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THERMOMECHANICAL PROPERTIES OF SHAPE MEMORY EPOXY FILLED NANOCLAY / CARBON NANOTUBE HYBRID COMPOSITES FOR AIRCRAFT MORPHING WING APPLICATION

MUHAMAD HASFANIZAM MAT YAZIK

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By

MUHAMAD HASFANIZAM MAT YAZIK

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Shape memory material is a smart material that can be programmed to a temporary shape and recovered its permanent shape when stimulated. Utilising the shape memory polymer for aerospace structure application such as morphing wing is a promising concept due to its versatility and functionality. However, pure shape memory polymer has a lot of drawbacks that limit its usage. In this study, shape memory nanocomposite was fabricated and characterised to determine the suitable material for aerospace structures. Three different loadings of nanoclay consisting of 1%, 3%, and 5% by weight percentage, three different loadings of Carbon Nanotube consisting of 0.5%, 1.0%, and 1.5% by weight percentage and hybrid loading of the same weight percentage were applied in preparation of the nanocomposites with epoxy as the polymer matrix. Single filler nanocomposite shows superior performance for 3% nanoclay and 1.0% Carbon Nanotube. For hybrid nanofiller, the glass transition temperature (T_g) of nanocomposite decreases as the nanofiller increase. The thermal properties of shape memory epoxy nanocomposite show a decrease in thermal degradation as the filler loading increase above 3% for nanoclay and 1.0% for Carbon Nanotube. For both tensile and flexural test at elevated and room temperature shows that the modulus increases as the hybrid filler increase and decrease as the loadings increase above 3% nanoclay and 1% Carbon Nanotube. The difference in modulus at elevated and room temperature shows the effect of molecular mobility above T_g as the shape memory epoxy is in the rubbery state. Thermal actuation of the hybrid nanocomposite shows increasing recovery rate as the nanofiller content increase. Generally, hybrid filler of 3% nanoclay and 1.0% Carbon Nanotube produces the best overall properties among the nanocomposites and has the potential be used for aerospace morphing structures. This study contributes to the development of hybrid nanofiller in shape memory polymer to understand the behaviour of shape memory epoxy nanocomposite.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
Sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

SIFAT TERMOMEKANIK KOMPOSIT NANO EPOKSI INGAT BENTUK DIISI DENGAN TANAH LIAT NANO/ TIUB KARBON NANO UNTUK APLIKASI SAYAP KAPAL TERBANG MORFING

Oleh

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Polimer ingat bentuk adalah bahan pintar yang boleh diprogramkan ke bentuk sementara dan dipulihkan ke bentuk kekal apabila dirangsang. Menggunakan polimer ingat bentuk untuk struktur aeroangkasa seperti sayap morphing adalah konsep yang mempunyai potensi yang serba boleh dan pelbagai fungsi. Walau bagaimanapun, polimer ingat bentuk tulen mempunyai banyak kelemahan yang menghadkan penggunaannya. Dalam kajian ini, rekaan dan pencirian komposit nano polimer ingat bentuk dijalankan untuk menentukan bahan yang sesuai untuk ultrasonikasi untuk mencipta komposit nano polimer ingat bentuk. Tiga berat tanah liat nano yang berbeza iaitu peratusan 1%, 3%, dan 5% mengikut berat, tiga berat yang berbeza dari tiubkarbon nano iaitu peratusan 0.5%, 1.0%, dan 1.5% mengikut berat dan isian campuran mengikut berat yang sama digunakan dalam penyediaan komposit nano dengan epoksi sebagai polimer. Komposit nano berisi tunggal menunjukkan prestasi cemerlang untuk 3% tanah liat nano dan 1% tiubkarbon nano. Manakala untuk komposit nano berisi campuran suhu, peralihan kaca (Tg) berkurangan dengan peningkatan isian nano. Ciri-ciri haba komposit nano epoksi ingat bentuk menunjukkan penurunan dalam degradasi haba apabila peningkatan isian nano meningkat melebihi 3% untuk tanah liat nano dan 1.0% untuk tiubkarbon nano. Untuk kedua-dua ujian tegangan dan lenturan pada suhu tinggi dan suhu bilik menunjukkan peningkatan modulus apabila pengisi campuran bertambah kemudian berkurang apabila beban melebihi 3% tanah liat nano dan 1% tiub karbon nano. Perbezaan dalam modulus pada suhu tinggi dan suhu bilik menunjukkan kesan pergerakan molekul di atas Tg ketika epoksi ingat bentuk berada dalam keadaan getah. Ransangan haba komposit nano campuran menunjukkan peningkatan kadar pemulihan apabila kandungan isian nano meningkat. Secara umumnya, pengisi campuran 3% tanah liat nano dan 1.0% tiubkarbon nano menghasilkan sifat terbaik di kalangan komposit nano dan mempunyai potensi digunakan sebagai struktur aeroangkasa. Kajian ini menyumbang kepada pembangunan

