

RHEOLOGICAL PROPERTIES OF MAGNETORHEOLOGICAL ELASTOMER
WITH FOUNTAIN-LIKE PARTICLE CHAIN ALIGNMENTS

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DEDICATION

This thesis is dedicated to my parents for their motivations and unwavering support.

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ABSTRACT

Magnetorheological elastomer (MRE) consists of magnetic particles known as carbonyl iron (CIPs), which are locked in a silicone-based matrix in various configurations or alignments, depending on the curing process of the MRE. However, current MREs exhibit different properties due to different CIP's alignments in the MRE. In fact, most previous studies have focused on a specific angle of the aligned particles to achieve the enhanced viscoelastic properties of MRE. Thus, its effect on the MRE's stiffness is still rather limited in various devices. In addition, the changes in directions of applied shear force could not result in maximum stiffness or MR effect of MRE, since the interaction of the applied force with the material is effective only in one direction of the particle's chain alignment. Therefore, in this study, an approach of particle's alignment of CIPs in an MRE namely, fountain-like structure is introduced to produce numerous angles of CIPs arrangement in the MRE. This study began with the development of a mould to produce numerous directions of magnetic flux lines, in order to have a fountain-like structure for the CIPs to be cured accordingly in the MRE during the curing process. The simulation of the fountain-like magnetic flux lines was done via FEMM analysis. Three types of MREs having different curing structures namely isotropic, fountain-like MRE, and inverted fountain-like MRE were fabricated. The rheological properties of these MREs in terms of storage modulus and magnetorheological (MR) effect were measured in an oscillatory shear mode using a rheometer upon input parameters of sweep strains, sweep frequency and sweep magnetic fields. Meanwhile, the micrograph analyses of all MRE samples were done via FESEM. The results revealed that both fountain-like MREs exhibited higher storage modulus than the isotropic MRE, about 0.06 to 0.1 MPa under the absence of magnetic field (off-state condition), and the values were further increased with the applied magnetic field (on-state condition). In particular, storage modulus of fountain-like MRE was higher as compared to inverted fountain-like MRE. However, the MR effect of inverted fountain-like MRE has overridden fountain-like MRE attributed to its lower initial storage modulus. On the other hand, the phenomenon of higher storage modulus in fountain-like MRE is due to the cramped CIPs upon applied shear stress, thus it was stiffer to resist deformation, as compared to inverted fountain-like MRE which was more expanded towards the applied shear stress. The findings show that fountain-like MREs exhibit the utmost response in an oscillatory shear mode application, for both the off- and on-states conditions, which this novel approach has the potential to be used for the in-situ fabrication method of MRE devices.

