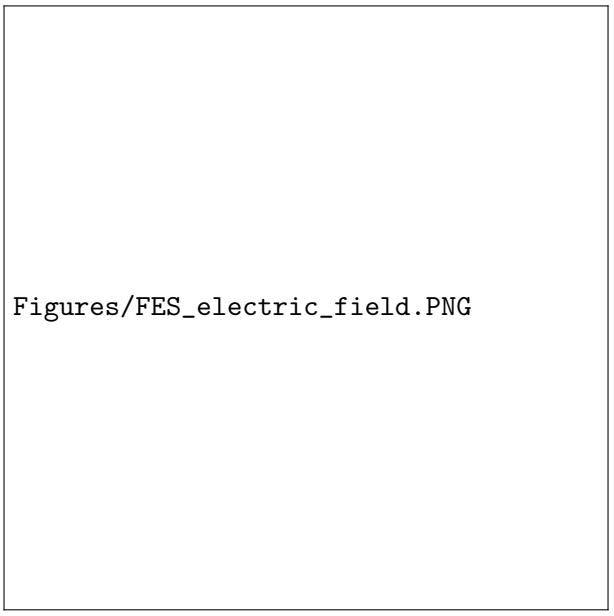


FIGURE 2.8: Diagram an electric field generated by two electrodes on the surface of the skin above a specific muscle and hence it's activating nerve bundle[**?**]

idealistic nature of all of the biological components involved, so it's a lot simpler and more practical to use the electro-chemical models of the system.

2.3.4 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, magnetorheolgical 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-the-art soft electromagnetically driven actuator technologies.

2.3.4.1 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 being less 10V. IPMCs are also desirable as artificial muscles as they can display large bending deformations, simple to fabricate, light weight and thin in design, and can have a fast actuation response time (¿15Hz) at small displacements[**?**]. IPMCs also have a high work density and maintain a constant volume during actuation like biological muscles. 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

FIGURE 2.9: Diagram of the typical architecture of an IPMC actuator[**?**]

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[**?**]. This swelling adjacent to the cathode provides the device's bending actuation.

There are many variations of the design and manufacturing of IPMCs to optimise the actuator for an application as shown by [**?**].Although the process of manufacturing IPMCs is simple, it takes a long amount of time (often can be over 48hours[**?**]) 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 [**?** **?** **?**]. The use of additive manufacturing has been used successfully to generate more complex geometries using fused filament deposition[**?**].

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[**?**].

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 [**?** **?** **?**]. 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 [**?**], small biomimetic robotics [**?** **?**], aquatic robotics[**?** **?**], with many other applications yet to be discovered.

2.3.4.2 HASEL actuator

A hydraulically amplified self-healing electrostatic (HASEL) actuator is a recent soft actuator technology developed in 2018[**?**] 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[**?**]. 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[**? ?**]. 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[**?**]. Recorded efficiency values of HASEL actuators of 21% are

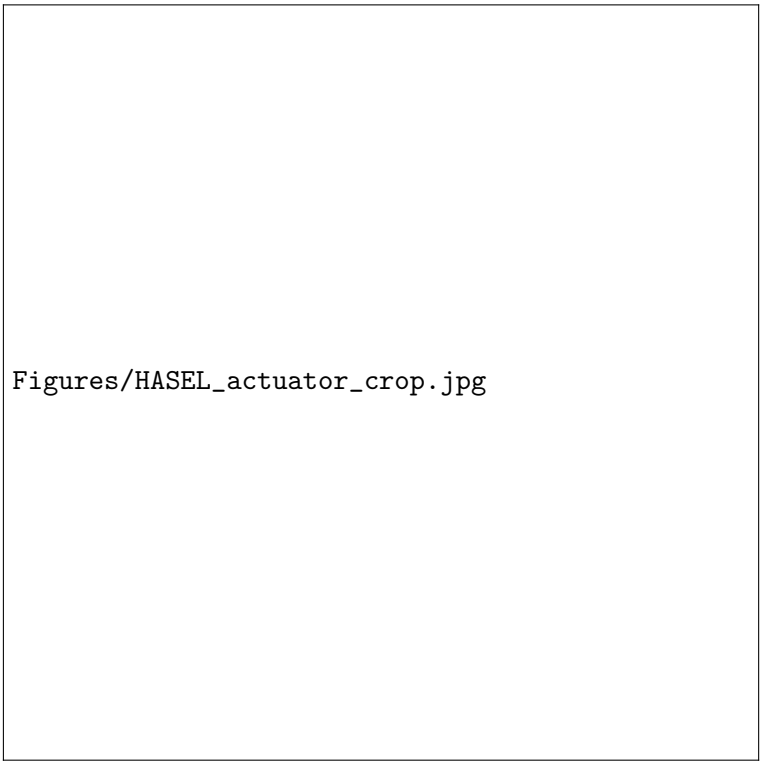


FIGURE 2.10: Diagram of the typical architecture and the contraction stages of a HASEL actuator[**?**]

comparable to that of human muscles of 20 - 35% [**?**]. 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[**?**]. 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:

