WATER AND SALT WATER ABSORPTION, HYGROTHERMAL AND TRIBOLOGICAL PROPERTIRES OF NICKEL ZINC FERRITE MAGNETORHEOLOGICAL ELASTOMERS

BY:

CHANG FAN RU

(Matrix no: 125398)

Supervisor:

Dr Ramdziah Md Nasir

May 2018

This dissertation is submitted to

Universiti Sains Malaysia

As partial fulfillment of the requirement to graduate with honors degree in

BACHELOR OF ENGINEERING (MANUFACTURING ENGINEERING WITH MANGEMENT)

School of Mechanical Engineering

Engineering Campus

Universiti Sains Malaysia

DECLARATION

This work has not previously been accepted in substance for any degree and is not being concurrently submitted in candidature for any degree.

Signed: ………………………………………………… (CHANG FAN RU)

Date: …………………………………………………

Statement 1

This paper is the result of my own investigations, except where otherwise stated. Other sources are acknowledged by giving explicit references. Bibliography/references are appended.

Signed: ………………………………………………… (CHANG FAN RU)

Date: …………………………………………………

Statement 2

I hereby give consent for my paper, if accepted, to be available for photocopying and for interlibrary loan, and for the title and summary to be made available outside organizations.

Signed: ………………………………………………… (CHANG FAN RU)

Date: …………………………………………………

ACKNOWLEDGEMENT

First and foremost, I would like to express my deepest thanks to my supervisor, Dr Ramdziah Md Nasir precious advices and instructions for completion of the project. Whenever any problems and challenges faced, she always guide and discuss with me to come out ideas of solutions in order to make this project successful completed. I respect her for valuable guidance and ideas to make me more understandable this project.

Besides, I would like to dedicate my great appreciation to Dr Raa Khimi, lecturer from School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia whose expertise in magnetorheological elastomer (MRE). I would like to thanks for his assistance, suggestion, encouragement and guidance to let me understand on this material well. He also assigned his master students to guide me along during fabrication at rubber laboratory.

Next, a lot of thanks to technicians from School of Mechanical Engineering and School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia who's always help me when I faced problems during experimental works. I would like to appreciate their effort, helpful and friendly guidance and contribution who lend me helpful hands during my difficulties through this journey of completion of project.

Table of Contents

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

Symbols	**Descriptions**
%	percent
°C	degree Celcius
Φ or D	diameter
cm	centimeter
g	grams
g/cm^3	grams per centimeter cube
k	average specific wear rate
mT	millitesia
mm	millimeter
mm/s	millimeter per seconds
m/s	meter per seconds
m	meter
ml	milliliter
mm^3	millimeter cube
m.m	minutes of cure per millimeter of wall thickness
mm^3/Nm	millimeter cube per Newton meter
rpm or RPM	revolution per minutes
N	Newton
w/w	mass fraction
µm	micrometer

phr	parts per hundreds rubber
ppt	parts per thousands
M	increase of weight
MPa	megapascal
M_a	mass of dry and clean volumetric cylinder
M_b	mass of NZF MRE sample in air
M_c	weight of conditioned
M_w	weight of wet sample
M_1	initial mass before wear test
M_2	mass after wear test
T_{90} or t_{90}	optimum cure time
t_{s2}	scorch time
V_a	volume filled with distilled water and sinker
V_b	volume of water displaced by submerged NZF MRE sample

LIST OF ABBREVIATIONS

Abbreviations	Descriptions
ANISO	Anisotropic
ANISO CB	Anisotropic with added carbon black
CBS	N-Cyclohexyl-2-benzothiazole sulfenamide
COF	Coefficient of Friction
EDX	Energy Dispersive X-Ray Analysis
FTIR	Fourier Transform Infrared Spectroscopy
IPPD	N-Isopropyl- N'-Phenyl-P-Phenylenediamine
ISO	Isotropic
MRE	Magnetorheological Elastomer
MRF	Magneto rheology fluid
Ni	Nickel
NaCl	Sodium chloride
NZF	Nickel Zinc Ferrite
POD	Pin On Disc
SEM	Scanning Electron Microscopy
SMRL	Standard Malaysia Rubber Grade L
Zn	Zinc

ABSTRAK

Magnetorheological Elastomers (MREs) merupakan sejenis bahan baru di bawah penyelidikan pembangunan untuk menawarkan keupayaan redaman yang tinggi kepada aplikasi kegunaan yang banyak supaya boleh menggantikan penggunaan getah sebagai peredam. Kebanyakan kajian penyelidikan hanya memberi tumpuan kepada sifat-sifat bahannya seperti kesan magnetorheologi dan kekurangan kajian terhadap sifat-sifat mekanikal kepada aplikasi spesifiknya. Ciri-ciri mekanikal seperti kekerasan, ciri-ciri tribologi dan penyerapan air dan air masin dengan kesan higrohaba akan menjadi kajian lanjut mengenai prestasi Magnetorheological Elastomer dalam aplikasi peralatan marin. Isotropik, anisotropik dan anisotropik dengan pertambahan pengisi hitam karbon N330 bagi Ni Zn Ferrite Magnetorheological Elastomers (MREs) telah disediakan dengan Ni Zn Ferrite, $Ni_{1-x}Zn_xFe_2O_4$ dengan berat zarah sebanyak 70%. Gambar-gambar Pengimbasan Mikroskopi Elektron (SEM) dan Analisis Diserap X-Ray Tenaga (EDX) akan diujikan dengan kehadiran struktur Ni Zn Ferrite dalam matriks getah asli. Penyerapan air garam and air dengan kesan hygrothermal, peningkatan peratusan berat sample semasa rendaman dalam tempoh 336 jam akan dianalisa. Ciri-ciri Tribologi bagi ketiga-tiga sebatian ini akan dipelajari oleh mesin pin-atas-cakera berdasarkan perspektif spesifik purata koefisien geseran (COF), spesifik kadar kehausan, dan jumlah kehilangan kehausan dalam keadaan awal dan selepas rendaman air dan air garam dengan peraplikasian kelajuan dan beban yang berlainan. Prestasi kehausan bagi ketiga-tiga sebatian MREs tersebut akan berkadar terus dengan kekerasannya. Anisotropik dengan pertambahan pengisi hitam karbon akan diberi nilai kekerasan tertinggi 90.78; kekurangan penyerahan kesan dalam air garam dan air dengan kesan higrohaba; prestasi kehausan yang lebih baik disebabkan kerendahan spesifik purata koefisien geseran, spesifik kadar kehausan dan jumlah kehilangan kehausan yang paling kurang. Oleh itu, ia merupakan sebatian yang paling sesuai untuk aplikasi masa depan dalam persekitaran yang basah pada jangka masa yang panjang.