isian nano campuran polimer ingat bentuk untuk memahami tingkah laku komposit nano epoksi ingat bentuk.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philisophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

ASTM	American Society For Testing Materials
CMC	Ceramic Matrix Composites
CNT	Carbon Nanotube
CRG	Cornerstone Research Group
CY	Cyanate
DC	Direct Current
DMA	Dynamic Mechanical Analysis
DSC	Differential Scanning Calorimetry
EP	Epoxy
E’	Storage Modulus
E’’	Loss Modulus
FBG	Fibre Bragg Grating
FESEM	Field Emission Scanning Electron Microscope
FTIR	Fourier Transform Infrared Spectroscopy
ISO	International Organization For Standardization
MMC	Metal Matrix Composites
MMT	Montmorillonite
MWCNT	Multi-Walled Carbon Nanotube
NGDE	Neopentyl Glycol Diglycidyl Ether
PI	Polyamide
PMC	Polymer Matrix Composites
PUR	Polyurethane
PP	Polypropylene
PS	Polystyrene
RTM	Resin Transfer Moulding
SM	Shape Memory
SMA	Shape Memory Alloy
SMC	Shape Memory Ceramic
SME	Shape Memory Effect
SMEP	Shape Memory Epoxy
SMEPC	Shape Memory Epoxy Nanocomposite
SMP	Shape Memory Polymer
SMPC	Shape Memory Polymer Composite
SEM	Scanning Electron Microscopy
SWNT	Single-Walled Carbon Nanotube
TGA	Thermo-Gravimetric Analysis
Tg	Glass Transition Temperature
UAV	Unmanned Aerial Vehicle

LIST OF SYMBOLS

^{o} C	Degree Celsius
^{o} C/min	Degree Celsius per Minute
%	Percentage
Å	Angstrom
σ	Stress
ε	Strain
Ωm	Ohm metre
cm	Centimetre
mm	Millimetre
GPa	Giga Pascal
g/cm^3	Gram per Metre cube
h	Hour
kg/m^3	Kilogram per Metre cube
kPa	Kilo Pascal
kV	Kilo Volt
m^2/g	Metre square per Gram
mA	Milli Ampere
mbar	Millibar
mg	milligram
min	Minute
mm/min	Millimetre per Minute
MPa	Mega Pascal
mPa.S	Milli Pascal Second
N/m	Newton per Metre
nm	Nanometre
TPa	Tera Pascal
S/m	Siemens per Metre
s	Second
µm	Micrometre
V	Volt
W/mK	Watt per Metre per Kelvin
wt %	Weight Percentage

CHAPTER 1

INTRODUCTION

1.1 Overview

Morphing aircraft is a concept in an attempt to mimic the flight of a bird. A morphing aircraft can be defined as an aircraft that can significantly change configurations to maximize its performance at different flight conditions. This concept had already been applied by the Wright brothers to their airplane in their early flying days. In 1903, the Wright brothers employed wing warping with active camber and twist change for aircraft control. However, an increase in the payload and the speed of a fixed-wing aircraft caused the flexible wing to be replaced by a stiff structure to withstand the large aerodynamic forces and to prevent aeroelastic phenomena (Pendleton, 2000). Wings build are more rigid and control surface has become discrete, mechanical, and discontinuous as seen in most aircraft nowadays. A fixed-wing aircraft now are only optimized to have maximum performance at a single flight condition and acceptable performance at different flight conditions according to the aircraft's mission.

