UNIVERSITI PUTRA MALAYSIA

DETERMINATION OF DEFECTS AND DAMAGE MODES IN KENAF-REINFORCED EPOXY COMPOSITES UNDER FATIGUE LOADING USING THERMAL IMAGING AND SEM TECHNIQUES

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By

SURIANI BINTI MAT JUSOH

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATIONS

Thanks to my endless loves;
My husband,
Mohd Nizam Ngah

My kids,
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My parents and parents in law,
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DETERMINATION OF DEFECTS AND DAMAGE MODES IN KENAF- REINFORCED EPOXY COMPOSITES UNDER FATIGUE LOADING USING THERMAL IMAGING AND SEM TECHNIQUES

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SURIANI BINTI MAT JUSOH

October 2012

Chairman : Professor Aidy Ali, PhD

Faculty : Faculty of Engineering

In engineering design, concerns arose on the fatigue behaviours of composite materials. Much effort has been done to estimate the fatigue life by which destructive techniques were commonly used. Recently, non-destructive techniques (NDT) are increasingly being used on composite materials to detect defects. Parallel to the development, shifting interests from traditional monolithic materials to fiber reinforced polymer based materials have been demonstrated by researchers and engineers. Nevertheless, information on the use of NDT on natural fiber reinforced composite materials is still sparse.

The present study was set out to detect defects and estimate fatigue life in natural fiber reinforced composite materials. In making the composite, kenaf bast was used as a reinforcement fiber with epoxy as the matrix. The NDT employed to serve the study

purposes are Infrared (IR) thermal imaging and optical microscope. In parallel, destructive technique (DT) was also used in this study specifically in carrying out the fatigue tension-tension test and scanning electron microscope (SEM). By and large, the DT was used just to verify all the results by NDT.

The advantages of using NDT via IR thermal imaging in kenaf reinforced epoxy composites to estimate the fatigue life are evidenced in the following results: IR has successfully detected five types of manufacturing defects in kenaf reinforced epoxy composites due to manufacturing process. The defects are voids, resin rich area, pockets of undispersed cross-linker, misaligned fiber and regions where resin has poorly wetted the fibers. Subsequently, IR thermal imaging has significantly determined fatigue damage in kenaf reinforced epoxy composites. In addition, fatigue damage modes has also been predicted and determined by the types of defects occurs due to manufacturing process. This proves that IR has successfully been a significant NDT in estimating fatigue life due to fatigue damage, proved experimentally and interpreted by the *S-N* curve. In terms of fatigue resistance, it is found that 60% fiber volume fraction kenaf reinforced epoxy composites specimen has the highest resistance at 119.71-53.20 MPa. Finally, based on damage accumulation, a model of fatigue life estimation, namely S-IR has been proposed.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENENTUAN KECACATAN DAN MOD KEROSAKAN KOMPOSIT EPOKSI BERTETULANG KENAF DI BAWAH BEBAN KELESUAN MENGGUNAKAN TEKNIK PENGIMEJAN TERMA DAN SEM

Oleh

SURIANI BINTI MAT JUSOH

Oktober 2012

Pengerusi : **Profesor Aidy Ali, PhD**

Fakulti : **Fakulti Kejuruteraan**

Dalam reka bentuk kejuruteraan, perhatian pada sifat kelesuan telah meningkat dalam bahan komposit. Banyak usaha telah dilakukan untuk menganggar jangkahayat kelesuan yang mana teknik ‘destructive’ lebih biasa digunakan. Kini, banyak teknik ‘non-destructive’ lebih kerap digunakan dalam bahan komposit untuk mengesan banyak kecacatan. Sejajar dengan perkembangan, perubahan minat yang telah beralih dari bahan monolitik ke bahan berasaskan polimer yang bertetulangkan gentian telah ditunjukkan oleh para penyelidik dan jurutera. Namun begitu, maklumat penggunaan teknik ‘non-destructive’ dalam bahan-bahan komposit bertetulangkan gentian semulajadi masih tidak mencukupi.

Kajian ini telah dijalankan untuk mengesan pelbagai kecacatan dan menganggar jangkahayat kelesuan dalam bahan komposit gentian semulajadi. Komposit disediakan menggunakan kulit kenaf sebagai gentian tetulang dengan epoksi sebagai matrik. Teknik 'non-destructive' yang digunakan adalah pengimejan terma Inframerah (IR) dan mikroskop optik. Secara selari, teknik 'non-destructive' juga digunakan dalam kajian ini khusus untuk menjalankan ujian kelesuan (tegangan-tegangan) dan mikroskop imbasan elektron. Secara keseluruhannya, teknik 'destructive' (DT) digunakan untuk mengesahkan semua keputusan oleh teknik 'non-destructive' (NDT).