ABSTRAK

Elastomer reologi magnet (MRE) terdiri daripada partikel magnetik yang dikenali sebagai partikel ferum karbonil (CIP), diletakkan dalam matrik berasaskan silikon dalam pelbagai konfigurasi atau susunan, bergantung kepada proses penghasilan MRE. Walau bagaimanapun, MRE semasa menunjukkan sifat yang berbeza disebabkan penjajaran CIP yang berbeza dalam MRE. Malah, kebanyakan kajian terdahulu telah tertumpu pada sudut khusus penjajaran partikel untuk mencapai sifat likat anjal MRE yang dipertingkatkan. Oleh itu, kesan perubahan sifat MRE masih agak terhad dalam pelbagai peranti. Tambahan pula, perubahan arah daya ricih yang dikenakan pada MRE mungkin tidak menghasilkan kekakuan atau kesan MR yang maksimum pada MRE, kerana interaksi daya yang dikenakan dengan material tersebut hanya lebih berkesan pada satu arah penjajaran rangkaian partikel. Jadi dalam kajian ini, satu pendekatan yang menumpukan pada pelbagai penjajaran CIP dalam MRE diperkenalkan, iaitu penjajaran berupa struktur seperti pancutan air untuk menghasilkan pelbagai sudut susunan CIP dalam MRE. Kajian ini bermula dengan penghasilan acuan MRE yang dapat menghasilkan garisan-garisan fluks magnet pelbagai arah yang bertujuan menghasilkan struktur seperti pancutan air untuk membolehkan CIP mengikut arah fluks magnet tersebut semasa proses pembuatan MRE. Simulasi garisan fluks magnet tersebut dilakukan melalui analisa FEMM. Tiga jenis MRE telah dihasilkan, iaitu MRE isotropik, MRE seperti pancutan air dan MRE seperti pancutan air songsang. Sifat reologi MRE-MRE ini dari segi modulus penyimpanan dan kesan magnet reologi (MR) diukur dalam mod ayunan ricih menggunakan reometer pada pelbagai parameter berbeza seperti sapuan ricih, sapuan frekuensi dan sapuan medan magnet. Sementara itu, semua sampel MRE telah melalui analisa mikrograf menggunakan mikroskop elektron pengimbasan pelepasan medan (FESEM) untuk melihat struktur penjajaran CIP dalam MRE. Hasil kajian menunjukkan bahawa kedua-dua MRE seperti pancutan air mempamerkan modulus penyimpanan yang lebih tinggi berbanding MRE isotropik, dengan peningkatan kira-kira 0.06 hingga 0.1 MPa tanpa pengaruh medan magnet dan nilainya telah bertambah dengan pengaruh daya medan magnet. Secara khususnya, modulus penyimpanan MRE seperti pancutan air adalah lebih tinggi berbanding dengan MRE seperti pancutan air songsang. Namun begitu, kesan MR pada MRE seperti pancutan air songsang telah mengatasi MRE seperti pancutan air disebabkan oleh modulus penyimpanan awalnya yang lebih rendah. Sebaliknya, fenomena modulus penyimpanan yang lebih tinggi bagi MRE seperti pancutan air adalah disebabkan oleh penjajaran CIP yang terhimpit apabila daya ricih dikenakan pada sampel tersebut menyebabkan ia menjadi lebih kaku untuk menahan sebarang perubahan, berbanding dengan MRE seperti pancutan air songsang yang lebih merenggang dengan arah daya ricih yang dikenakan pada sampel tersebut. Penemuan menunjukkan bahawa MRE seperti air pancut mempamerkan tindak balas terbaik dalam aplikasi mod ayunan ricih, sama ada tanpa atau dengan pengaruh medan magnet, yang mana pendekatan novel ini berpotensi untuk digunakan untuk kaedah pembuatan in-situ bagi peranti MRE.

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LIST OF ABBREVIATIONS

CIP	-	Carbonyl Iron Particle
FEMM	-	Finite Element Magnetic Method
FESEM	-	Field Emission Scanning Electron Microscope
ht.	-	Height
ID	-	Inner Diameter
MR	-	Magnetorheological
MRE	-	Magnetorheological Elastomer
MREs	-	Magnetorheological Elastomers
OD	-	Outer Diameter
PU	-	Polyurethane
Ref.	-	Reference
RTV	-	Room Temperature Vulcanization

LIST OF SYMBOLS

δ	-	Minimal error
μ_r	-	Relative magnetic permeability
μ	-	Magnetic permeability
μ_0	-	Magnetic permeability in vacuum space
B	-	Flux density
δ	-	Phase angle
G'	-	Storage modulus
G''	-	Loss modulus
G^*	-	Complex modulus
$\tan \delta$	-	Phase angle
σ	-	Stress
ε	-	Strain
ω	-	Angular frequency
M_s	-	Magnetization saturation
vol.%	-	Volume percentage
wt.%	-	Weight percentage

CHAPTER 1

INTRODUCTION

1.1 Background of Research

Magnetorheological elastomer (MRE) is a polymer composite that consists of magnetically permeable particles distributed within a non-magnetic elastomeric matrix [1]. MRE exhibits rheological properties and offers variable stiffness, which can be controlled under the influence of external magnetic field. The changeable properties are attributed by the locked magnetic particles in the elastomer matrix that operatively respond to the applied magnetic field. The behaviour of fast responsiveness and changeability of its stiffness has rendered MRE that belongs to a group called smart material, particularly magnetorheological (MR) materials [2]. Possess such advantages, MRE has created wider application opportunities including semi-active vibration dampers, vibration isolators and sensors [3,4]. In the presence of magnetic field, changes in the viscoelastic properties of MRE are typically described by MR effect [2]. The effect is a behaviour that defines the changes in the storage modulus of MRE in response to the tuneable magnetic fields that against a set of specified strains [5]. MR effect of MRE is comparable depending on the composition of magnetic particles and matrix components, types of matrices, concentrations and sizes of magnetic particles, additives and types of curing process that simultaneously affect the resultant viscoelastic properties of MRE [6–9].

