IMPROVING RUBBER-TO-METAL BONDING THROUGH THE USE OF SILANE COUPLING AGENT

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

by

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Sesi Pengajian: **2019/2020 Semester 1**

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Tajuk PSM: Improving Rubber to Metal Bonding Through The use of Silane Coupling Agent

Nama Syarikat: HML Auto Industries Sdn.Bhd.

Sesi Pengajian: 2018/2019

Adalah saya dengan ini memperakui dan bersetuju bahawa Projek Sarjana Muda (PSM) yang bertajuk seperti di atas adalah merupakan satu projek yang dijalankan berdasarkan situasi sebenar yang berlaku di syarikat kami sepertimana yang telah dipersetujui bersama oleh wakil syarikat kami dan penyelia serta pelajar dari Fakulti Kejuruteraan Pembuatan, Universiti Teknikal Malaysia Melaka yang menjalankan projek ini.

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I hereby, declared this report entitled "Improving Rubber-to-Metal Bonding Through the use of Silane Coupling Agent" is the results of my own research except as cited in references.

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfillment of the requirement for the degree of Bachelor of Manufacturing Engineering (Hons.). The members of the supervisory committee are as follow:

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(**DR. CHANG SIANG YEE)**

ABSTRAK

Pada masa kini, silana digunakan secara meluas untuk bon dua bahan yang tidak serupa seperti getah-logam. Tujuan kajian ini adalah untuk menilai kaedah untuk meningkatkan ikatan kimia antara aluminium-to-getah dengan menggunakan bis-(trimethoxysilylpropyl) amina dan bis-(triethoxysilylpropyl) tetrasulfide. Objektif kajian ini adalah untuk mengkaji kesan teknik permohonan silana yang berbeza pada kekuatan ikatan antara muka getah logam dan menyiasat kesan komposisi penyelesaian silana pada kekuatan ikatan antara muka getah logam. Bahan yang digunakan terdiri daripada aluminium 6061-T6, dan getah asli, manakala bahan kimia adalah dua jenis silana, metanol, aseton, larutan alkali, asid asetik dan natrium hidroksida. Kekasaran permukaan aluminium diubahsuai oleh letupan pasir kemudian diikuti oleh alkali dirawat dengan hasil akhir 3.943µm. Hasil XRD telah membuktikan hidroksida pada permukaan aluminium selepas alkali dirawat. Penyelesaian silana bercampur dalam tiga komposisi yang berbeza dan diuji bawah FTIR. Dalam hasilnya, kuasa kulit maksimum HML Auto Industries yang dijalankan dengan CHEMLOK adalah 323.671N dengan tenaga mengelupas nominal 12,947 KJ/mm^2. Selain itu, teknik memberus adalah digalakkan untuk memohon silana pada substrat aluminium antara penyemburan dan mencelup. Manakala 1-1 tetrasulfide-amino campuran silane mempunyai kekuatan tertinggi melekat dalam ikatan 34.084N dan tenaga mengelupas nominal 1,605 KJ/mm^2. Kesan komposisi campuran silana tidak disediakan kekuatan ikatan yang tinggi pada antara muka getah logam kerana ia digalakkan ikatan tidak stabil dengan hasil FTIR dengan puncak kecil dalam kumpulan memfungsionalisasikan. Kesimpulannya, penggunaan ejen gandingan silana dalam aluminium-to-getah tidak mengatasi penggunaan pelekat biasa CHEMLOK.

ABSTRACT

Nowadays, the silane coupling agent had been widely used to bond the two dissimilar materials such as rubber-to-metal. The aim of this study is to evaluate the methods of improving the chemical bonding between aluminum-to-rubber by using the bis-(trimethoxysilylpropyl) amine and bis-(triethoxysilylpropyl) tetrasulfide. The objectives of this work are to investigate the effect of different silane application techniques on the bond strength of rubber-metal interface and investigating the effect of composition of silane solutions on the bond strength of rubber-metal interface. The materials used in this research were the aluminum 6061-T6, and natural rubber, where else the chemicals used were the two types of silanes, methanol, acetone, alkaline-cleaned solution, acetic acid and sodium hydroxide. The surface roughness of aluminum was modified by sand blasting then followed by alkaline treated with final result of 3.943µm. The XRD result has proven the presents of hydroxide element on surface of aluminum substrate after alkaline treated for bonding purpose. Two types of silane solutions were mixed in three different compositions and tested under the FTIR for the chemical structure. In the control sample result, the maximum peel force of HML Auto Industries Sdn Bhd that carried out with the CHEMLOK 205 and CHEMLOK 220 was 323.671N with the nominal peeling energy of 12.947 KJ/ mm^2. Besides, brushing technique is promoted to apply the silane on aluminum substrate among spraying and dipping. The result showed that the 1 to 1 tetrasulfide to amino silane mixture has the highest adhesion strength in bonding with 34.084N and nominal peeling energy of 1.605 KJ/ mm^2. However, the effect of composition of silane mixture were not provided the high bond strength on the rubber-metal interface as it promoted unstable bonding by the FTIR result with the small peak in functionalize group. As a conclusion, the use of silane coupling agents in aluminum-to-rubber had not overcame the use of ordinary adhesive of CHEMLOK.