Kelebihan menggunakan NDT melalui teknik pengimejan terma IR dalam komposit-komposit epoksi bertetulang kenaf untuk menganggar jangkahayat kelesuan dibuktikan dengan keputusan berikut: IR telah berjaya mengesan lima jenis kecacatan dalam komposit epoksi bertetulang kenaf yang diakibatkan oleh proses pembuatan. Kecacatan berkenaan ialah lompang, kawasan resin berlebihan, poket rentasan pemaut gentian tidak terserak, gentian yang tidak sejajar, dan gentian yang gagal ditutupi epoksi. Kemudiannya, pengimejan terma IR, telah ternyata berkesan menentukan kerosakan kelesuan dalam komposit epoksi bertetulang kenaf. Di samping itu, keadaan kerosakan kelesuan juga telah diramal dan ditentukan berdasar jenis kecacatan yang diakibatkan oleh proses pembuatan. Ini menunjukkan pengimejan terma IR telah berjaya menjadi NDT dalam menganggar jangkahayat kelesuan berdasarkan kerosakan kelesuan, dibuktikan secara ujikaji dan pada lengkungan *S-N*. Dari segi rintangan kelesuan, telah didapati spesimen 60% isipadu komposit epoksi bertetulangkan kenaf mempunyai nilai tertinggi pada 119.71-53.20 MPa. Sebagai rumusan, berasaskan kerosakan terkumpul, model untuk anggaran jangkahayat kelesuan, iaitu S-IR telah dicadangkan.

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I certify that a Thesis Examination Committee has met on 15th October 2012 conduct the final examination of Suriani Binti Mat Jusoh on her thesis entitled "Determination of Defects and Damage Modes in Kenaf-Reinforced Epoxy Composites Under Fatigue Loading Using Thermal Imaging and SEM Techniques" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U. (A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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Date: 29 November 2012

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

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Date:

DECLARATION

I declare that the thesis is my original work for my quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any institution.

SURIANI BINTI MAT JUSOH

Date: 15 October 2012

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LIST OF ABBREVIATIONS

ACFM	Alternating current field measurement
ACPD	Alternating current potential drop
ASTM	American Standard for Testing Material
CCD	Charges-coupled device
CFC	Carbon fiber composite
DAT	Active-dynamic area telethermometry
DCPD	Direct current potential drop
DVT	Deep Venous Thrombosis
EC	Eddy current
FEA	Finite element analysis
HFC	High fatigue cycle
IC	Integrated circuit
INTROP	Institute of Tropical and Forestry Product
IR	Infrared
IT-SOFCs	Intermediate temperature solid oxide fuel
IUPAC	International Union of Pure and Applied Chemistry
LFC	Low fatigue cycle
NaCl	Natrium Chloride
NDE	Non- destructive examination
NDT	Non-destructive technique
NDTE	Non-destructive thermal evolution

PC	Polycarbonate
PE	Polyethylene
PP	Polypropylene
PS	Polystyrene
PBT	Polybutlene tere
PDF	Probability density function
PET	Polytthylene terephthalate
PFC	PFC
PMMA	Polymethyl methacrylate
PPCF30	Polytthylene coir fiber without compatibilizer
PPFC30	Polytthylene coir fiber with compatibilizer
SARS	Secure Acute Respiratory Syndrome
SEM	Scanning electron microscope
S-IR	Suriani-Infrared
TI	Thermal investigation
TTM	Thermal mapping
UTS	Ultimate tensile strength

NOMENCLATURE

α	Material dependent parameter
β	Material dependent parameter
γ	Material dependent parameter
q	Material dependent parameter
m	Material dependent parameter
N	Number of cycle
N_f	Number of cycle to failure
S_f	A fatigue strength
S_e	Endurance strength
σ_R	Residual Stress
τ	Shear Stress
S-N	Stress-life curve@ Goodman diagram
E-N	Crack initiation strain-life
R	Stress ratio
σ_{max} or S	Maximum stress
σ_{mean}	Mean stress
σ_{min}	Minimum stress
σ_{UTS}	Ultimate tensile stress
σ_y	Tensile stress at yielding
ε	Strain
a	Crack length

c	Constant
D	Damage
da/dN	Crack propagation rate
E	Residual modulus
E_o	Initial Young's modulus
E_f	Failure Young's modulus
T_m	Higher melting temperature
T_g	Transition temperature
v	Ultrasonic wave in material inspect
d_0	Distance of defect from the surface of specimen
λ	Ultrasonic wave length
f	Frequency
C_p	Specific heat
k	Thermal conductivity
Q	Input energy
Z	Function of depth
μ	Thermal diffusion length

CHAPTER 1

INTRODUCTION

1.1 Fatigue Life Estimation in Composite Materials

Little do realize that the daily equipment we are using, from domestic appliances, to aerospace and aircraft industries are produced from composite materials. Generally speaking, composite materials form the base in most of the equipment and apparatus that facilitate lifestyle today. Due to its' strength, light yet durable, composite materials are favored by manufacturers and designers. In engineering design, with increasing use of composite materials in primary structures, the fatigue behaviors of composites are of serious concerns. Whilst profit is important, the safety and comfort of the consumers, gain equal attention. Thus, the prediction of evaluation of fatigue performance becomes a crucial aspect of structural analysis in maintaining the performance of the composite materials.