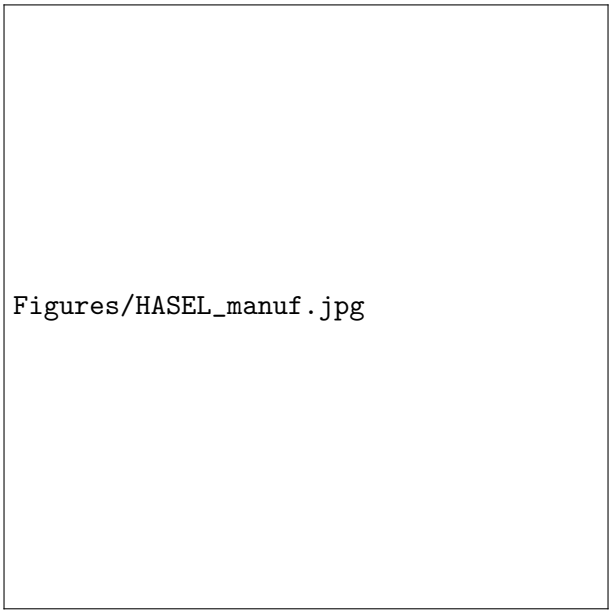


FIGURE 2.11: Diagram of the simplified stages of HASEL actuator production[**?**]

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[**?**].

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 a 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[**?**].

The number topologies possible with HASEL actuators is limitless. Some topologies of HASEL actuators include torus, planar linear[**?**], scorpion metasoma[**?**].

2.3.4.3 Magnetorheological Elastomer

Magnetorheological elastomer (MRE) actuators are a relatively new form of actuator however the theory reinforcing operating principle has been known since at least the 1980s [**?**]. 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 The operating principle of these are that magnetic

flux travelling through the 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).

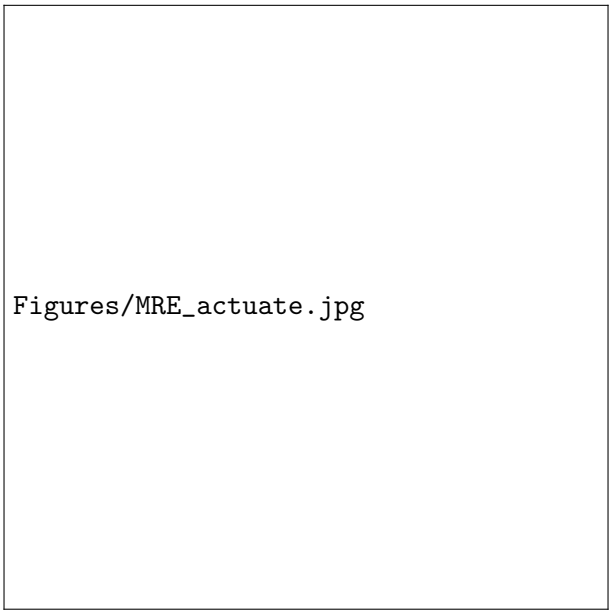


FIGURE 2.12: Diagram showing MRE contraction actuation when a magnetic field is applied[**?**]

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[**?**].

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 fixed between the crosslinked polymers. A key issue when creating an MRE is the conglomeration 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. During the curing process a magnetic field can be applied to align the particles within the elastomer as it becomes more rigid.

There have been attempts to use additive manufacturing to make MREs[**?**], however the method described has not optimsied the structure of MRE for any application and the dipersion of MRE is not 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[**?**], medical assitive devices[**?**]

and helicopter seat damping [**?**]. Potential MRE actuator applications include fluid valve control[**?**] and active vibration control similar to that mentioned for MRFs[**?**].

2.3.4.4 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

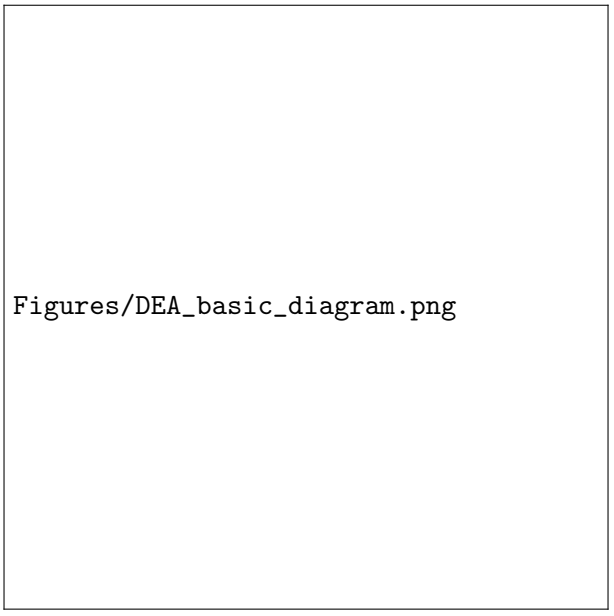


FIGURE 2.13: Diagram of a DEA with a with no voltage and a voltage applied across the electrodes. [**?**]

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

$$\sigma_{es} = \varepsilon_0 \varepsilon_r \frac{V^2}{z^2} \tag{2.5}$$

Where p_{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 predicatively 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[**?** **?** **?** **?**]. 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[**?** **?**], helical[**?**], bending[**?**], lens[**?**], cylindrical, and rolled shaped actuators[**?**]. Each of which having a range of applications.