ABSTRACT

Magnetorheological Elastomers (MREs) known as new material under development research to offer high damping capability for many applications use to replace limitations usage of rubber as a damper. Many research studies only focused on its material properties such as magnetorheological effect and lack of studies on mechanical properties of its specific applications. Mechanical properties such as hardness, tribology characteristic and salt water and water absorption with hygrothermal effect will be further studies on wear performance of Magnetorheological Elastomers on marine equipment applications. Isotropic, anisotropic and anisotropic with added N330 carbon black filler of Ni Zn Ferrite Magnetorheological Elastomers (MREs) are prepared with Ni Zn Ferrite filler, $Ni_{1-x}Zn_xFe_2O_4$ particles weight fraction of 70%. Images of Scanning Microscopy Electron (SEM) and Energy Dispersive X-Ray Analysis (EDX) are identified with the presence of the structure of Ni Zn Ferrite in the natural rubber matrix. In salt water and water absorption and hygrothermal effect, an effect on the percentage of increase in weight of samples during immersion within the period of 336 hours will be analyzed. Tribology characteristics of these three compounds are studied by pin-on-disc machine based on the perspective of the average specific coefficient of friction (COF), specific wear rate, and loss of wear volume initial condition and after water and salt water immersion with different speeds and loads applied. Wear performance of three compounds of MREs is directly proportional to its hardness. Anisotropic Ni Zn Ferrite MREs with added carbon black filler given highest hardness value of 90.78; least effect on salt water and water absorption and hygrothermal effect; better wear performance with a low average specific coefficient of friction, specific wear rate and least loss of wear volume. Therefore, it is most suitable use for future application in a wet environment for a long period.

CHAPTER 1.0 INTRODUCTION

1.1 Introduction to Magnetorheological Elastomer

Magnetorheological elastomers (MREs) classified as smart materials that have the capability to alter their mechanical, electrical, magnetic properties with external factors applied such as temperature, magnetic or electrical field, stress and moisture. Combination of rubber matrix (non-magnetic) with ferrite particles (magnetically permeable) in the scale of a micron or nano size with external magnetic field applied on the magnetic mold during curing process provide them to enhance rheological properties. The reasons of chosen ferrite particles as the main component of MREs as they have high permeability and low hysteresis loss compared to other magnetical particles [1] .

There are two types of solid-state Magnetorheological elastomers (MREs) which are isotropic MREs and anisotropic MREs. With the discovery of phenomena structure of Isotropic MREs and anisotropic MREs by S.Raa Khimi shown in Fig 1.1 [2]. Structure of isotropic MREs will show a noticeable structure of a uniform distribution of ferrite particles inside the rubber matrix. Anisotropic MREs will give special chain-like structures of ferrite particles linked arrangement in rubber matrix from the process of curing matrix when applied the magnetic field.

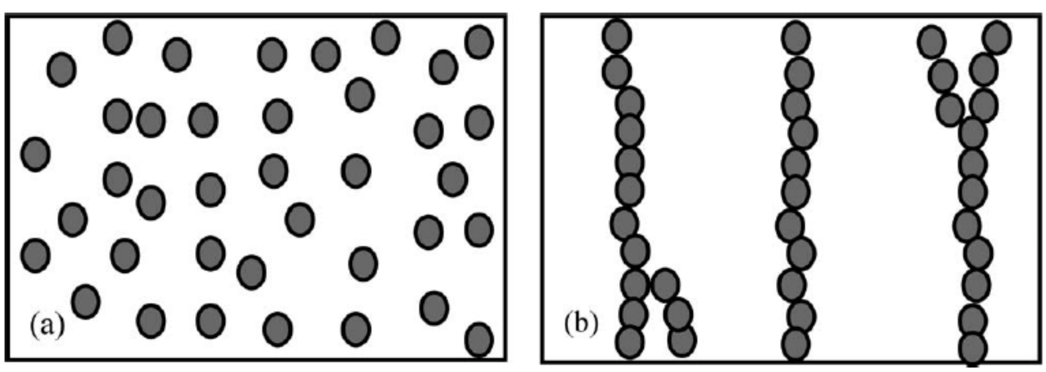


Fig 1. 1 shows the phenomena structure of MREs (a) Isotropic (b) Anisotropic[2]

The chosen of ferrite particles are Ni Zn Ferrite (NZF) $Ni_{1-x}Zn_xFe_2O_4$, where x>1 with particle weight fraction is 70% inside a base natural rubber matrix and applied magnetic field with 230 mT. Particles size of NZF in between 10 to 30 µm with irregular shape form with added to rubber matrix during compounding stage. Advantages of input

NZF particles used in rubber matrix as formed of MREs provide high saturation magnetization, high electrical resistivity, enhance mechanical hardness, tensile strength and cost-effectiveness [3, 4]. Physical properties of Ni Zn Ferrite Magnetorheological Elastomers (NZF MREs) such as mechanical hardness and tribology studies on wear and friction characteristic will be studied with specially applied of the external factor such as water and salt water absorption at room temperature and a high temperature about 60˚C.

1.2 Problem Statement

Magnetorheological Elastomers (MREs) usually applied to vibration absorbers; engine mount; seismic isolation for bridge and building structure due to its excellent dynamic performance, damping properties, low hysteresis loss and good low temperature properties[5]. Many research studies on MREs only focused on their material properties such as MR effect without considering on their applications. There is lack of studies on MREs tribology properties when applied to a certain application. The durability and hardness of MREs will be affected by wear and friction properties. Studies of wear and friction characteristics on MREs is significant because this property is highly related to durability performance of MREs when leading to the expansion of the area where the elastomer can be applied in future prediction[6].