DEDICATION

Only

my beloved father, Tan Ling Soon

my appreciated mother, Yung Chung Ee

my adored sister and brother, Simon and Sharon

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

Thank You so much & love you all forever

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LIST OF ABBREVIATION

AFM	-	Atomic Force Microscope
Al	-	Aluminum
APS	-	3-(Triethoxysilyl)-propylamine
ASTM	-	American Society for Testing and Materials
BTSE	-	1.2 bis (triethoxysilyl) ethane
CR	-	polychloroprene
CS	-	Carbon Steel
EGMA	-	ethyleneglycol monoacetate
ETOH	-	Ethyl alcohol
FTIR	-	Fourier Transform Infrared Radiation
IR	-	polyisoprene
NBR	-	nitrile
NR	-	Natural Rubber
OM	-	Optical Microscope
pH	-	Potential of Hydrogen
SBR	-	styrene-butadiene
SEM	-	Scanning Electron Microscope
THF	-	toluene
VS	-	vinyltrimethoxysilane

LIST OF SYMBOLS

°C	-	Degree Celsius
K	-	Dielectric Constant
F	-	Feed Rate
N	-	Force
K_{IC}	-	Fracture Toughness
GPa	-	Giga Pascal
HB	-	Hardness Brine
J	-	Joule
MPa	-	Mega Pascal
m	-	Meter
µm	-	Micrometer
ml	-	Milli liter
mm	-	Millimeter
Min	-	Minutes
%	-	Percentage
n	-	Refractive Index
s	-	Second
rpm	-	revolution p

CHAPTER 1

INTRODUCTION

This chapter introduces the subject matter and problems being studied and indicates its importance and validity. The chapter comprises of research background, problem statement, objectives, and research scope of the work entitled “Improving rubber-to-metal bonding through the use of silane coupling agent”.

1.1 Research Background

Rubber-to-metal bonding is a conventional expression covering various related procedures of the mechanical and chemical bonding. The rubber reinforced units that outcome from the procedure are utilized for the segregation of commotion and vibration in car and designing applications.

The Figure 1.1 showed the strut mount from the HML Auto Industries Sdn Bhd. Strut mounts is one of the automotive parts that used the rubber and metal to combine them together. It can also be defined as one of the most important parts in the vehicle’s suspension system. They are exerting to a heavy vibration during their lifespan and like any rubber-to-metal bonded parts, they are susceptible to the wear over time (MONROE Shock Absorbers 2018).

Figure 1.1: Car Strut Mount

Over the past decade, there has been an increasing emphasis on rubber-to-metal bonding through various type of bonding mechanism. Reliable rubber-to-metal bonding is important for various automotive parts such as engine mounting, strut mount, and rubber bushing etc. Basically, rubber-to-metal bonding can generally be classified into two variants. There are mechanical bonding and chemical bonding. Mechanical bonding basically involves providing a rough surface texture on the metal surface and incorporation of holes, ribs or cuts on the metal parts to promote bonding. While chemical bonding refers to the use of adhesive and coupling agent to enhance interfacial adhesion. Among these two techniques, chemical bonding has proved to be more effective than the mechanical bonding that had been widely used technique to bond rubber-to-metal parts in recent. The use of coupling agent such as silane has proved to be effective in bonding natural rubber with various metallic materials such as steel type (Van Ooij, 2018).

Regarding the rubber-to-metal that used the mechanical bonding of processing the strut mount, it applied the primer and adhesive as a type of linking rubber and metal together. The adhesive used is generally CHEMLOK 220 and the primer used is CHEMLOK 205 for the bonding between natural rubber and aluminum. They are uniformly sprayed onto the metal parts. Once the part is ready for rubber over molding, the metal parts are inserted into the mold cavity then followed by compressed with the rubber. As the elevated molding temperature cures the natural rubber into the vulcanized rubber, it activated the mechanical bonding locking with the adhesive on the surface between rubber-to-metal (Martin Rubber Company, 2018).

