



**SURFACE MODIFICATION OF ALUMINIUM BY GRAPHENE
NANOFILLER IMPREGNATION FOR RUBBER-ALUMINIUM
BONDING SYSTEM**

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

by

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:

.....
(PROFESOR DR. QUMRUL AHSAN)

ABSTRAK

Tujuan kajian ini adalah untuk melaporkan demonstrasi teknik pengubahsuaian permukaan untuk mengfabrikasi Aluminium-Graphene Nanoplatelet (Al/GNP) sebagai nanokomposit dengan kaedah metallurgi serbuk yang dibantu oleh kaedah pengikisan geseran. Graphene adalah salah satu nanopartikel karbon yang dimanfaatkan kebanyakan penyelidik di atas sifat luar biasa mereka dalam alam untuk mengekstrak keupayaan peningkatan kekuatan matriks tersebut. Terdapat dua peringkat utama yang terlibat dalam kaedah permukaan geseran ini di mana peringkat pertama adalah pembuatan alat gabungan komposit Al-GNP dengan kaedah metallurgi serbuk dan kedua adalah penggunaan alat tersebut untuk memperoleh substrat Al-komposit melalui geseran permukaan. Hasilnya ditafsirkan dengan SEM bersama EDX spektrum analisis menunjukkan nanokomposit matriks logam yang disediakan oleh permukaan geseran telah memaruhkan secara berkesan ke dalam Al matriks semasa proses pengikisan geseran. Teknik pencirian SEM telah dilaksanakan untuk mengkaji permukaan morfologi dan kadar pengedaran GNP. Pelaksanaan kandungan graphene yang tinggi telah memperkenalkan ciri-ciri mekanikal yang optimum dengan penyebaran homogen graphene di dalam Al substrat pada kelajuan pusingan yang optimum bagi mata alat komposit semasa pengikisan geseran. Micro-kekerasan komposit yang terbentuk dijumpai meningkat dengan kandungan penggabungan GNP dalam sampel yang juga memberikan kuasa pengasingan yang lebih kuat dalam kajian pengasingan sesama Al dan getah jika dibandingkan dengan kandungan GNP yang lebih rendah dan dengan Al yang belum diubahsuai.

ABSTRACT

The aim of this study is to report the demonstration of surface modification technique in fabrication of Aluminium-Graphene Nanoplatelet (Al/GNP) composite utilizing powder metallurgy method assisted by friction stir surfacing. Graphene is one of the carbon nanoparticle that harnessed most of the researcher's interest on top of their extraordinary properties in nature to extract their strength enhancement capability. There were two main stages involved in friction surfacing (FS) method by which the first stage was the fabrication of Al-GNP consumable composite tool with powder metallurgy method and followed by the utilization of tool made to acquire a composite-impregnated Al substrate. The result interpreted from the SEM with EDX spectra analysis showed the metal matrix nanocomposite prepared by friction surfacing had impregnated effectively into Al matrix. Characterization technique of SEM was implemented to study the surface morphology of the surface composite on Al substrate and powder composite tool to see the distribution of reinforcement. Micro-hardness test was conducted and proved that modifying task with tool of highest 5 wt% GNP composition bring the highest hardness of 24.48 ± 3.85 HV to powder composite. The resulting modified aluminium was brought to adhere with uncured rubber in way of gaining the optimum rubber-metal bond. High graphene composition within the powder composite had proved to increase the maximum peeling force between rubber and metal during bond peel test by obtaining 9.54 kJ/m^2 nominal peeling energy as compare to Al substrate with lowest GNP composition (8.52 kJ/m^2) and the unmodified Al (5.85 kJ/m^2).

DEDICATION

Only

my beloved father, Wong Siew Huey

my appreciated mother, Chong Choy Wan

my adored brother, Wong Ding Jiu

for giving me moral support, money, cooperation, encouragement and also understandings

Thank You So Much & Love You All Forever

.

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LIST OF ABBREVIATIONS

Al	-	Aluminium
GNP	-	Graphene Nanoplatelet
CNT	-	Carbon Nanotube
GNS	-	Graphene Nanosheet
VOC	-	Volatile Organic Compound
MMC	-	Metal Matrix Composite
SEM	-	Scanning Electron Microscopy
XRD	-	X-Ray Diffraction
FS	-	Friction Stir Surfacing

CHAPTER 1

INTRODUCTION

This chapter discusses the background study, problem statements, objectives and research scope of this project.

