EFFECT OF THERMAL CYCLES ON TENSILE PROPERTIES OF NR/EPDM NANOCOMPOSITES FOR ENGINE MOUNTING

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2017

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This report is submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Engineering Materials) (Hons.).

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2017

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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SESI PENGAJIAN: 2016/17 Semester 2

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ABSTRAK

Laporan ini menumpukan kesan kitaran haba ke atas sifat tegangan adunan getah asli (NR)/ etilena-propilena-diena getah (EPDM) dan NR / EPDM yang diisi kepingan nano-zarah grafin (GNPs) untuk pemegang enjin yang kebiasanya mengalami kesan pemanasan dari enjin dalam perkhidmatan. Selain daripada sifat redaman, kekerasan dan tegangan, haba juga penting untuk menentukan sifat-sifat mekanikal pemegang engin. Kajian yang berkaitan dengan kesan kitaran haba ke atas sifat-sifat mekanikal bahan ini masih terhad. Campuran dan nanokomposit disediakan melalui penebatian lebur menggunakan peralatan pencampuran dalaman Haake pada suhu 110 °C, kelajuan rotor 40rpm dan dicampurkan selama 7 minit dan dimatangkan dengan mesin penekan panas pada suhu 150 °C. Campuran dan nanokomposit terdedah dua suhu pada 60 °C dan 120 °C dan disejukkan sehingga suhu bilik alternatif untuk jangka masa 10 minit dan diulangi untuk 0,35,70 dan 150 kali. Sifat tegangan NR/EPDM dan GNPs nanokomposit menunjukkan penurunan ~ 9% dan ~14% masing-masing pada 60 °C kitaran haba, manakala kedua-dua getah berubah secara drastik ke ~ 61% dan ~ 64% dari 5 sampai 35 kitaran haba pada 120 °C. Ia mengalami kemusnahan molekul disebabkan penyerapan tenaga haba semasa kitaran haba dan mengurangkan penghabluran daripada transformasi sifat bergetah ke sifat berkaca dan disokong dengan mengimbas mikroskopi elektron (SEM) dan X-ray pembelauan (XRD). Keputusan SEM menunjukkan campuran dan nanokomposit berubah sifat rapuh apabila ricih matriks menipis dan mengecilkan saiz permukaan. Jenis I mempaparkan penurunan pada pemanjangan takat putus (E_B) dan modulus pada 100% (M_{100}) secara beransur-ansur semasa kitaran suhu haba yang lebih rendah. Bagi jenis II keadaan patah mengalami penurunan drastik dalam E_B pada 5 sampai 35 kitaran haba dan sentiasa menurun pada M_{100} di suhu yang lebih tinggi. Keseluruhannya, GNPs dipengisi NR/EPDM berupaya mengekalkan kestabilan haba daripada kesan kitaran haba terutamanya untuk getah pemegang engin.

ABSTRACT

This report focusing on the effect of thermal cycles on tensile properties of NR/EPDM blend and GNPs filled NR/EPDM nancomposites for engine mounting which normally experience heating effect from engine in service. In this case, apart from damping, hardness and tensile properties, thermal also plays an important role to determine the mechanical properties of engine mounting. However, the study related to the effect of thermal cycles on mechanical properties of this material are still scarce. The blends and nanocomposites were prepared via melt compounding using a Haake internal mixer at a temperature of 110 °C, rotor speed of 40 rpm and mixed for 7 minutes and subsequently cured using a hot press machine at 150 °C. The blends and nanocomposites were then exposed to two different temperatures at 60 °C and 120 °C and cooled down to room temperature alternately for durations of 10 minutes and repeated for 0, 35, 70 and 150 times. The tensile behaviours of NR/EPDM blend and GNPs filled nanocomposites exhibited dramatically drop of ~ 9% and ~ 14% respectively at 60 °C thermal cycles, whereas both rubbers changed drastically down to ~ 61% and ~ 64% from 5 to 35 thermal cycles at 120 °C. It experienced molecular degradation due to absorption of thermal energy during the thermal cycles and decreased the crystallinity from transformation to rubbery to glassy behaviour and supported by scanning electron microscopy (SEM) and X-ray diffraction (XRD). From SEM result showed the blends and nanocomposites changes to brittle behaviour as the matrix shear yielding change thinner and the size of interface getting small. Type I fracture behaviour indicated decreased in elongation at break (E_B) and modulus at 100% (M_{100}) gradually at lower temperature thermal cycles. For type II fracture behaviour experienced drastic drop in E_B from 5 to 35 thermal cycles and decreased constantly in M_{100} at higher temperature. In overall, GNPs filled NR/EPDM are capable to sustain the thermal degradation from thermal cycles effect particularly for a mount rubber.