DEAs are often fabricated in a laboratory environment using a pre-strained elastomer. The pre-straining does three key things; stores elastic strain energy, ensures DE is planar within the bounds of the jig, and controls the initial thickness of the elastomer. 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[**?** **?**].

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

Currently dielectric elastomer actuators all require voltages within the kilo-volt range to generate what can be called a useful stress and strain for many applications. A key problem encountered by researchers designing DEAs is the trade-off between actuation force and strain magnitude [**?**]. This high voltage requirement makes 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 Piezoresistive Composites

A core part of this work is understanding the behaviour of conductive particle elastomer composites as they are EAPs which can be used for a range of sensing and actuating purposes. The characteristics that make conductive particle elastomer composites (CPECs) ideal for soft sensor and actuator devices include:

1. Low stiffness
2. Changeable conductivity
3. Piezoresistivity
4. Mouldable

5. 3D printable
6. Low toxicity
7. Durable
8. Inexpensive
9. Easy to obtain
10. Simple fabrication process

2.5 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 sonication bath. The sonication bath performs a frequency sweep whereby the resonant modes of the agglomerates are met causing separation of the agglomerates into their primary particles []. The degree of dispersion is governed by the time in the sonication bath, the sonication frequencies, and sonication amplitudes []. 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 []. During the mixing process often the liquid solvent used in the dispersion stage is evaporated, leaving only the curable elastomer and the conductive particles. 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 [] or traditional moulding [] or film making techniques []. During the moulding process the material undergoes a form of curing, such as UV curing, catalysed curing, 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 [].

2.5.1 Conduction mechanism

The brief introduction of the typical fabrication process for CPECs shows that the dispersion of conductive particles will always vary. 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:

1. Conductive particle
    (a) Aspect ratio []
    (b) Inherent conductivity []
2. Conductive particle dispersion []
    (a) Inter-particle distance distribution []
    (b) Particle agglomeration distribution []
    (c) Isotropy/anisotropy []
    (d) Sedimentation []
3. Elastomer matrix []
    (a) Viscosity
    (b) Elastic modulus
    (c) Dielectric permittivity
4. Impurities []
5. Voids []

Microscale models for CPECs and the relationship between particle and electric charge motion are often computationally heavy, overly idealised, and non-invertible []. However, in future 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, which usually make many assumptions to simplify the problem [].

Electrical conduction through a CPEC occurs using two main mechanisms, Coulomb conduction and quantum tunneling [**?** **?** **?** **?**]. Coulomb conduction uses the conduction band electrons are shared by adjacent atoms allow conduction throughout chains of cascading conductive particles. 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 [**?** **?**].

Chapter 3

A Simple Conductive Elastomer Composite Material with Complex Behaviour

3.1 Introduction

As discussed in Section 2.4 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 are a simple concept of dispersing particles throughout an elastomeric matrix, the electromechanical behaviour is not well understood on a macro or micro-scale. This section 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 as a stress and/or strain sensor.

3.2 Stress and Resistance Relaxation For Carbon Nanoparticle Silicone Rubber Composite Large-Strain Sensors

From a conference paper presented at IDETC-MESA 2021

Carbon nanoparticle-silicone elastomer composites are stretchable conductive materials with diverse applications such as, highly elastic strain sensors [**?** **?** **?**], dielectric elastomer actuators [**?** **?**] and electromyography electrodes[**?** **?** **?**]. Understanding the dynamic resistance relaxation characteristics of carbon black (CB) polydimethylsiloxane (PDMS) 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, CB-PDMS composites can have strains of over 300% without yielding[**?**] depending on the type of PDMS and CB used and the method of fabrication.

Some characteristics of CB-PDMS composites which make it suitable for strain sensors include that, the material is relatively inexpensive and readily available; non-toxic and

is bio-compatible; and has a significant and readily measurable resistance change when stretched. Whereas, alternatives to CB nanoparticles, such as carbon nanotubes[**?** **?**] and metallic particles[**?** **?**], have been seen to be more carcinogenic than the CB alternative[**?** **?** **?**]. The fabrication of the CB-PDMS composite 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 carbon black particles means better repeatability of experimental results and more accurate models for the eventual applications of CB-PDMS composites. A sufficiently comprehensive model of how the resistivity changes with strain has not yet been developed. 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 CB-PDMS can be used as a mechanically activated switching device[**?**]. If modelled, this non-linearity could extend the range of strains that can be measured.

While previous work from our research group [**?** **?**] has focused on the response to quasi-static and low speed behaviour, these materials show dynamic effects where resistance depends on the speed of stretching. The characterisation investigated for the CB-PDMS sensor 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[**?** **?** **?** **?**], but never accurately modelled with the physical theory explained. The current limitations of predictability and repeatability of resistance relaxation hinders the accuracy of fitting models. An understanding of this resistance relaxation phenomena would mean an accurate model could be made to predict the relationship between stress, strain and resistance within a CB-PDMS composite. Finding this relationship model would also allow us to understand the limitations of using this composite in sensing applications and also the use of the material in dielectric elastomer actuators, whereby the material can be used simultaneously as an actuation excitation electrode and a strain sensor. The composite material can also be used in human motion measurement as a skin stretch 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.[**?**], 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 [**?**] depending on the material modelled. The research discussed will focus primarily on only tensile stress of the specimen, and how it relates to the electrical resistive relaxation.