An ideal morphing application of an aircraft with a contradicting mission requirement would involve a sudden or significant configuration change in an efficient and reversible manner. A significant amount of researches has been devoted into developing morphing aircraft as evidenced by a few research collaborations over the past decades from a large-scale morphing such as wing sweep of the F-111 Aardvark (1964) and F-14 Tomcat (1970) (Min et al., 2010) to a small-scale morphing such as control surface deflection on the F-16 Falcon (1974) and AAW Program of NASA (2002) (Pendleton et al., 1996). Most of the research conducted previously focused mainly on developing a morphing enabler by mechanical devices. This mechanism comes with a weight penalty due to the additional structures required. To overcome this drawback, smart materials were proposed which showed a promising potential for morphing applications (Perkins et al., 2004; Yin et al., 2008; K. Yu et al., 2009). Various research has been conducted to explore the potential of smart materials for morphing skin. These include materials like elastomer, flexible matric composite, and shape memory materials (Gross & Weiland, 2007; Khan, 2008; Kudva, 2004; Murray et al., 2010; Thill et al., 2008). There are a number of properties required for a material that is suitable for morphing skin (Kikuta, 2003).

Among the smart materials listed, shape memory polymer (SMP) shows a high potential due to its low actuation force and ability to undergo high reversible deformation which is ideal for morphing skin application. SMPs are deformable materials that can undergo large deformation. They can be programmed into a temporary shape and can recover their permanent shape (as in Figure 1.1) upon external stimulations such as heat, light (Lendlein et al., 2005), electricity (Y.

Wang et al., 2016), magnetism and microwave (K. Yu, Liu, & Leng, 2014), etc. The application of SMP varies across many industries including aerospace (Jee, 2010), medicine, electronic devices, and self-assembling structures (Berry & Seo, 2015). However, SMP itself cannot withstand high operating condition of an aircraft wing. Limited number of available SMPs in the market also contribute to the lack of research in this area. Unlike shape memory alloys (SMAs) which change shape due to the crystalline phase transformation, SMPs change their shape due to the thermo-molecular relaxation of their polymer chain. This enables a high deformation of polymer without apparent material degradation. Experiments showed that existing SMPs have very low cycle to failure and are too brittle in the hardened state to withstand vibrational loads (Thill et al., 2008). This indicates a need of reinforcement to modify the performance and to overcome this limitation.

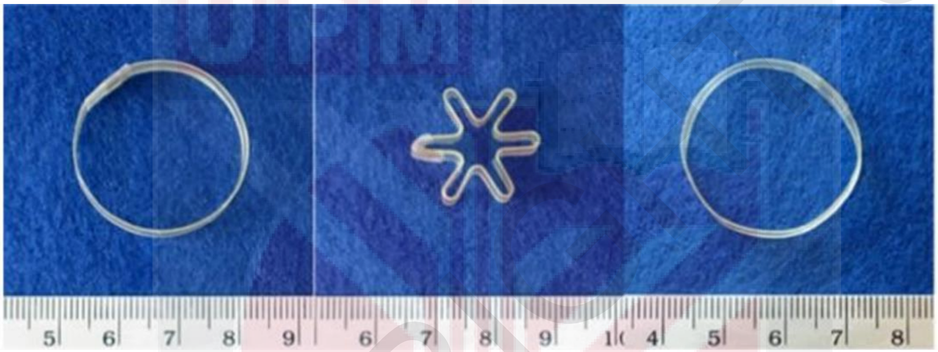

Figure 1.1: Shape memory cycle of SMP. The temporary shape is obtained after applying heat and mechanical deformation followed by rapid cooling. The original shape is then recovered after heating it again (Berry & Seo, 2015)**.**