1.1 Research Background

In the recent industrial applications, there are many situations that should involve the adhesion or interaction between more than one material in order to achieve a higher functionality and usability and yet trigger contribution of those materials for advanced development. The interaction or molecular adsorption between the materials is the main agent that foster adhesion while those intermolecular interactions mostly include the ionic, covalent or Van der Waals forces of attraction which will lead to the rubber bondability with different substrates. The surface tension of a liquid and the solid surface energy will be the elements that affecting rubber adhesion strength with other materials (Payam *et al.*, 2012). There are several theories used in the rubber adhesion mechanism which include mechanical interlocking theory, electrostatic, diffusion, adsorption, and acid-base theory. Also by now, there are several applications that need adhesion of rubber with other types of substrate material for various purposes. Luda *et al.*, (2003) proposed the used of ethylene-propylene rubber (EPR) in polypropylene matrix in car bumpers to enhance a great impact strength,

ductility, mechanical properties and durable adhesion between them. Figure 1.1a and 1.1b show the adhesion theory of adsorption with good and poor wetting conditions.

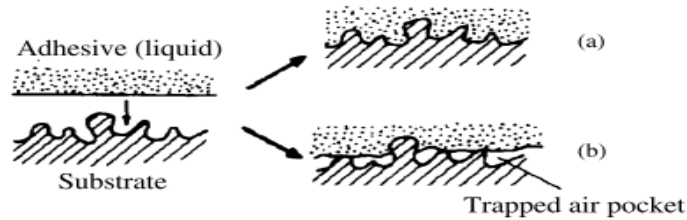


Figure 1.1: Mechanism of adsorption with (a) good and (b) poor wetting (Iraj *et al.*, 2012)

From the perspective of industrial and practical, adhesion of rubber to different substrate has a significant role in recent development especially with metal substrate mainly on automotive industry (Ansarifar *et al.*, 2001). According to Iraj *et al.* (2012), when rubber is to adhere with a solid state, the parameters that should be concern are mainly on their interfacial interaction, cross-linkage and entanglement of rubber chain in its adhere mechanism. Rubber to metal bonding is use for typical application consisting of parts requiring both the combination of metal stability and rubber flexibility. In some cases, rubber to metal bonding is used to reduce sound and vibration (Wen-Bin *et al.*, 2017). Traditionally, industry had implemented the bonding of rubber with steel for many fixation purposes but in recent world aluminium is gradually replace steel due to their lower cost, lightweight, high strength-to-weight ratio and better mechanical properties for many applications like engine mounting or damping purpose as the incremental on demand and consumption of energy put much restrictions on heavy component. Due to their great importance in automotive industry, the effort on ensuring a better adhesion between rubber and aluminium always be the prior concern and hence make up this research topic. Below is the schematic diagram (Figure 1.2) illustrate the complex mechanism of adhesion between rubber and metal mostly involving diffusion, chemisorption, inter/intra-crosslinking, adsorption or wetting and also inter-diffusion between rubber, metal, primer and adhesive. In this theory, the adhesive and adherend will having an interfacial interaction when they contact with each other and there will be a great adhere effect when the surface molecule of solid intimately interact together

that causing inter-diffusion to occur at the molecular level within primer, adhesive and rubber substrate.

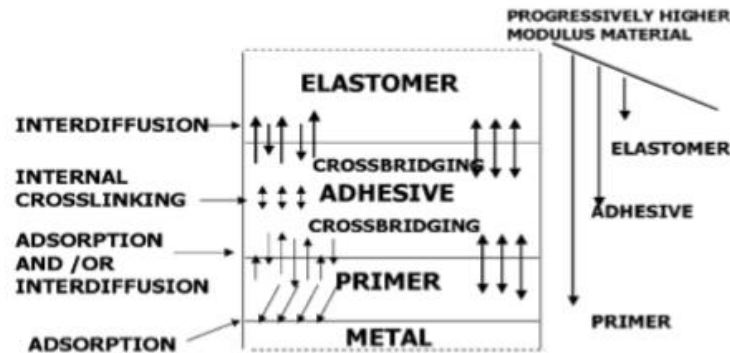


Figure 1.2: Interfacial interaction of rubber-to-metal bond (Polaski *et al.*, 2005)

One potential and promising way to improve mechanical properties of aluminum and their adhesiveness with rubber is by the fabrication of surface nanocomposite. This surface composite will be made by reinforcing nano-scale particles or filler into aluminium matrix to tailor industrial needs for numerical mechanical components. This formation of surface nanocomposite not only refines a better microstructure but also hinder dislocation to trigger greater bond strength.