BACKGROUND

The Composite

The CB-PDMS composite was composed of carbon black powder(Vulcan XC 72R, average particle size: 50 nm, typical bulk density: 96 kg/m^3) and two part Pt cured PDMS(Smooth-On Dragon Skin 10 NV). This grade of PDMS was chosen due to the following characteristics [**?**]:

1. Low elastic modulus, E, of 186 kPa
2. Tensile strength, σ_y of 2.75 MPa
3. Low mixed viscosity, η, of 6,000 cps

The volume resistivity of pure carbon black powder itself is between 10^{-1} and 10^{2} Ωcm depending on how densely the particles are packed and the purity of the CB[**?**]. The ability of a carbon black matrix embedded within a highly insulative PDMS substrate to become conductive is determined mainly by the dispersion of the CB particles, and the tunneling that occurs between conductive CB and insulative PDMS bodies within the material volume[**?** **?**]. 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 [**?**] 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 our composite was is difficult to predict analytically due to the unknown configurations of aggregates and agglomerations formed by the CB within the composite material. Experimentally we found that a CB volume percentage of 7.5% or greater meant the composite material had a resistivity of less than 3.5 kΩcm consistently with the fabrication method used.

The Mechanics

It is known that PDMS composites are viscoelastic materials and clearly exhibit the three traits of a viscolelastic material[**?**]: stress relaxation, strain creep and stress-strain hysteresis. Stress relaxation is 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 viscoelasticty is the generalized Maxwell body model of order n shown in Fig. 3.1.

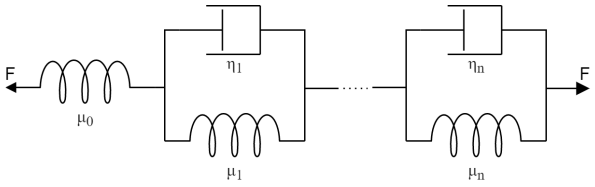


FIGURE 3.1: MECHANICAL SPRING DASHPOT DIAGRAM OF THE GENERALIZED MAXWELL BODY MODEL ADAPTED FROM FUNG ET AL.[**?**]

In Fig 3.1 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 Eqn. 3.1, for, n, serial repeating units.

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

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 generalized model can easily over-fit the data, if n is too high, due to it's generality.

MATERIALS AND METHODS

Composite Fabrication

The first step in fabrication was to mix the CB nano-powder with the silicone part A (the liquid PDMS elastomer) using a Kurabo KK-50S planetary mixer. 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 material was then mixed with the silicone part B (the liquid PDMS elastomer cross-linker) using the same planetary mixing function to ensure adequate dispersion of the CB particles throughout the PDMS volume as well as de-aeration.

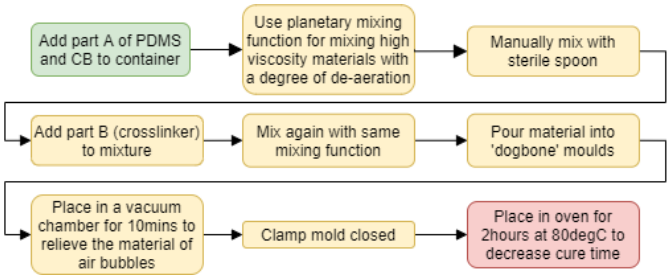


FIGURE 3.2: THE STEPS INVOLVED IN CREATING THE CB-PDMS COMPOSITE MATERIAL

For the fabrication of the CB-PDMS specimens, a standard dog-bone shaped mould was developed for the mixed CB-PDMS to cure in, based on ASTM standard D412[**?**]. 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 CD-PDMS mixture. The specimen was placed in a controlled oven at a temperature of 80 °C for a two hours to maintain the repeatability of the curing stage of the fabrication process. The temperature at which the silicone contributes towards the elastic modulus and yield strength of the material, with increasing curing temperatures giving increasing elastic moduli and decreasing yield strength values.

Measurement

A custom test measurement device was made for measuring the desired characteristics of the CB-PDMS material, so that parameters driving the data collection could be easily altered. The strain, stress and resistivity of the specimen were measured in in parallel. The setup included the use of a 500 gram loadcell (HT sensor - TAL221) in combination with a linear actuator stage driven by a NEMA23 stepper motor and an source measurement unit (Keithley 2634B SMU). A custom electrode clamp mechanism was designed to fix the electrodes onto the test specimen during the straining of the

specimen. This consists of two copper plates sandwiching the composite material at each end of the dogbone test specimen.