1.3 Research Objectives

Firstly, objectives of this research are stated below:

- To study on changes of tribological characteristics such as average specific coefficient of friction (COF), specific wear rate, and loss of wear volume of isotropic, anisotropic and anisotropic with added carbon black filler NZF MREs based on material hardness and the magnetic field applied.
- To investigate the wear performance of these three compounds based on the external factors such as water and salt water absorption and hygrothermal effect with the specified application on the wet environment for future prediction.
- To study on microscopic changes on identification wear and friction characteristic on three different compounds of MREs.

CHAPTER 2.0 LITERATURE REVIEW

Magnetorheological elastomers (MREs) consists of liquid-state and solid-state material. They have their advantages of providing MR fluid (MRF) which can changes their rheological properties frequently and provides better mechanical, electrical, damping properties compare to the conventional rubber products application. However, for liquid-state MREs has occurred a problem of particle sediment which can be solved by solid-state MREs. Solid-state MREs are formed with the rubber-matrix such as natural rubber as the major component to provide excellent mechanical performances without disturbing the performance of MRF effect [7]. In this research, solid-state MRE is chosen with added NZF filler as the experimental specimen.

Normally traditional rubber material is commonly used for damping applications that rely on its energy absorb due to occur in viscous flow when deformation happen in the viscoelastic material [2]. Therefore, adding natural rubber to the formation of magnetorheological elastomers that can provide features of damping capability of this material composites. Besides of the perspective of its excellent material characterization, studied on its performance behavior through its application is highly important to estimate its service life based on durability on wear performance when it's applied to different environmental conditions.

Based on the study of aging on natural rubber in air and seawater when attached to air environment at different temperatures, some basic study on mechanical properties such as result of elongation on stress-strain curve of rubber is investigated. When natural rubber tested at the highest temperature (120°C), although elongation on the stress-strain curve is less than 50% but still proven temperature factor has caused the aging effect on rubber composites as shown in Fig 2.1. When natural rubber immersed in seawater , aging shown less effect on the changes of stress-strain curve and only will start failure when exposure long time. These concluded that failure properties decline with the extent of aging and the fatigue life of natural rubber performance decrease depend on the exposure of time of aging. Besides that, environment assumption made on the temperature dependence of oxygen solubility in seawater and air on the samples shows the oxidized layer only happened on the surface of samples due to bacterial attack that degrades the natural rubber is not relevant herein. Fluid absorption and leaching can influence the performance of rubber exposed to liquid media but it takes very long time to degrade in seawater [8].

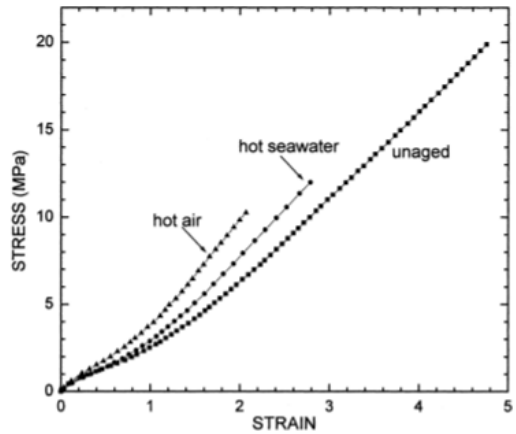


Fig 2. 1 Failure behavior of rubber on the elongation of stress strain curve under seawater aged (328.2h at 98°C) and air aged (44.8 h at 120°C) [8]

In the case of hygrothermal aging of rubber-modified and mineral-filled Dicyandiamide-Cured DGEBA Epoxy Resin, the sample was immersed to deionized water and 5% mass fraction (w/w) sodium chloride solution at 65°C with the different period of immersion. The result shown that percentage of increasing weight during absorption of deionized water up to 4.3% will lead to plasticization of material and the absorbed water are remains in the microcavities. The presence of sodium chloride will prevent the microcavity growth and reduce the percentage of increasing weight by absorption NaCl solution in the material. According to Karasz,F research, water entrapped in the material will not lead to failure on its mechanical properties [9]. Throughout this immersion period, the weight of material will slightly reduce due to the leaching of material [10].

Therefore, advanced composites structures material such as MREs can be tested on its hygrothermal aging due to it can be used in the various external environment. According to Hiratsuka and Hosotani research has proven that MREs has a weak resistance during changes in environmental condition such as temperature and relative humidity that will lead to the effect of friction and wear properties of MREs [11]. Effect of temperature and relative humidity on wear and friction properties on Silicon-Based MREs has been studied using reciprocating tribology tester under various temperatures (25 °C, 50 °C, 75 °C and 100 °C) and relative humidity (40 % to 80 %). The reciprocating tester was set as velocity 10 mm/s and load 2 N with non-applied magnetics and applied magnetics on the silicon-based MREs. It also is proven that MREs surface could not withstand high load and high heat. Benefits of fillers with applied magnetics can contribute to the hardness of the MREs structure which leads to low effect of high temperature, low coefficient of friction, small deformation on the contact surface, small and stable wear rate. Besides that, investigation on the coefficient of friction of elastomer has affected by surface temperature when sliding speed increases. A large coefficient of friction generated by surface flow increased and surface temperature increased as shown in Fig 2.2 [12].

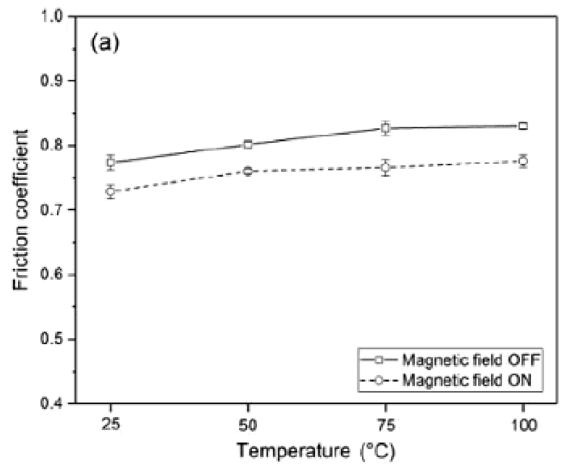


Fig 2. 2 Average coefficient of friction at different temperature with applied magnetic field and without applied magnetic field[12]

The coefficient of friction is inversely proportional to relative humidity. This may due to the local hydrodynamic effect of microasperities and microcavities in the interface giving a lubricating effect to the elastomer. This also decreased energy loss to the friction applied which also lead to low wear rate. Therefore, water absorption could reduce the shear strength of contact surface between MREs and tester pin used that also lead to the reduction of a coefficient of friction shown in Fig 2.3.