Two types of MRE are characterized by ways of magnetic particles disperse in the elastomeric matrix. The first dispersion is called as isotropic MRE, which can be identified by a uniform distribution of magnetic particles in an MRE. This kind of MRE can be prepared by curing the melt MRE in a mould without applying a magnetic field, thus the particles are uniformly dispersed in the cured matrix phase. Meanwhile, the second type of MRE is called as an anisotropic, represents by the aligned magnetic particles at a specific degree, in the MRE. The magnetic field that is applied during the

pre-structure or crosslinking process of MRE allows the particles inside the elastomeric matrix to align in a chain or columnar configuration, forming a chain-like structures according to the lines of magnetic field [5,10].

Generally, anisotropic MREs possess higher MR effect and wider magneto-induced modulus compared to the isotropic MRE [5,11–14]. It is due to smaller gap between the inter-particles that are arranged in an aligned manner, resulting the magnetic fluxes flow easily along the aligned particles in the anisotropic MRE [15,16]. This concept gives rise to the MRE to highly respond towards the applied magnetic field and subsequently enhance the stiffness of the MRE. The closer gap between the particles also offers a higher permeability for the magnetic flux to flow within the elastomeric matrix of the MRE [15,17]. On the other hand, in the presence of magnetic field, the aligned particles in an MRE are magnetically at the lowest energy state, making the attraction forces between the particles are at maximum strength [5,12,18–20]. This phenomenon in return has enhanced the capability of MRE to resist deformation when a shear force is applied onto it, or known as stiffness and reasonably, the storage modulus as well as MR effect of the anisotropic MRE increase. This respective behaviour has been supported by Yao et al. [6] who stated that the interaction forces between the magnetizable particles in the aligned structure has resisted more deformation when the MRE sample was magnetized and sheared.

Furthermore, the study was also paid particular attention to the magneto-induced modulus of the anisotropic MRE in which the corresponding behaviour was improved by changing the orientation angle between the particle chains, respective to the applied magnetic field. The result demonstrated that the highest magneto-induced of MRE was achieved at 30° of particles chain's angle, with the use of bigger particle sizes. Despite that, for the smaller size of the particles at below 10 μm , the magneto-induced modulus of MRE was noted higher at 45° [6]. Another study done by Boczkowska et al. [11] focussed on the polyurethane-based MREs that were fabricated with different angles of particles chain alignments. The result reported that the samples with 30° of particles chain alignment to the applied magnetic field (y-axis) exhibited the highest storage modulus compared to samples with 0°, 45° and 90°. The finding also demonstrated that the magneto-induced modulus as well as the MR effect of

MREs could be enhanced by manipulating different particle's chain alignments to some degree. The general reason of this phenomenon was related to the magnetized aligned particles that are normally have higher attraction and interaction magnetic forces between the particles could withstand greater deformation upon the applied shear stress. However, no detail analysis and mechanism have been carried out on the correlation between the aligned particles at variety of angles with the final enhanced properties of the MREs.

In addition, the storage modulus as well as MR effect of MRE was also affected by the direction of shear force towards the alignment of the particle's chain inside the MRE [21,22]. The works in investigating the response of MRE with respect to shear direction and alignments of particle's chains have recently become an interesting topic by researchers. For instance, Tian et al. [21] who focused on the viscoelasticity properties of MRE with 45° of iron particle's alignment stated that the movement of the rheometer plate (shearing mode) that could stretch the particle chains or vice versa has affecting the resultant storage and loss moduli of the material. In fact, both storage and loss moduli of MRE were achieved higher when the applied shear stress direction has crammed the particle chains as compared to the shear direction that stretched the particle chains. Besides, under the applied shear stress, the particle chains that were crammed along the shear direction would create more restrains on the matrix phase as the movement of the molecular chains was hindered by a higher density of the particles. This finding was also consistent with another work by Zhang et al. [16], who stated in a theoretical model that when the average distance between the sheared particles decreased, the resultant shear stress as well as the storage modulus of MRE increased.

Despite of many investigations to discover the most significant orientation of particles that results in higher performance of MRE, the inconsistency of shearing force that distributed in the MRE during the oscillatory shear mode should be highlighted as well. The homogeneity in shearing force distribution is presume to be an important factor to acquire maximum impact of the rheological properties of MRE, by considering both the orientations of the particle's chain and the shear force direction. Zhang et al. [22] have investigated the relationship between the orientation

of particles-chains and shear force directions in an MRE. Prior to the investigation, the sample was cut and repeatedly positioned in a rheometer symmetrically to ensure that the angle between the particles chain and shear path direction were in-line considering the shear path of a circular direction. This placement technique of the study highlighted that both parameters; particle alignments and shear force directions could be integrated by the oscillating plates and as a result, the MRE produced a consistency value of the storage modulus.