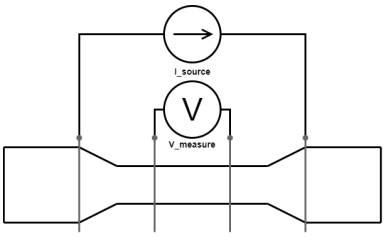


FIGURE 3.3: THE COMPOSITE DOGBONE TEST SPECIMEN PIERCED BY 4 PIN ELECTRODES. THE OUTER AND INNER ELECTRODES CONNECTED TO AN SMU CURRENT SOURCE AND VOLTMETER RESPECTIVELY

Two configurations of resistance measurement were tested, a two wire and a four wire method. The two wire measurement method used two electrodes which also clamped the test specimen at each end. It was observed that compressive strain applied to CB-PDMS composite will increase the resistivity of the specimen in a similar fashion to tensile stress. Only a compressive strain was applied to the material by the clamps such that the material would not slip during tensile testing and not deform giving erroneous resistance results. The Poisson's ratio of the material which was found experimentally to be 0.29 for both CB percentages. The two wire method used a controlled current source in parallel with a voltmeter attached to the same two electrodes to derive a resistance. The four wire method uses four pin electrodes as seen in Fig. 3.3. 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 and hence resistivity of the material. The four wire electrode configuration meant that the resistivity had a smaller signal to noise ration compared to a two wire alternative.

Metallic pin electrodes were selected over copper clamp and conductive adhesive alternatives as they deformed the material the least, had a consistently low specimen-electrode contact resistance, and did not slip during test sequences. The inner pin electrodes were symmetric about the centre and placed 20 mm apart with the outer pin electrodes being 40mm apart as shown in Fig. 3.4.

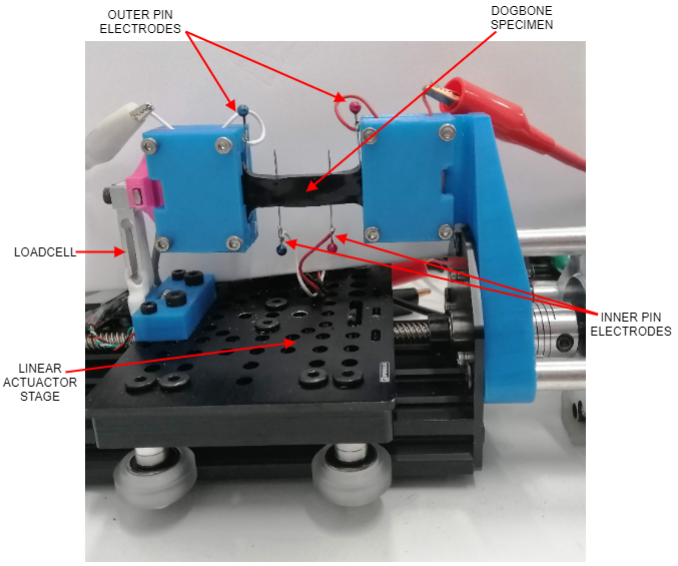


FIGURE 3.4: PHOTO OF TEST MEASUREMENT SETUP

The measurements were completed using finite 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 strains tested in this work was 10%, 20%, and 30%. This strain percentage is higher than commonly used constantan strain gauges, which typically have a maximum strain of approximately ±3%[**?**], with traditional metal alloy based strain gauges often having significant plastic deformation after less than 10^4 cycles[**?**] at 3% strain.

RESULTS AND DISCUSSION

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 Fig. 3.5. The 0, 7.5, and 10 w.t.% CB specimens have average elastic moduli, as measured in the loading phase, of 205.2 kPa[1], 321.4 kPa, and 342.1 kPa, respectively. 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 PDMS specimen had no discernible hysteresis from the data as shown in Fig. 3.5. The difference in hysteresis and hence viscolelastic properties, across the specimens will lead to different stress relaxation properties across the three composite materials.

[1]Different from the 186.2 kPa elastic modulus specified by the manufacturer due to the temperature accelerated curing method used