One approach in toughening polymer is to add a second phase polymeric property such as fibre. Fibre-reinforced polymer produces a composite with properties that exceed either of the components alone. Fibre can be incorporated into a polymer matrix of either continuous length or discontinuous length. Addition of nanofiller is a recently developed reinforcing method. Materials reinforced with nanoscale reinforcement usually exhibit superior properties compared to macroscale reinforcement (L. Wang, 2005). Fillers have an important role in tailoring properties of various polymers and their addition have been proven to effectively improve the mechanical properties of polymeric materials (Chrissafis & Bikiaris, 2011). Among various nanofillers, clay minerals, carbon nanotube (CNT), and silica nanoparticles are the most used fillers in enhancing the physical, mechanical, and thermal properties of polymers.

Arguably the most studied natural product, nanoclay can be obtained in a large amount at a low cost, making it very popular as a filler nanocomposite in various applications. Initially introduced as a hybrid material rather than a nanocomposite by Usuki et al. (Kojima et al., 1993) and Okada et al. (Okada & Usuki, 1995) on nylon-6, nanoclay has kick-started the technique of using nanofiller in polymer matrix. Typical types of nanoclay includes montmorillonite (MMT), hectorite, mica, and bentonite. As a natural product, MMT are generally highly hydrophilic and thus, are compatible with a vast range of polymer types. nanoclay has provided a range of benefits in enhancing polymers such as mechanical properties (Athimoolam & Moorthy, 2015; Ho et al., 2006) and thermal stability (Barmar et al., 2006; Lakshmi et al., 2008).

CNT is one of the most used nanofillers for polymer nanocomposites. Since it was first observed by Iijima as a multi-walled carbon nanotube (MWCNT) structure in 1991 (Iijima, 1991) and then later as single-walled CNT structure in 1993 (Iijima & Ichihashi, 1993), the interest in CNT filler skyrocketed. CNT can be found in various structural forms, i.e., single-walled, double-walled, and multi-walled. They are made of one-atom thick graphite, called graphene, which are rolled into a cylindrical shape. An addition of CNT in a minute volume could lead to a considerable increase in the polymer's mechanical (Ni et al., 2007) and electrical properties (M. R. Ayatollahi et al., 2011). MWCNT are easily obtained due to their stable structure and low manufacturing cost which render it cost-effective and become the common selection for reinforcement. CNTs is considered as a unique reinforcement for polymer due to their exceptional electrical properties as well as mechanical properties.

The simultaneous presence of nanoclay and CNT in SMP could provide advantageous properties of both fillers and produce a multifunctional material that is suitable for morphing skin application. The addition of CNT in nanoclay increases the thermal decomposition temperature of epoxy nanocomposite (Hesami et al., 2014). However, the simultaneous presence of nanoclay and CNT results in a lower flexural strength than non-hybrid nanocomposites (Tüzemen et al., 2017). Hybridization bring about a safe and effective method of dispersibility of nanofiller using other nanofiller (Papageorgiou et al., 2017; Szeluga et al., 2015). It also introduces synergetic effect between nanoparticles (Nurul & Mariatti, 2013) as well as facilitating interaction within the polymer matrix which aid dispersion (Safdari & Al-Haik, 2013). To the author's knowledge, the practice of incorporating CNT and nanoclay is yet to be applied in SMPs. Therefore, there is a need to investigate SMP nanocomposite for morphing applications. Hence, this research focuses on investigating the suitability of SMP nanocomposite as morphing skin. In the process of determining the performance of the shape memory nanocomposite and subsequently, the hybrid nanocomposite, additional analysis is conducted that relates indirectly to the application of aircraft structure.

1.2 Problem Statement

The challenge in developing a morphing wing lies in finding the suitable materials that could fulfil the required properties, enabling it to change shape accordingly while maintaining the structural integrity under air load (Friswell, 2014; La et al., 2018; Thill et al., 2008). SMP is a promising smart material which can change shape when a certain stimulus is applied. This material have been studied in the past (Khan, 2008; Murray et al., 2010; Reed, Jr. et al., 2005; Y. Yu et al., 2007) however, it suffers a number of drawbacks such as low recovery stress and low deformation stiffness (Bashir et al., 2017; Rousseau, 2008).