DEDICATION

Dedicated to

my beloved father, Lim Tang Lai

my appreciated mother, Lim Meng Chu

and my adored siblings Lim Ruoh Yih, Lim Hong Jie and Lim Ruoh Ing

for giving me moral support, cooperation, encouragement and also understanding

Thank You So much & Love You All Forever

ACKNOWLEDGEMENT

First of all, I would like to express my highest gratitude to my highest gratitude to my respected supervisor, Prof Madya Dr. Noraiham binti Mohamad for her advices, valuable informations and guidance, which helped me in completing the Final Year Project on time and fulfil the requirements.

Besides, I also would like to show appreciation to Miss Khairul Ilwani binti Karim, Graduate research assistance under Faculty of Manufacturing Engineering from Universiti Teknikal Malaysia Melaka (UTeM) for the research grant, PJP/2016/FKP/H16/S014848 to support my study. Thanks to UTeM for providing opportunity to me for pursue the final year project as a partial fulfilment of the requirement for the degree of Bachelor of Manufacturing Engineering.

To all my beloved friends, thank you so much for your support and encouragement. They had given me the inspiration for ideals and solution during this period. Thanks for the great friendship. I would like to express my sincere gratitude to everyone who has directly or indirectly contributed to this project.

Last but not least, I would like to show my deepest appreciation to my family members for their moral support, understanding and encouragement throughout this project.

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LIST OF ABBREVIATIONS

χ	-	Rubber Interaction Parameter
E_B	-	Elongation at Break
2D	-	Two Dimensional
6PPD	-	N-(1, 3-dimethylbutyl)- N’-phenyl-p-phenylenediamine
ASTM	-	American Society for Testing and Materials
Al_2O_3	-	Alumina
C_p	-	Heat Capacity
CRI	-	Cure Rate Index
CTE	-	Coefficient of Thermal Expansion
C=C	-	Carbon-carbon double bond
CV	-	Conventional Vulcanization
CO_2	-	Carbon Dioxide
CNT	-	Carbon Nanotube
GNPs	-	Graphene nanoplatelets
ENR	-	Epoxidized Natural Rubber
EPDM	-	Ethylene-propylene-diene Monomer
EV	-	Efficient Vulcanization
F1	-	NR/EPDM blends
F2	-	GNPs filled NR/EPDM nanocomposites
ISO	-	International Organization for Standardization
M_{100}	-	Modulus at 100%
M_{300}	-	Modulus at 300%
M_c	-	Molecular Weight between Crosslink
MBTS	-	2, 20-dithiobis (benzothiazole)
MH	-	Maximum torque
ML	-	Minimum torque
N&V	-	Noise and Vibration
NR	-	Natural Rubber
PEN	-	Polyethylene-2, 6-naphthalate

PP	-	Polypropylene
SEM	-	Scanning Electron Microscopy
SEV	-	Semi-efficient Vulcanization
T_{90}	-	Optimum cure time
T_g	-	Glass Transition Temperature
T_{s2}	-	Scorch time
TGA	-	Thermogravimetric Analysis
TMTD	-	Tetramethyl Thiuram Disulfide
TPE	-	Thermoplastic Elastomeric
Q_m	-	Weight Increase of The NR/EPDM Blends in Toluene.
V_c	-	Density of Crosslink
V_r	-	Volume Fraction of the Swollen Rubber
V_s	-	Rubber Density
W_0	-	Samples Initial Mass
W_1	-	Samples Final Mass
XRD	-	X-ray Diffraction