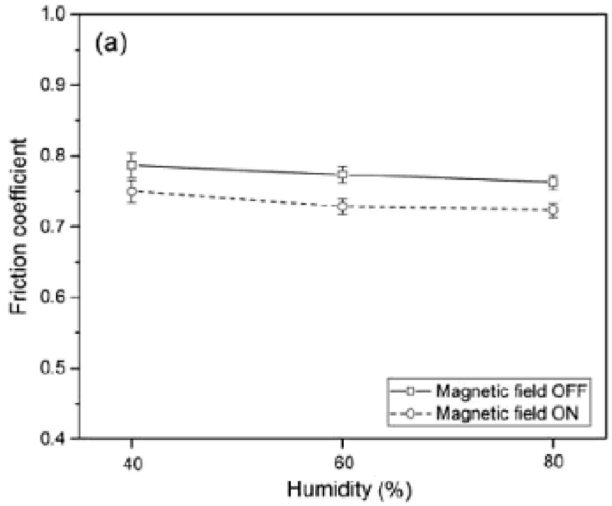


Fig 2. 3 Average coefficient of friction at different relative humidity with applied magnetic field and without applied magnetic field[12]

Wear surface such as Schallamach waves are generated at normal room temperature and relative humidity 60 % as shown in Fig 2.4. When temperature increases, worn holes are being observed due to the high surface flow of MREs.

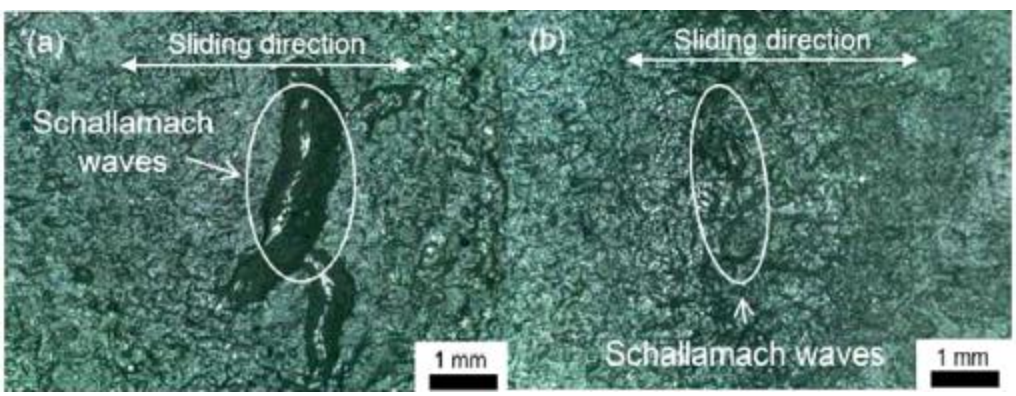


Fig 2. 4 Wear surface of MREs (a) without applied magnetic field (b) with applied magnetic field[12]

Tribological characteristics of MREs to further understand the behavior of wear and friction of MRE with different loading and velocity applied. Five types of macromolecules with different viscosities and particles powder filled in the elastomer matrix such as Fe, Ni and Co was prepared. Wear and friction characteristics of MRE is studied using pin on disc tester and linear sliding friction tester with different parameters setup such as velocity (1 mm/s, 2.36 mm/s, 10 mm/s and 37.5 mm/s); load applied (2 N, 5 N, 6 N, 10 N and 15 N); 2 hours test time; track distance 20 mm and applied or non-applied magnetic field. The sample of MREs was prepared with diameter 59.5 mm and thickness 6mm and 15 mm. MRE with the applied magnetic field has given the low coefficient of friction and wear due to the magnetic field has changed the stiffness and modulus of the MRE as shown in Fig 2.5 [13].

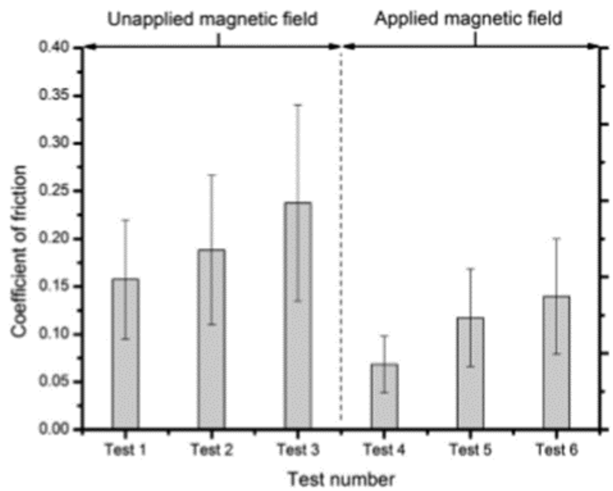


Fig 2. 5 Average coefficient of friction with applied magnetic field and non-applied magnetic field[13]

When load applied increases, the coefficient of friction of MRE also will increases. Therefore, MRE has better friction performance lower load and applied magnetic field. Surface morphology on Schallamach waves and wear particles of MRE with applied magnetic field and non-applied magnetic field are shown in Fig 2.6.