Moreover, most SMPs show undesirable properties such as low degradation, brittle behaviour, and low shape memory properties. As a solution, shape memory polymer composite (SMPC) has been developed to overcome these drawbacks and develop SMP for practical application. The results of studies on SMPC shows desirable properties where SMPC has higher strength and stiffness compared to SMP. To further improve the properties of SMP, the idea of developing composites by incorporating different filler particles into the polymer matrix has become a topic of interest (Gunes & Jana, 2008; Lu et al., 2014; Madbouly & Lendlein, 2010). The demand of advanced nanotechnology has motivated researchers to modify polymers with nanoparticles such as CNT and nanoclay. However, the effect of MWCNT and MMT reinforcement on properties of SMP is still an active field of research (Abishera et al., 2017). In order to optimize the reinforcement of nanofillers, an optimum amount of fillers must be established in introducing nanofillers in the SMP.

An analytical study showed that the strategy of embedding multiple nanofillers into a polymer could significantly improve the performance and promote additional functionality to the shape memory material (Darder et al., 2018). Therefore, it was predicted that the infusion of hybrid nanofillers could result in additional functionality in the SMP (Mu et al., 2018). As the properties of hybrid shape memory nanocomposites are not widely explored, their basic properties must be examined. The mechanical, thermal degradation, and viscoelastic properties are among the essential properties that need to be analysed for newly developed materials.

The hybridization of nanofillers brings about additional functionalities of hybrid SMP. In addition to thermal actuation method, electrical stimulation method is commonly used to trigger the shape memory effect through Joule heating (J Karger-Kocsis & Kéki, 2015; Mu et al., 2018). The effects of hybridization on shape memory and actuation response need to be investigated in order to prepare the material for commercial use in real-life applications. In addition, the developed hybrid filler SMPs are predicted to be suitable for morphing wing application.

1.3 Research Objective

The current study aims to investigate the suitability of shape memory epoxy polymers (SMEP) for aerospace structural applications. MWCNT and MMT filler are embedded into the epoxy to modify the performance of SMEP. The performance and properties of the nanocomposite are analysed via experimental and fundamental material characterization. To achieve the aim of the study, the following specific objectives are carried out:

i. To identify the effect of MMT loading on thermal and shape memory properties of SMEP
ii. To analyse the effect of MWCNT loading on thermal and shape memory properties of SMEP
iii. To determine the effect of hybrid MMT/MWCNT loading in SMEP on its thermal properties
iv. To analyse the mechanical properties and morphology of hybrid MMT/MWCNT SMEP
v. To investigate the shape memory and actuation properties of hybrid MMT/MWCNT SMEP

1.4 Research Scope

The scope of the current research work is limited to the experimental evaluation of thermal, mechanical, and shape memory properties of MMT- and MWCNT-reinforced SMEP for morphing skin application. The materials used in this study are limited to modified MMT, MWCNT, and SMEP resin. Other nanofillers are not studied in this research. The laminated nanocomposite was fabricated through the open moulding method, followed by curing in a thermal oven. Other fabrication method was not utilised in this research. The influence of adding MMT and MWCNT on the thermal degradation, mechanical (tensile and flexural), morphological, viscoelastic, and shape memory effect were evaluated. Due to limitation of available nanofillers, the study was conducted according within the specified weight percentage. The research conducted does not study the application of developed materials on morphing wing which limit the study to material characterizations.