LIST OF SYMBOLS

g	-	gram
%	-	Percent
$	-	US Dollar
°C	-	Degree Celsius
a.u.	-	Arbitrary Unit
cm^3/mol	-	Cubic Meter Per Mole
g/cm^3	-	Gram Per Cubic Centimetre
GPa	-	Giga Pascal
µm	-	Micrometer
d	-	Diameter
K	-	Kelvin
L	-	Length
lbs	-	Pound
phr	-	Part Per Hundred Rubber
t	-	Thickness
TPa	-	Tetra Pascal
MPa	-	Mega Pascal
mg	-	Milligram
m^2g^{-1}	-	Square Meter Per Gram
mm/min	-	Millimeter Per Minute
N	-	Newton
N/m	-	Newton per meter
nm	-	Nanometer
W/m K	-	Watts Per Metre Kelvin
wt %	-	Weight Percentage

CHAPTER 1
INTRODUCTION

This chapter covers the background study, the problem statement, the objectives, the scopes of study and chapter overview.

1.1 Background Study

Engine mount is the component that support the engine to the car body or the engine cradle. Normally, the engine and transmission are locked together and held in place by 3 or 4 mounts. The mounts that holds the transmission are known as the transmission mount, others are introduced as engine mounts. Generally, engine mount is made from combination of rubber and steel. It also absorbs the road shocks and engine vibrations in order to keep apart both noise and vibration (N&V) acting on the driver and passenger. According to El-Sharkawy and Uddin (2016) mentioned that the engine mount is sensitive to temperature. The life span and performance of engine mount will be affected by different temperature acting on engine mount. The demand for high-performance engine mounting increases with the development of modern vehicles offering efficiency, economical and comfort. Hence, the perfect engine mount system should enhance the frequency and amplitude dependent properties, thermal stability and weight reduction (Vishwas & Ravi, 2016).

Among various type of rubber blend, vulcanized natural rubber with ethylene-propylene-diene monomer (NR/EPDM) based materials have attracted various researchers to further investigate and improve the formulation of NR/EPDM compounds (Razak *et al.*, 2015). This due to its outstanding performances in industrial application such as engine mounting. Various type of functional nanofillers has been added to NR/EPDM rubber to boost the added-value of blends and for the different purpose applications (Alipour *et al.*, 2011; Motaung *et al.*, 2008; Razak *et al.*, 2014).

Natural rubber (NR) is a natural biosynthesis polymer with excellent properties such as superior elasticity, high resilience, low level of strain sensitivity, fatigue resistance, and great processing characteristics. However, the highly unsaturated and chemically reactive of NR caused it to be highly sensitive to environmental factors include radiation, humidity, moisture, light and ozone. Consequently, it limits the utilization of NR for many superior performance industrial applications such as the high thermal resistance. The limitations of NR having poor resistance to chemicals and oil substances, the high susceptible to degradation and low ozone resistance, can be overcome by mixing NR with the low unsaturated rubber phase like EPDM.

Ethylene-propylene-diene (EPDM) is a saturated carbon-hydrogen polyolefinic rubber, acquired by polymerizing ethylene and propylene with little amount of a nonconjugated diene. It always acts as an impact modifier and the incorporation of this rubber phase in elastomer and thermoplastic elastomeric blends (TPE) convey excellent ageing properties including the resistances of thermal and chemical, weathering as well as oxidation. Unfortunately, the non-polar and unsaturated characteristics of this synthetic rubber induce major incompatibility and immiscibility problem of existence of EPDM in the formulation of rubber blends (Yaakub *et al.*, 2014). Hence, there are several established methods to improve the miscibility between EPDM and rubbers with success (Jones and Tinker, 1997; Yaakub *et al.*, 2014).

In this study, graphene nanoplatelets is selected as nanofillers into NR/EPDM rubber blends to produce a NR/EPDM nanocomposite. Graphene, a basic unit of graphene nanocomposites (GNPs), is a special monolayer of hexagon-lattice are stiffer and stronger than carbon nanotube (CNT) (Liu *et al.*, 2015). Just like CNT, the different types of graphene ribbon edges will affect the electronic properties of GNPs. GNPs is considered as the strongest material since it possesses a high elasticity modulus of around 0.5~1.0 TPa which is very near to the accepted value for bulk graphite with breaking strength to be approximately 40 N/m (Wang *et al.*, 2012). It can be elongated to a quarter of its original length. Apart from size and chirality dependence, temperature also plays the important role to determine the mechanical properties of graphene. According to Wang *et al.* (2012), Young's modulus of a graphene nanocomposite only influence significantly at the temperature of about 1200 K and afterwards graphene turns softer. At this point, the fracture strength and fracture strain reduce remarkable with rising the temperature.