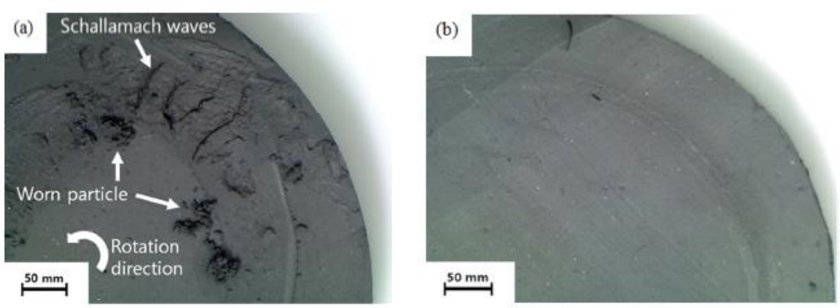


Fig 2. 6 Wear surface of MRE (a) non-applied magnetic field (b) applied magnetic field[13]

Another observation based on the beginning of the friction test, graph of coefficient of friction will not began at the zero reference point. The sudden increase of coefficient of friction due to the compression of surface contact between MRE and sliding disk with load applied. When sliding speed increases and applied magnetic field, stick-slip phenomena of

MRE will decreases. The slip-stick phenomena occurred when tearing of skin layer away from MRE [13].

Tribology characteristic on a hybrid of silicon(Si) and polyurethane(Pu) in MRE also has been studied using reciprocating friction tester with the parameter setting such as sliding speed 5 mm/s; load applied 2 N and test time 2 hours. Four types of different fabrication method to make a hybrid Si and Pu MRE with diameter 60 mm and thickness of 15 mm was prepared. First, the hardness of MRE is determined because the hardness of material related to friction characteristic of MRE. The result shows that MRE with the applied magnetic field has higher hardness value as shown in Fig 2.7 [6].

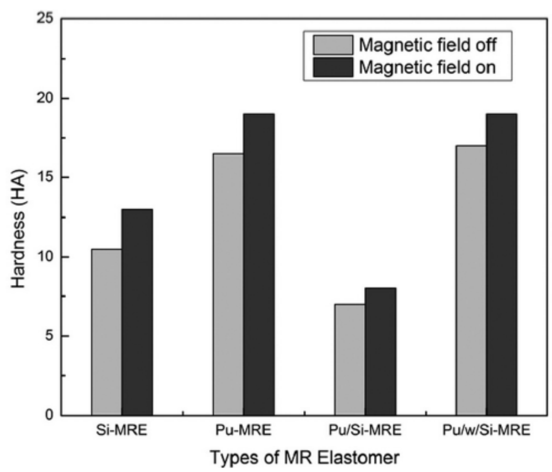


Fig 2. 7 Hardness value of different types of Si/Pu MRE with applied and non-applied magnetic field[6]

The coefficient of friction for non-applied magnetic field MRE will increase sharply will lead to poor friction performance while applied magnetic field MRE will have the low coefficient of friction as shown in Fig 2.8.

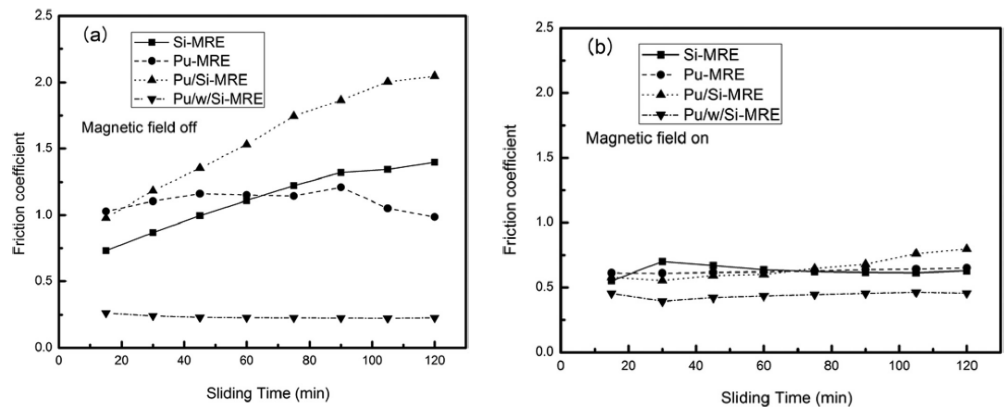


Fig 2. 8 Coefficient of fiction of different types of Si/Pu MRE with applied and non-applied magnetic field[6]

The contact area and pressure increases between MRE and sliding disc, more friction heat will be generated. This will reduce adhesion during friction test. Wear depth can be measured by cutting the MRE sample into half and measure through cross section. Low wear depth lead to good heat resistance and thermal conductivity of MRE. When more friction heat generated at contact interface, the flowability of MRE will leads to increase of wear. The high hardness of material will be more stable and small coefficient of friction, small wear depth and less wear damage on the surface of MRE [6].

This is a new study will be carried out to understand mechanical properties such as wear and friction characteristic on the NZF magnetorheological elastomer. Another method of applied magnetic field is used during fabrication curing stage where magnetic mould is used for pre-alignment of NZF particles in rubber matrix during curing instead of using a reciprocating friction tester or pin on disc with providing magnetic field during testing. The wear and friction characteristic of this material will be investigated with another way of the applied magnetic field. Besides that, changing in environmental condition such as temperature effect and immersion samples with water or other solution may change the wear and friction characteristic of MREs when MREs are applied in the dry and wet environment in various applications.

CHAPTER 3.0 METHODOLOGY

3.1 Overview

Three types of magnetorheological elastomer (MRE) compounds which natural rubber filled with NZF particles sized between 10 to 30 µm and comprising with particles weight fraction 70%. Compound 1 is an isotropic compound without applied magnetic field. Compound 2 and 3 are an anisotropic compounds with the applied magnetic field 230mT whereby Compound 2 without added filler of N330 carbon black and Compound 3 with an added filler of N330 carbon black with particles sized around 300µm. These three compounds were formulated using a conventional two roll mill machine (model XK 160). Cure times were determined using rheometer MDR 2000. The three compounded MREs were cured using hot press under temperature of 150 °C.

Scanning Microscopy Electron (SEM) and Energy Dispersive X-Ray Analysis (EDX) are used to identify the presence of the structure of NZF in the natural rubber matrix. Experimental and analytical works were done to study wear and friction characteristic of these three compounds using pin on disc (POD) machine before and after salt water and water absorption at room temperature and hygrothermal effect at 60˚C. Surface morphology was captured and measured using USB optical microscopy with software Dino Capture.