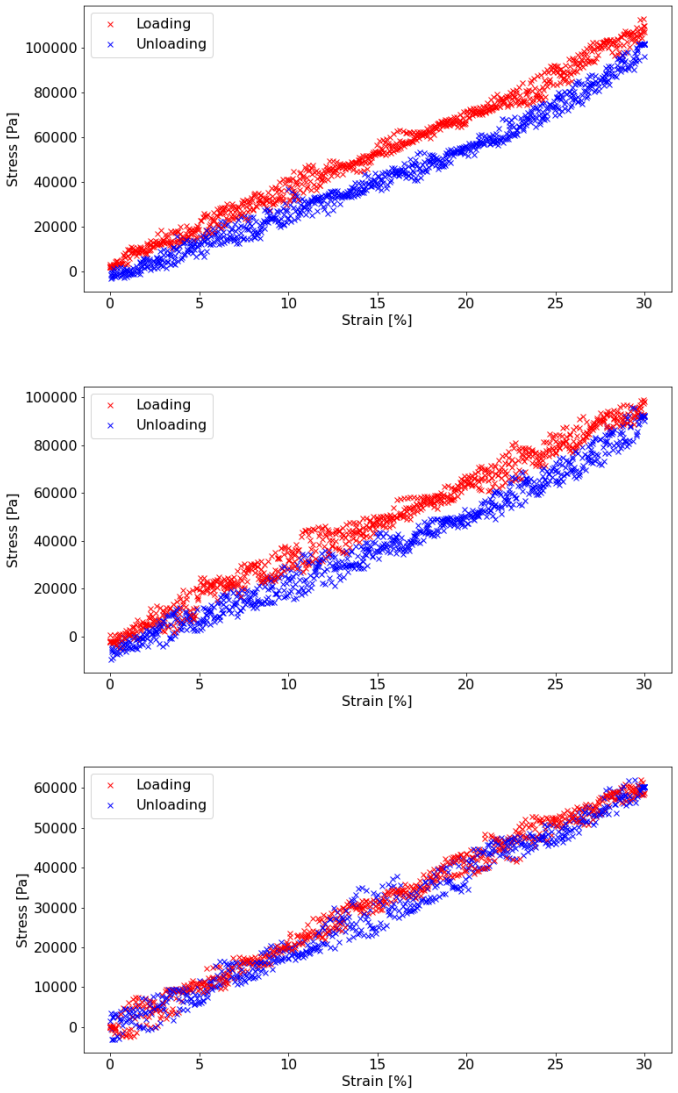


FIGURE 3.5: THE LOADING AND UNLOADING OF 30% STRAIN ON A COMPOSITE TEST SPECIMENS WITH CB WEIGHT PERCENTAGES FROM TOP TO BOTTOM OF 10%, 7.5%, AND 0% WITH DATA COLLECTED OVER FIVE LOADING AND UNLOADING CYCLES

Resistance Relaxation Model Fitting

The initial model chosen to fit the stress and resistance relaxation data was the generalized Maxwell body model shown in Fig. 3.1 with n = 3 cascading elements using Eqn. 3.2 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 constants as initial conditions for the fitting of the next stress relaxation data set, the values of the constants diverged exhibiting signs of overfitting. This divergence of the model constants meant that they had a large standard deviation showing the model was changing significantly each iteration of fitting. Hence a more simple model using Eqn. 3.1 with n = 2 was used to fit the stress relaxation data to Eqn. 3.3 with lower standard deviation of the

model constants. Conversely when the resistance relaxation model analogous to stress relaxation model, shown in Eqn. 3.4, 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 Fig. 3.6. Below in stress relaxation models $G_{1,2}(t)$, shown in Eqn. 3.2 and 3.3, the constants a_{0-3} and τ_{S1-S3} represent the components of magnitude and time decay of the stress relaxation, respectively.

$$G_1(t) = a_0 + a_1 e^{-t/\tau_{S1}} + a_2 e^{-t/\tau_{S2}} + a_3 e^{-t/\tau_{S3}} \tag{3.2}$$

$$G_2(t) = a_0 + a_1 e^{-t/\tau_{S1}} + a_2 e^{-t/\tau_{S2}} \tag{3.3}$$

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.

$$H(t) = b_0 + b_1 e^{-t/\tau_{R1}} + b_2 e^{-t/\tau_{R2}} + b_3 e^{t/\tau_{R3}} \tag{3.4}$$

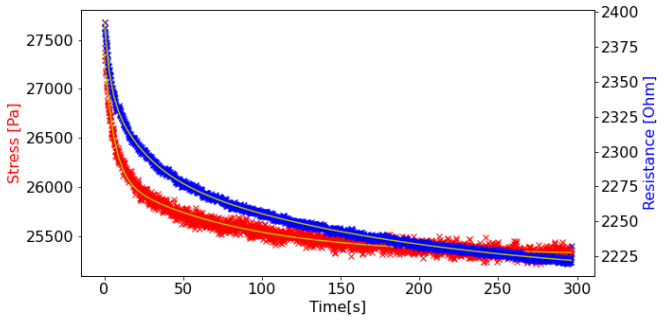


FIGURE 3.6: COMPARING THE RELAXATION DECAY TIME CONSTANTS OF STRESS AND RESISTANCE FOR A 7.5 W.T.% CB-PDMS COMPOSITE AFTER A 10% STRAIN STEP INPUT AND FITTING GENERALIZED MAXWELL BODY MODELS TO EACH.

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 3.3. The data gathered show that the stress relaxation time constant values decrease with an increasing carbon black percentage, indicating that all constants in Equations 3.4 and 3.3 are also functions of the carbon black percentage.

TABLE 3.1: FITTED CONSTANTS AND THEIR MEAN, μ, STANDARD DEVIATION, σ, AND COEFFICIENT OF VARIATION, CV, VALUES FOR 0%, 7.5%, AND 10% CB-PDMS COMPOSITE SPECIMENS USING EQUATION 3.3.

Stress Model

0 % CB Specimen Constant	μ	σ	CV
a_0	20344.71	42.61	0.20%
a_1	387.28	59.86	15.45%
a_2	526.82	57.65	10.94%
τ_{S1}	72.08	23.46	32.54%
τ_{S2}	5.77	1.48	25.75%
7.5 w.t.% CB Specimen Constant	μ	σ	CV
a_0	25363.89	33.62	0.13%
a_1	802.32	43.59	5.43%
a_2	1242.32	52.67	4.24%
τ_{S1}	71.01	9.49	13.37%
τ_{S2}	5.79	0.65	11.32%
10 w.t.% CB Specimen Constant	μ	σ	CV
a_0	32303.01	165.62	0.51%
a_1	1071.38	54.32	5.07%
a_2	1649.82	47.31	2.86%
τ_{S1}	84.07	10.55	12.54%
τ_{S2}	6.52	0.74	11.35%

TABLE 3.2: FITTED CONSTANTS AND THEIR MEAN, μ, STANDARD DEVIATION, σ, AND COEFFICIENT OF VARIATION, CV, VALUES FOR 0%, 7.5%, AND 10% CB-PDMS COMPOSITE SPECIMENS USING EQUATION 3.4.