1.2 Problem Statement

There are significant number of studies conducted to enhance the properties of engine mounting such as saving power consumption, isolated engine vibration and increase engine performance for high speed as well as extending the engine's life span (El-Sharkawy and Uddin, 2016; Ngolemasango *et al.*, 2008; Yu *et al.*, 2001). Some studies incorporate of graphene nanoplatelets into NR/EPDM blends to increase the heat dissipation (Razak *et al.*, 2015) and improve vibration damping (Valentini *et al*., 2016). Unfortunately, there are scarce research conducted to understand the effect of thermal cycles on mechanical properties of NR/EPDM nanocomposites especially for engine mounting. There are tensile loading acting on engine mounting from the vibration of engine when the vehicle is activated. In addition, the activated engine imposed constant heating on engine mounting. The standard temperature in an engine mount area are between -30 °C to 120 °C (Scott *et al.*, 2012). As the temperature of engine increases, the generated heat will be absorbed by the mounting hence lead to degradation. This research compare the degradation properties between NR/EPDM rubber blends and NR/EPDM filled GNPs nanocomposites under the influence of thermal cycles. Incorporation of GNPs in NR/EPDM rubber blends is hypothesis to exhibit numbers of improves properties including improvement in damping and heat dissipation performance as well as thermal conductivity which can extend the life span of engine mounting (Guo, 2000). These improvement can delay the degradation of NR/EPDM nanocomposites from thermal cycles. Despite the un-avoided occurrence of material degradation by thermal effect, the life of an engine mounting would be extended by adding GNPs. The good heat adsorption and faster heat dissipation may enhance the performance and prolong the life span of an engine mounting.

1.3 Objective

There are three objectives of this research as following:

(a) To determine the effect of thermal cycles on tensile properties of unfilled GNPs NR/EPDM blend and NR/EPDM filled GNPs nanocomposites

(b) To evaluate the failure behaviours of the NR/EPDM filled GNPs nanocomposites via morphological analysis, thermal analysis and compositional analysis

(c) To model the fracture behaviour of the NR/EPDM filled GNPs nanocomposites under low temperature (60 °C) and high temperature (120 °C) thermal cycles

1.4 Scope

This study focuses on the effect of 0, 5, 35, 70 and 150 thermal cycles on tensile properties of NR/EPDM nanocomposites for engine mounting under 60 °C and 120 °C. The nanocomposites were prepared via melt compounding and cured by hot press machine before undergoes thermal exposure and tensile testing. The morphology of fracture samples under tensile loading were then analysed. The tensile properties of NR/EPDM filled GNPs nanocomposites were evaluated by various analysis such as morphological, compositional and thermal analysis. The degradation rate for weight loss/time of NR/EPDM nanocomposites at different duration with constant thermal cycle was determined by using TGA. Next, the comparison of tensile strength, elasticity modulus, elongation at break and swelling rate between NR/EPDM blends and NR/EPDM filled GNPs nanocomposites was carried out. Lastly, model of failure behaviour of NR/EPDM filled GNPs nanocomposites under the thermal cycle effect was stipulated.

1.5 Chapter Overview

This report is divided into five chapters that introduce the reviews and describe the analytical and experimental research performed. Chapter 1 is an introduction to the study that brief about objectives, problem statement, significant of study and the thesis overview. Chapter 2 presents the literature review related to the theories on NR/EPDM composites of added of graphene and previous investigations on the issues and current prefers on the topics. The significant element that covered in this chapter is about the materials as well as processing involved and also linked with experimental testing. Chapter 3 provides details explanation on the methodology used for overall research work, raw materials, characterization of the materials, samples preparation and procedure, property analysis and testing. In Chapter 4, the result of the characterization and analysis on the properties for the NR/EPDM nanocomposites were explained in details. The Chapter 5 summarizes major findings and offers some concluding remarks. The recommendation for future projects is also included in the Chapter 5.