3.2 Scope of Research

The aim of this research is to study the relationship between water and salt water absorption properties along with hygrothermal effecting tribological performance on isotropic, anisotropic and anisotropic with added carbon black filler NZF MREs prepared. Overview of flowchart scope investigation based on experimental and analytical works are shown in Fig 3.1.

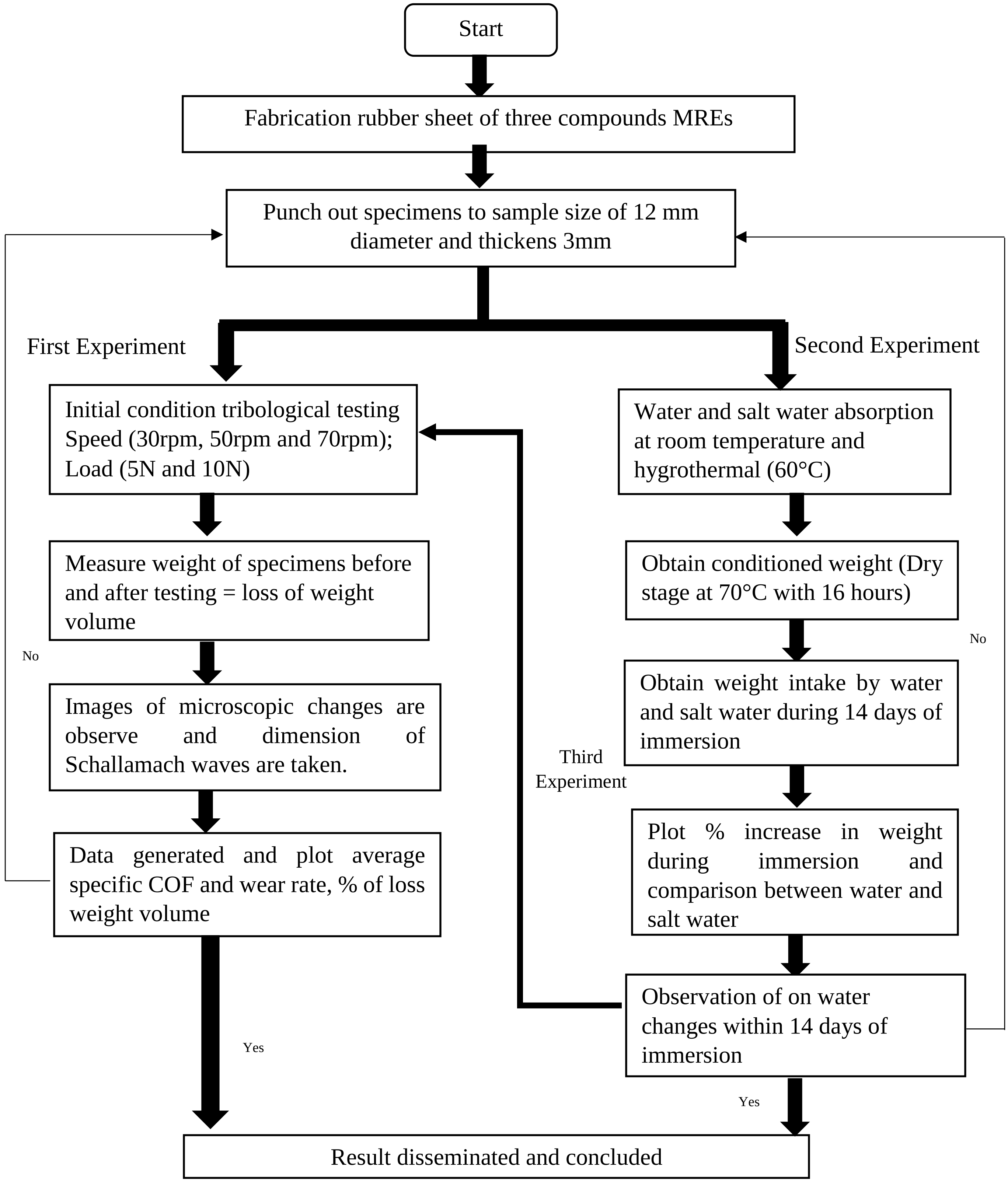


Fig 3. 1 Overview of Flowchart on Scope Research

3.3 Selection of Materials in Rubber Compounding

Natural Rubber – SMR L Grade

Fig 3. 2 Natural Rubber – SMR L Grade

Natural Rubber is one of the main components in the raw magnetorheological elastomer in this study. Natural Rubber used was under the Standard Malaysia Rubber Grade L (SMRL). Natural rubber is a form of polymer polyisoprene that harvested from trees and sensitive to vulcanization when reacted to sulphur. Reasons of chosen natural rubber compared to synthetic rubber in this study is based on its applications used for producing universal products that provide high tensile strength and moderate wear resistance for MRE materials. Natural rubber also is known as the ecological friendly natural product that provides less pollution and waste to the environment [14].

Zinc Oxide

Fig 3. 3 Zinc Oxide

Zinc Oxide is known as an activator of accelerating in the vulcanization of rubber compounding process. With the added of Zinc Oxide, it will speed up the rate of curing and function as neutralization of sulphur-based rubber compounding.

Stearic Acid

Fig 3. 4 Stearic Acid

Stearic Acid is another type of ingredient used as rubber accelerator in rubber vulcanization. It function as an activator, dispersing agent, plasticizer and lubricant in rubber compound processing. It usually will be added to the presence of Zinc Oxide forming during compounding.

Parafin Oil

Fig 3. 5 Parafin Oil

Paraffin oil is a type of natural oil known as a plasticizer to soften the rubber compounds when mixing with the filler such as carbon black and easy processing of finished rubber compound. It also provides easy flow when makeup primary linking with hard filler and rubber compound that is resistant to flow.

Ni Zn Ferrite

Fig 3. 6 Ni Zn Ferrite

Ni Zn Ferrite is known as primary filler and type of the main component in the recipe of forming MREs where it provides magnetic rheological properties for rubber compounding. It given high saturation magnetization, high electrical resistivity, enhance mechanical hardness and tensile strength and cost-effectiveness for finished MREs compound.