Resistance Model

7.5 w.t.% CB Specimen Constant	μ	σ	CV
b_0	2154.31	52.68	2.44%
b_1	81.13	5.39	6.65%
b_2	56.37	3.67	6.52%
b_3	42.16	3.42	8.12%
τ_{R1}	181.10	33.57	18.54%
τ_{R2}	22.84	3.81	16.71%
τ_{R3}	3.46	0.56	16.35%
10 w.t.% CB Specimen Constant	μ	σ	CV
b_0	1649.55	97.44	5.90%
b_1	55.19	8.85	16.04%
b_2	77.39	12.23	15.80%
b_3	38.35	9.47	24.69%
τ_{R1}	169.63	61.72	36.38%
τ_{R2}	21.85	9.66	44.21%
τ_{R3}	3.02	1.59	52.72%

Our aim 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 Fig. 3.6. 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 Fig. 3.7, where the data for the first relaxation is displayed in green and the last relaxation in blue.

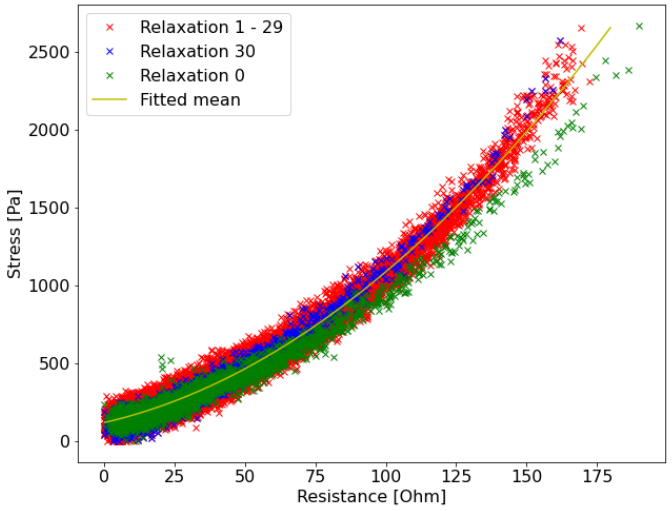


FIGURE 3.7: COMPARING RESISTANCE AND STRESS RELAXATION DATA AGAINST EACH OTHER OCCURRING DURING 30 PULSES OF A 10% STRAIN STEP INPUT FOR A 7.5 W.T.% CB-PDMS COMPOSITE

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

$$\sigma(R) = aR^2 + bR + c \tag{3.5}$$

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 3.3: FITTED CONSTANTS AND THEIR MEAN, μ, STANDARD DEVIATION, σ, AND COEFFICIENT OF VARIATION, CV, VALUES FOR 7.5%, AND 10% CB-PDMS COMPOSITE SPECIMENS USING EQUATION 3.5

7.5 w.t.% CB Specimen Constant	μ	σ	CV
a	0.055	0.006	11.1%
b	4.146	1.058	25.5%
c	121.845	16.338	13.41%
10 w.t.% CB Specimen Constant	μ	σ	CV
a	0.098	0.007	7.48%
b	6.374	0.757	11.87%
c	155.812	38.753	24.87%

Strain Velocity Resistance Relationship

A narrow peak in the apparent resistance has been observed in the collected data when changing from 10% strain to a zero strain. This peak is not present in the stress plot, hence is a proposed characteristic of electrical behaviour only as a function of strain. In previous literature, the effects of the rate of change of strain on apparent resistance of the CB-PDMS material has not been modelled or shown. When the material has finished a tensile cycle of strain and is returning a zero strain state the a component of the resistance, 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 Fig. 3.8. Hence we form an equation which relates a component of resistance,

$$\frac{dR_p}{dt} = E(\varepsilon)t + c \tag{3.6}$$

where E is a function of strain, $\varepsilon(t)$, and c is an offset bias determined by the initial strain condition. To show the strain velocity resistance relationship, more strain pulse

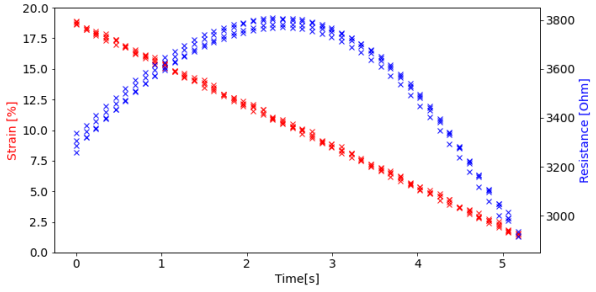


FIGURE 3.8: STRAIN VELOCITY RESISTANCE RELATIONSHIP SHOWING THE SPECIMEN IS RETURNING TO A 0% TENSILE STRAIN STATE FROM 10% AT A STRAIN RATE OF 80mm/s FOR FOUR TESTS FOR A 7.5% CB-PDMS SPECIMEN

train tests of 20% strain were completed. Using 20% strain allowed us to see a sufficient number of data points to observe a trend. The pulses had four repetitions with a range of strain velocities, $\dot{\varepsilon}(t)$, of 40, 80, 120 and 160 mms^{-1}. Using a 7.5 w.t.% CB-PDMS

specimen we obtain a relationship that agrees with the strain resistance component equation 3.6. As $\dot{\varepsilon}(t)$ increases through strain speeds so does the magnitude of the resistance peak (i.e. maximum height of the resistance peak - the previous steady state of value resistance) of 400, 510, 569, and 641 Ω for $\dot{\varepsilon}(t)$ of 40, 80, 120 and 160 mms^{-1} respectively. A new model is required which can accurately reproduce the additional decay time constant and small peak features seen in the resistance relaxation data, so that the resistance can inversely calculate the strain in the material.