According to the Senthil *et al.* (2012), silane coupling agent is a type of chemical bonding that have been used for improving the adhesion strength between inorganic fibers and polymer matrices in many composite applications as well as in many paint-to-metal adhesions systems. The adhesion of sulfur-vulcanized rubber-to-metal has not been adequately supported by the past research. However, the preliminary investigations on the mechanism of adhesion are reported the effectiveness of the silane coupling agent in bonding the various rubber type to metal steel. According to the Sang *et al.* (2017), initially, the use of one silane solution is implemented into the research such as 3-(Triethoxysilyl)-propylamine (APS) silane is proved to be effective in improving the bond strength between the natural rubber and carbon steel. However, based on Van Ooij *et al.* (2002), it stated that in the latest research, the silane has been found to improve the adhesion between a metal substrate and a polymer layer by using the combination of two silane solutions with organic and inorganic silane type. For example, the combination of non-organofunctional silane of 1,2 bis (triethoxysilyl) ethane (BTSE) and organofunctional silane of vinyltrimethoxysilane (VS) may provide improved adhesion strength between a metal substrate to a peroxide-cured rubber, noted that the metal substrate is mainly for the cold roll steel (CRS). However, these same silane solutions were found out not being used when replacing by other type of rubber such as the sulfur-cured rubber. Thus, there is a need for the research of improving the methods of generating the adhesion between a metal substrate and a polymer layer.

In short, the usage of silane coupling agent has been appeared in the industry field nowadays for the purpose of research about the dissimilar materials bonding as it provided the chemical bonding between the organic and inorganic material. The chemical bonding has the stronger bond compared to the mechanical bonding as the chemical bonding involve the changing in the molecules structure while the mechanical bonding involves only the surface part but not the internal molecule structure.

1.2 Problem Statement

The material used by the HML Auto Industries Sdn Bhd in strut mount is natural rubber and aluminum 6061 type. This is because natural rubber is a material that has a high tensile strength and it is resistant to the fatigue from wear such as chipping, cutting or tearing (Labbe 2004). The aluminum 6061 type is used as it is lighter than the steel material in the automotive industry. For the current situation, the usage of the ordinary adhesive for the two dissimilar materials to bond together caused the failure of the parts after a short period of time. Ordinary adhesive involves only the surface part of the material which does not provide a good adhesion strength for the bonding. Therefore, the problem is needed to be solved by adding another type of adhesive which can give the better bonding.

CHEMLOK 220 adhesive is a cover coat adhesive designed for use over CHEMLOK 205 primer. This adhesive system will bond a wide variety of elastomers such as natural rubber (NR), styrene-butadiene (SBR), polychloroprene (CR), nitrile (NBR) and polyisoprene (IR) to various metals and other rigid substrates during vulcanization of the elastomer. It is composed of a mixture of polymers, organic compounds and mineral fillers dissolved or dispersed in an organic solvent system (LORD technical Data 2012). However, the use of CHEMLOK as the primer and adhesive were not improving the bonding between the rubber and metal as it does not occur any chemical bonding which strengthen the interfacial bonding between them. The mechanical bonding is just taking place on the surface of the metal and rubber without modifying the bond structure. Therefore, it resulted in the removal of rubber part from the aluminum part of the strut mount when serious vibration or damping occurs after a period which shown in the Figure 1.2.

Figure 1.2: Removal of rubber part from aluminum of strut mount

Based on the Auto Industries Sdn Bhd, cover coated adhesive CHEMLOK caused the failure of the parts between rubber-to-metal interfacial after a period of time when expose to the vibration and heat. To improve the bonding between the rubber and metal parts in automotive strut mount application, the traditional method of applying the primer and adhesive are replaced by adding the silane coupling agent.

Hence, there is a need to go for chemical bonding such as adding the coupling agent onto the metal parts. By using the coupling agent, the bonding between the rubber-to-metal changed from mechanical bonding to chemical bonding which can change the interfacial structure between the rubber-to-metal bonding and improving the bond strength between them. According to the Senthil *et al.* (2012), the combination of bis-(trimethoxysilylpropyl) amine and bis-(triethoxysilylpropyl) tetrasulfide have proven to be effective with the bonding between the sulfur-cured rubber and CRS. However, there was no significant research regarding the uses of these combination of these silanes to bond with the aluminum substrate. Based on the Jing Sang *et al.* (2017), it stated that the APS is proved to be functionalize between the natural rubber and carbon steel (CS).

Therefore, the research is moving forward towards using the two types of organofunctional and non-organofunctional silanes to show the hypothesis of the improvement the bonding between the aluminum substrate and vulcanized rubber.