1.5 Thesis Organization

This thesis is divided into five different chapters that describe the study and development of the hybrid nanocomposite SMEP and are presented in the following manner:

i. Chapter 1 introduces a general researcher's view on the shape memory polymer and materials for morphing skin. This chapter also provides an overview of alternatives for the reinforcement of SMP.
ii. Chapter 2 provides an overview of morphing aircraft and the different materials that were tested as a candidate of morphing skin. In this chapter, a

brief introduction, and an in-depth literature review of SMP and epoxy nanocomposite are also provided. Besides that, various results of standardized testing and characterization are compared among the reported literatures.

iii. Chapter 3 describes the complete procedure of fabricating the SMEP nanocomposite. Additionally, the standard testing and characterization method are explained in this chapter.

iv. Chapter 4 presents the results and findings of the tested materials. Discussion on the result of each experimental testing is done separately with the aid of figures and tables to clearly highlight the research findings.

v. Chapter 5 is dedicated to summarizing the result obtained and concluding the study. A separate section provides recommendations and suggestions to improve the research method. In another section, a discussion of future works that can be conducted is outlined.

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Muhamad Hasfanizam Mat Yazik was born in Hospital Besar KL, W. Persekutuan in June 1992 to Mat Yazik Zakaria and Inche Marinah Che Abdullah. After finishing primary school at the Sek. Keb. Gombak Setia (SKGS) in 2004. He continued his journey in one of the boarding schools in Gombak; Gombak Integrated Boarding School (INTEGOMB). After 5 years of great success in academic, co-curriculum and leadership. Hasfanizam was offered scholarship from Malis Amanah Rakyat (MARA) to pursue his study in A-Level at MARA College Seremban (KMS) for two years. After completing A-Level with full colours, he was accepted to further his studies in Aerospace Engineering at The University of Sheffield (UoS), United Kingdom. After finishing his Master in Engineering, the pursued his postgraduate studies in Doctor of Philosophy in University Putra Malaysia under department the Department of Aerospace Engineering. Progressing well in his research studies on polymer nano-composites and hybrid composites, under the supervision of dedicated supervisors and co-supervisor, Hasfanizam passed his Viva Voce on October 2020. He is currently working with Malaysian Industry-Government Group for High Technology (MIGHT) as a Project Researcher developing the National Advanced Material Technology Roadmap 2021-2030 (NAdMTR).

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Internship

Student Exchange Support Program 2018; Scholarship for Short-term Study in Japan from Japan Student Services Organization (JASSO) at Kyushu Institute of Technology, Japan, 6 January 2018 - 31 March 2018 – Participant

Workshops and Seminars

3rd Malaysia Super Satellite Campus (MSSC) International Workshop on Engineering and Health Science 2017: Collaboration for Bioengineering, Mechanics and Nano-materials, Aerospace Manufacturing Research Centre, UPM, 31 January 2017 – Participant

Global Aerospace Industry Outlook and Insight into Malaysia's Aerospace Initiatives Talk, Aerospace Manufacturing Centre, UPM, 2 March 2017 – Participant

Composite Technology - Current and Future Trends Talk, Aerospace Manufacturing Research Centre, UPM, 16 March 2017 – Participant

International Workshop on Advanced Composites and its Manufacturing, Aerospace Manufacturing Research Centre, UPM, 10 April 2017 – Participant

Introduction to Taguchi Method, Aerospace Manufacturing Research Centre (AMRC) UPM, Universiti Kebangsaan Malaysia (UKM), The Institute of Engineers Malaysia (IEM), 26 April 2017 - Committee, Participant

Sharing Session on Student – Supervisor Relationship, Aerospace Manufacturing Research Centre, UPM, 2 May 2017 – Participant

Technical visit Airbus Helicopters Malaysia (AHM), Aerospace Manufacturing Research Centre (AMRC) UPM, The Institute of Engineers Malaysia (IEM),3rd May 2017 – Participant

Technical visit to Centre of Excellence Proton, Aerospace Manufacturing Research Centre (AMRC) UPM, The Institute of Engineers Malaysia (IEM), 15th May 2017, - Participant

Workshop on Predictive Engineering Analytical for Sustainability and Recent Trends, Aerospace Manufacturing Research Centre (AMRC) UPM, STRAND Aerospace Malaysia, Airbus Helicopters Malaysia(AHM), Siemens, IDS, DAG Technologies, 20 July 2017 - Committee, Participant

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