There are various types of carbon-based nanomaterial that can be a choice of candidate as reinforcing filler. However, graphene with its most outstanding properties of tensile strength up to 130 GPa and Young's modulus of 1 TPa attract most current researchers towards them (Lee *et al.*, 2011). Graphene is an sp^2 hybridized two-dimensional carbon atom densely packed in a honeycomb crystal lattice structure. Due to their multiple wrinkled structures and ultrahigh strength of graphene nanoplatelet up to 1060 GPa, the resulting metal matrix composite would have prepared an increased strength and ductility (Ma *et al.*, 2018) providing an area-to-area contact between the matrix and their reinforcement which makes the load transfer easier and hence increase strength (Horsell *et al.*, 2008).

Overall, the rubber-metal bond has significant importance in many potential applications such as engine mounting, solar panel, and heat absorber. However, in way of

enhancing a great and durable bond between rubber and metal, research will be done on the reinforcement of nanoscale particles impregnated into the metal matrix.

1.2 Problem Statement

Modern industrial application demands have led to the development of new generation of rubber usage compare to traditionally with better enhancement of wear, impact, corrosion and heat resistance. This would require the interaction of rubber compound with other substrates. Hence, the maximum adherence between rubber and its substrate had dragged interest of scientists to study on.

Conjunctionally, studies regarding ways to promote better adhesion between rubber and metal being implemented in various industries. The researchers' view is to report the best way to improve adhesion strength but this will indirectly put a cumbersome procedure and precaution during operations in order to achieve an expected outcome. There are thousands of ways to foster durable, high strength bonding with the most significant way is by preparing the substrate surface to get an active interface for linkage purpose. Iraj *et al.* (2012) proposed that the surface energy of substrate should be greater than liquid adhesive for them to spread throughout the surface of a solid substrate. The surface preparation precaution took by a variety of industries includes mechanical treatment, chemical oxidation, anodization and phosphatization before application of primer and adhesive to interlock between rubber and their substrates. Primer plays role on such combination by adjusting the surface free energy for better wetting effect, triggering a chemical reaction of adhesive and adherend, inhibiting corrosion of substrate during service life and as an intermediate film to improve bond strength. The dry film thickness that is recommended for high optimum end performance would be within 0.2 to 0.4 mil for primer and 0.5 to 1.0 mil for adhesive (Warren *et al.*, 2005). Combination of above statement are time-consuming that lower down the industrial cycle time leading to the introduction of nanofiller in this bonding relationship.

The acquisition of well- developed adherence between rubber and metal sufficient for most automotive component. However, for advanced mechanical spare part that involves cyclic movement will be having high potential to break apart as in engine mounting device involving shear force. The movement performs in such a way that they are un-resisted to directional changes and damping with speed frequency variation. According to Owolabi *et al.* (2006), a component that is being subjected to cyclic loading, failure will occur on the part with crack spread through the surface that generally initiated from the structural discontinuity notch as in the adhere joint surface between rubber and metal. Hence, there should be an alternative solution to aid as a supportive structure on the applications of adhesive and primer to reduce the area of the discontinuous notch.

Recent worldwide industries that involve usage of primer and adhesive are intended to meet the volatile emission standard for volatile organic compound (VOC) that is set by the government (Karl *et al.*, 2007). Some company had installed incinerators in helping conversion of resulting solvent into an acceptable form. However, chlorinated solvent is still needed to control the rate of evaporation and rheology of adhesive and primer. Their presence having a potential to convert into hydrochloric acid that would reduce the function of incinerator by referring to Polaski *et al.* (2005). On top of their long-term exposure risk on adverse health effects such as eyes, nose irritation, liver, kidney and central nervous system damage, level of VOC is always control by government. The research study by Low *et al.* (1998) indicated that the inhalation of specific VOC associates with health risk that indicates a chronic health effect. Hence, it is necessary to figure out a new alternative to improve the rubber-metal adhesion as to minimize the use of primer and adhesive on account of VOC reduction.

To compensate all those issues, study on the fabrication of metal matrix nanocomposite is compulsory. A tremendous effort had devoted as to study the enhancement of improved bonding strength between rubber and metal in revealing the mechanism of surface modification of aluminum to its entirety.