Carbon Black N330

Fig 3. 7 Carbon Black N330

Added of Carbon Black filler as secondary fillers can increase of viscosity, the rate of vulcanization, hardness, tan δ, and the tensile strength of the finished MREs compound. Besides that, added of carbon black filler absorb more energy and improve damping properties of MREs[5]. Therefore, N330 Carbon Black filler is chosen as another anisotropic compound of MRE with particle weight fraction of 50 wt % to study better wear performance.

Tetramethylthiuram Disulfide (TMTD)

Fig 3. 8 Tetramethylthiuram Disulfide (TMTD)

Tetramethylthiuram Disulfide (TMTD) known as a secondary accelerator to activate the primary accelerators for a sulphur donor. It provides the ultra-fast speed of curing, higher crosslink density under a long scorch safety, lower heat buildup during vulcanization of rubber compound [15].

N-Cyclohexyl-2-benzothiazole sulfenamide (CBS)

Fig 3. 9 N-Cyclohexyl-2-benzothiazole sulfenamide (CBS)

N-Cyclohexyl-2-benzothiazole sulfenamide (CBS) known as a primary accelerator which delayed action as well as the faster rate of cure when rubber compound containing furnace blacks. Therefore, TMTD is added to boost the delayed actions of CBS to give faster cure rate at the expense of scorch safety [15].

N-Isopropyl- N'-Phenyl-P-Phenylenediamine (IPPD)

Fig 3. 10 N-Isopropyl- N'-Phenyl-P-Phenylenediamine (IPPD)

N-Isopropyl- N'-Phenyl-P-Phenylenediamine (IPPD) known as an antioxidant for rubber compounding which used to protect degradation and failure of rubber product when it applied to ozone environment. It also provides good heat resistance and sustains physical and surface properties in the rubber compounding. The solid particles of IPPD need to be smashed to powder form before added to fabrication rubber compound to provide uniform distribution of IPPD particles during mixing.

Sulphur

Fig 3. 11 Sulphur

Sulphur was used for treatment to make rubber compounding harden. It usually added in the last step in mixing rubber compounding. When Sulphur added to rubber, a reaction will happen to increase cross-linking between molecules by providing sulphide bonds in the rubber chains. To this vulcanization treatment of rubber also can enhance elasticity at low and high temperature.

3.4 Formulation on Ni Zn Ferrite Magnetorheological Elastomer

Table 3. 1 Formulation of Ni Zn Ferrite MREs

Materials	Isotropic MRE		Anisotropic MRE		Anisotropic MRE	
	phr	grams	phr	grams	phr	grams
SMRL grade Natural Rubber	100	162.16	100	162.16	100	127.66
ZnO	5	8.11	5	8.11	5	6.38
Stearic acid	1	1.62	1	1.62	1	1.28
Paraffin oil	3	4.87	3	4.87	3	3.83
NiZn Ferrite	70	2.43	70	2.43	70	1.92
CBS	2	113.52	2	113.52	2	89.36
TMTD	1	3.24	1	3.24	1	2.55
Sulphur	1.5	1.62	1.5	1.62	1.5	1.28
IPPD	1.5	2.43	1.5	2.43	1.5	1.92
Carbon Black N330	-	-	-	-	50	63.83
Total Materials (phr)	**185**	**300.00**	**185**	**300.00**	**235**	**300.00**

Calculation on conversation of part per hundred of material to mass of material in Table 3.1 by using Formula 3.1,

$$\frac{Each\ material\ (phr)}{Total\ Material\ (phr)} = \frac{Mass\ of\ each\ material(grams)}{Total\ mass\ of\ materials(grams)} \quad (3.1)$$

Total mass of materials needed for fabrication MRE for each Isotropic and Anisotropic compound = 300 grams

3.5 Fabrication Process for Ni Zn Ferrite MREs

Preparation of compound formulation used in this study is followed in Table 3.1. The fabrication of Isotropic NZF MREs consists of two major steps which are mixing and curing without the applied magnetic field. While fabrication of anisotropic NZF MREs consists of three major steps which are mixing, pre-alignment and curing. The mixing process for both isotropic and anisotropic are using a conventional laboratory two roll mill with model XK 160 according to ASTM D3184-80 was shown in Fig 3.15 (Section 3.5 page 23). The distance between front and a back roller was maintained at 1 mm during compounding and lasting change to 2 mm for rubber sheet shape forming. In the beginning, mastication of SMRL natural rubber was carried out to soften rubber, reduces viscosity and increase the plasticity of it. After 3 minutes, the rubber became invested and activator was added such as Zinc Oxide and Stearic acid and mixed around 4 minutes to make sure Zinc Oxide and Stearic Acid fully mixed homogeneously with the help of shearing rubber regularly. Next, fillers such as NZF particles and N330 carbon black were added together with paraffin oil as a plasticizer to make sure the NZF particles are easy to flow and mix with the rubber compound and mixed around 5 minutes. Then, primary accelerator CBS and followed by secondary accelerator TMTD were added to the rubber compound to speed up the cure rate and mixed around 4 minutes. Antioxidant IPPD was mixed with the rubber compound around 2 to 3 minutes. Sulphur was added as curing agent at the last to prevent premature vulcanization during rubber compounding. The total cycle time of mixing was tabulated in Table 3.2 [5].