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 is 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 hypothesized 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 Fig. 3.9. 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 3.4.

TABLE 3.4: AVERAGE INNER ELECTRODE CAPACITANCES, C_i, MEASURED FOR VARIOUS STRAIN, ε, VALUES USING A 7.5 W.T.% CB-PDMS COMPOSITE, MEASURED USING AN LCR METER AT 1kHz AND 10kHz

$\varepsilon[\%]$	0	10	20	30
$C_i[pF]$	53	32	24	20

The generalized Maxwell model has been applied to predict the stress relaxation of the CB-PDMS 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 +10% strain to 0% is not yet explained, and consideration of temperature and strain history[**?**] will be useful to confirm the simple mathematical model given as Eqn. 3.6.

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

CONCLUSIONS

In order to improve the accuracy of dynamic strain measurements with CB-PDMS composites a stress and analogous resistance relaxation model was formed. The generalized

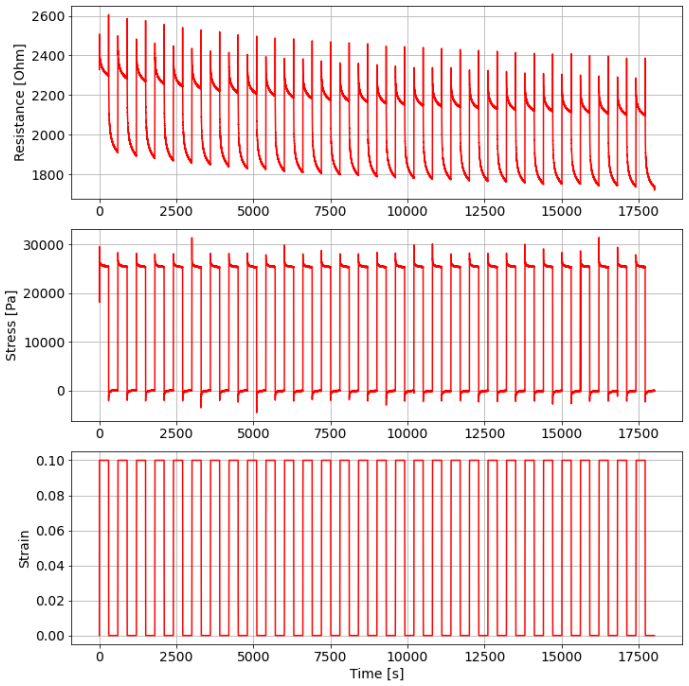


FIGURE 3.9: A TYPICAL TEST SEQUENCE OF A 30 PULSE STRAIN TRAIN RECORDING THE CALCULATED RESISTANCE AND STRESS OF A 7.5 W.T.% CB COMPOSITE

Maxwell model, Eqn. 3.3 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, Eqn. 3.4 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.

3.3 A Piece-wise Approach to Modelling Carbon Black Silicone Rubber Composites

One method for understanding the transient behaviour of CPECs is to create a classification system and determine mathematical relationships that can be matched to these

transient event. Mersch et al. have classified several shoulder events and the 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. This section aims to further classify these transient events and provide a mathematical relationship, for future use with model fitting methods.

3.3.1 Rising Edge Step Response

3.3.2 Falling Edge Step Response

As shown in Section 3.2 there has been a mathematical relationship observed between the falling edge of a strain input and the resultant resistance peak. Consequently a parameter fit study has been completed to determine how to predictably control the resistance peak through a controlled strain input. We can see a repeated property in Figure 3.8 whereby the derivative of the resistance signal seems to be equal to the strain curve.

To prove that there does exist a mathematical relationship between the two signals the relationship first each signal is given a generalised formula. The resistance signal is parabolic Equation 3.7.

$$R_p = A(t - H)^2 + K \tag{3.7}$$

Where strain rate changes the vertical shift, K, time shift, H, and concavity, A, of the parabola.

3.3.3 Strain Rate

3.3.4 Saw Tooth Response

3.4 Characterising Hysteresis

Chapter 4

A Novel Mixing Method for 3D Printing Conductive Particle Elastomer Composites

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4.1 Introduction

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Chapter 5

An Improved an Electrical Impedance Tomography Based Artificial Soft Skin Pressure Sensor

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Chapter 6

Giving Artificial Muscles the Sense of Touch

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Chapter 7

Hardware for a DEA-EIT Sensor Actuator Hybrid Device:

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7.1 Introduction

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Chapter 8

Modelling of DEA-EIT Capacitively driven Hybird Sensing and Actuation Device

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8.1 Objective One Conclusions

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Appendix A

An Appendix

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