1.2 Problem Statement

One of the key factors to affect the performance of MRE is by manipulating the alignments of magnetic particles; CIPs in an MRE. The previous studies somehow showed the importance of having various particle-chain alignments in an MRE that could facing the changing directions of applied shear force in order to produce maximum and consistent value of storage modulus. However, most of the studies have been focused on the specific angle of the aligned particles to achieve the enhanced viscoelastic properties of MRE. Thus, its effect on the MRE's stiffness is still rather limited in various devices. Besides, in oscillatory shear mode application, the changes in directions of shear force could not result in maximum stiffness or MR effect of MRE since the interaction of the applied force with the material is advantageous only in one direction of the particle's chain alignment. Therefore, various angles of particle's chain alignment, known as fountain-like is introduced to accommodate the changing direction of shearing force in order to obtain homogeneous stiffness and provide further enhance the resultant MR effect of MRE.

Prior to the target, this work presents the opportunity to thoroughly investigate the correlation between particle's chain alignment and shear force direction towards the behaviour of MRE. Therefore, the study offers a fundamental knowledge in designing MR devices especially focusing on the appropriate particle's alignment for a specific application. This approach has potentially to be applied for the in-situ fabrication of MRE devices where the particles will be cured and aligned following the direction of magnetic field during the production process of the device. In fact,

prior to the in-situ fabrication, the interactions between the CIPs and magnetic fluxes upon exact application of the device would be further strengthened. Thus, it would result a big impact on the performance of the device in real application, where the manipulation of magnetic fields will be in-line with the locked magnetic particles in the MRE.

1.3 Research Objectives

The main objective of this research is to enhance the viscoelastic properties of MRE via modification of magnetic particle's alignment (CIPs). The primary objectives for this research are listed as follow:

- (a) To examine the configuration of curing mould for fountain-like magnetic flux flow in the MRE.
- (b) To characterize the resultant structure of MREs with various alignments of CIPs.
- (c) To analyse the storage modulus of MREs correspond to fountain-like alignments of CIPs, in an oscillatory shear mode test.

1.4 Research Scopes

The scopes of this research are specified on the investigation on the rheological properties of MREs respective to the alteration of magnetic particle's alignment. The scopes of the research include:

- (a) The fabrication of MRE samples using silicone rubber (SR) as a matrix medium and magnetic particle of CIPs, with a fixed ratio of SR to CIPs is 30:70 (wt.%).

- (b) The application of magnetic flux density at ~ 0.2 T across the mould during curing the MRE with fountain-like CIPs.
- (c) Morphological characterization of MRE samples with different CIP's alignments, including isotropic and fountain-like CIPs of MREs for Side-1 and -2, using field emission scanning electron microscope (FESEM).
- (d) Carry out the rheological test for viscoelastic properties of MRE samples in terms of storage modulus and MR effect correspond to sweep strains amplitudes, sweep frequencies and sweep magnetic fields, using a rheometer.
- (e) The rheological tests of MRE samples will be done under the absence (0 T) and presence of magnetic fields (0.1 - 0.6 T), in an oscillatory shear mode test, at room temperature of 25°C .

1.5 Research Outline

There are five chapters in this thesis. Each chapter highlights the relevant information, accomplishments, and research findings. The following is the outline for each chapter:

- Chapter 1 : The thesis is introduced in the first chapter. A research background, motivation of research, problem statement, research objectives and research scopes are all covered in this section.
- Chapter 2 : The second chapter is devoted to a literature review of MRE, focusing on the parameters that must be considered while fabricating and analyzing the MRE samples. There is a review of several fundamental studies that were relevant to the research topics, including the resultant rheological properties of MRE with different alignments of CIPs.
- Chapter 3 : The third chapter describes the research methodology and the experimental component. The research process is described in detail, step-by-step, in order to achieve the intended objectives. This

chapter also includes the development process of the fountain-like MRE, including the design of the curing device, sample preparation, characterisations and rheological testing procedures.

Chapter 4 : The results of the physical characterization and rheological properties of MRE samples are presented in the fourth chapter. Correlations between the storage modulus as well as MR effect of MRE towards the rheological measurements are discussed in terms of sweep strain input, sweep frequencies and sweep magnetic fields. This chapter also presents a possible mechanism that interprets the physical interaction of the sample during testing process that resulted in changeable properties of the MREs.

Chapter 5 : This final chapter summarizes the main achievements of the research. The achievement of each objective and contribution of the research are highlighted. Finally, some recommendations are presented as an extension of the existing research.

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