Table 3. 2 Total cycle time of mixing process

Sequence of Materials compounding	Cycle Time (in minutes)		
	Isotropic MRE	Anisotropic MRE	Anisotropic MRE
SMRL grade Natural Rubber	3	3	3
Zinc Oxide	2	2	2
Stearic Acid	2	2	2
Ni Zn Ferrite with paraffin oil	5	5	5
Carbon Black with paraffin oil	-	-	7
CBS	2	2	2
TMTD	2	2	2
IPPD	2.30	2.30	2.30
Sulpur	2	2	2
Total Cycle Time	**20.3**	**20.3**	**27.3**

The cure time of three MREs compounds were determined by using MDR Rheometer with model 2000 according to ASTM D 2084 as shown in Fig 3.16 (Section 3.5 page 23). The optimum cure time (t_{90}) is the time required for the torque to reach 90 % of the maximum achievable torque. The curing characteristic was determined at 150˚C and a vulcanization curve was plotted from the graph of torque versus time to obtain t_{90} [16]. The value of t_{90} in units of m.m represented as required minutes of cure per millimeter of wall thickness. Therefore, the appropriate cure time of MRE compounds should multiply with the thickness of the MREs fabricated using Equation 3.2.

$$Approriate\ Cure\ Time = \ t90(m.m)x\ thickess\ of\ the\ frame \qquad (3.2)$$

Compounded rubber samples were weighted 35 g to 40 g and then placed in a frame mould with dimension 288 x 240 mm and covered by as shown in Fig 3.11.

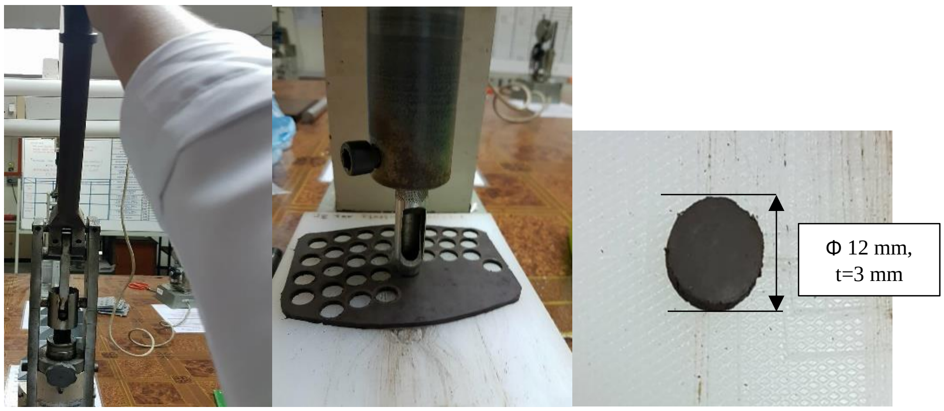
Fig 3. 12 compounded rubber sample weighted in frame mould

For compounded rubber to fabricate as isotropic NZF MRE were cured in compression frame mould at 150 ˚C under pressure 10 MPa by using hot press machine with model 120T as shown in Fig 3.17 (Section 3.5; page 23). For anisotropic NZF MREs were fabricated with external magnetic mould 230 mT. The external magnetic mould needs to preheat at 80 ˚C before compounded rubber sample was put between the external magnetic mould to undergo pre-alignment of NZF particles using manual hot press machine at 80 ˚C for 30 minutes under pressure 12 MPa as shown in Fig.3.12. Laser thermometer was used to check the temperature frequently. After the pre-alignment process, the compounded rubber samples undergoes post cure at 150 ˚C under pressure 10 MPa using hot press machine with model 120T. The fabricated NZF MRE sample was taken out and removed the excessive rubber compound as shown in Fig 3.13.

Fig 3. 13 Compounded rubber samples undergoes pre-alignment process

Fig 3. 14 Fabricated Anisotropic NZF

Treatment of cooling fabricated NZF at room temperature to re-orientate the magnetic dipoles after post cure [5]. After 24 hours of cooling treatment, NZF MRE can be punched out using manual puncher with the 12mm diameter puncher to a sample with dimension 12 mm diameter x 3 mm thickness as shown in Fig 3.14.



(a) (b) (c)

Fig 3. 15 (a) and (b) manual punching with 12mm puncher (c) punched sample 12mm diameter with 3mm thickness

3.6 Fabrication Machines Used and Standard Operating Procedure

Conventional Laboratory Two Roll Mill Model XK 160

Fig 3. 16 Machine used for mixing rubber compound

Before starting the machine, remove all the accessories on hand and do not use a glove during mixing. It is mandatory to wear a lab coat and safety shoes before operating machine. The main switch was turned on and ensure the emergency switch function was functioning. A sample of non- used rubber sheet was used for adjusting the gap of the roll to a proper thickness of 1 mm by turning 2 rounds from roll wheel. A stopwatch was used during mixing in order to follow the cycle time needed for rubber compounding. Mask was needed to be worn for rubber compounding included added of carbon black.

MDR Rheometer Model 2000

Fig 3. 17 Machine used for determine cure time

Main switch and pressure gauge were turned on. 4.0 grams uncured rubber sample from different MRE compounds was prepared and put in the center of the test die. At the menu buttons, the temperature was set to 150°C and test time set to 30 minutes. Next, "Motor" and "Platens" buttons were pressed to start measured the cure time.

Hot Press Machine 120T

Fig 3. 18 Machine used for curing rubber compound

Main power supply and main switch from the machine were turned on. Manual Mode was set and enter cure time (in seconds) and non-select all the pressers on the menu screen. The temperature was set at 150°C and wait for temperature constant. Safety gate was opened and mould was placed with MRE sample into machine. Safety gate was closed tightly and the machine set to Auto Mode. Auto Start was pressed to wait for the curing process.

3.7 Hardness Specification

The degree of hardness of isotropic, anisotropic and anisotropic with added of carbon black NZF MREs samples were measured using TECLOCK GS-706G Type A Durometer according to ASTM D2240. A base of durometer was in contact with NZF MREs samples by applied pressure force manually. The repulsive force was observed after applied pressure force that indicates the reading of the hardness value on the durometer shown in Fig 3.18. These tests were repeated on three times for each compound.

Fig 3. 19 Measuring Hardness of NiZn Ferrite MRE samples using durometer

3.8 Density Specification

The density of isotropic, anisotropic and anisotropic with added of carbon black MREs sample were determined by using indirectly method from Archimedes' Principle. Initial mass of dry and clean volumetric cylinder was measured using a digital analytic balance with precision 0.0001 g and recorded as M_a. The NZF MRE sample in air was weighed as M_b. Distilled water was filled in the volumetric cylinder and a sinker was put into water and read from the scale level from volumetric cylinder and recorded as V_a. The