To date, the most commonly used composite materials are of metal or synthetic fiber, to that of naturally-based composite materials. However, these composite materials are subjected to cyclic fatigue loading (Van Paepegem and Degrick, 2002). One of the common occurrences in all materials is fatigue failures (Liu and Mahadevan, 2005). Enhancing the fatigue life of these materials will potentially enhance the quality of product performances. Nevertheless, the knowledge in predicting the fatigue life of composite materials, let alone the natural-fiber composite, is somewhat limited.

There are differences between the fatigue behavior in metal (isotropic) and naturally-based namely fiber-reinforced composites. In brief, composite materials are anisotropic and their fatigue behavior is more complicated than that of conventional materials. This is due to the significantly different damage process in composites from observed in homogeneous and isotropic materials. Four main damage modes have been observed in composites under fatigue loading which are fiber matrix debonding, matrix cracking, fiber fracture and delamination (Fong, 1982; Reifsnider, 1983). Also as reported by Fong (1982) in Xiang and Liu (2011), fatigue analysis of composite materials is difficult due to several basic characteristics of the composite material.

With regards to fatigue life estimation, many attempts have been made for fatigue modeling and life prediction of composite materials. Although the fatigue behavior of fiber–reinforced composites is fundamentally different from the behavior exposed by metals, many models have been established which are based on the well-known *S-N* curves. For example, Degrieck et al. (2001), proposed fatigue models which can be generally classified in three categories which are fatigue life models, the phenomenological models for residual stiffness/strength and the progressive damage models. Shokerieh and Lessard (1997) proposed a new model based on experimental data from a unidirectional ply under uniaxial fatigue to simulate the behavior of that ply in multiaxial fatigue loading. The use of composite materials has been shown to reduce life-cycle cost in some cases. All of these works illuminate the crucial necessity in improving the fatigue models and life time prediction methodologies, hence may result in more efficient use of natural fiber-reinforced composite. In economic perspective, these properties will eventually aid in reducing the marketing time. Huimin and Wei (2009), report that the prediction and evaluation of fatigue performance has become a part of structural analysis in composite materials. Also claimed by others researchers (Peck and Springer, 1991; Bond and Farrow, 2000; Taylor et al., 2000; Anderson, 1987), because of their specific properties, the fatigue life of composite materials are affected remarkably by loading and environmental factors, which must be taken into consideration in engineering design.

By and large, the aim of the aforementioned works is similar; hence, this study is no exception. Thus far, most of the technique of predicting the fatigue life in the above works employs the destructive technique (DT). Whereas, in this particular study, the researcher uses and promotes a non-destructive testing (NDT) in detecting the defects, determining the fatigue damage and estimating fatigue life of natural-fiber composite materials.

1.2 Problem Statement

Since the past few decades, research and engineering interests have shifted from traditional monolithic materials to synthetic fiber reinforced polymer based materials due to their unique advantages of high strength to weight ratio, non-corrosive property and fracture toughness. These high strength fiber composite materials such as carbon, glass and aramid, with low strength polymeric matric have now been dominating the aerospace, leisure, automotive, construction and sporting industries. Unfortunately, it has been found that these synthetic fibers have some serious drawbacks including non-renewable, non-recyclable, high energy consumption in manufacturing process, health-risk when inhaled and non-biodegradable (Cheung et al., 2009).

Recently, due to a strong emphasis on environmental awareness worldwide, it has brought much attention in the development of recyclable and environmentally sustainable composite materials (Cheung et al., 2009). Due to the awareness, natural fiber reinforced polymer composites become an attractive replacement for heavier metals. In particular, this recent attention-seizing phenomenon owes to superior fatigue and corrosion properties of natural fiber composites. Although less susceptible to failure than metals as they might be seen, composite fatigue failure in natural fiber composites which is generally driven by fatigue failure in polymer matrix (Kawai et al., 2001a; Awerbuch and Hahn, 1981; Petermann and Plumtree, 2001) might still occur.

All these revelations signify tremendous efforts invested to understand the complexity of fatigue in natural fiber-reinforced composite material. Nevertheless, knowledge with regards to estimation of its fatigue life is still sparse. This includes the absence of a model and non-destructive testing on estimating fatigue life, unlike the one being used at present on natural and metal composite materials. Thus, in this study, the damage evolution mechanism is decided to be one of the crucial focuses of fatigue behavior investigation of natural-fiber reinforced composite materials, apart from being a foundation to predict fatigue life. The urging need to study the durability and fatigue life based on experimental determination via a non-destructive testing is felt timely to provide rigor empirical procedural fatigue life estimation.

Thus, this study promotes the usage of thermal imaging, namely infrared (IR) technique, to estimate the fatigue life of composite material whilst presenting ways to understand the use of non-destructive testing on fatigue failure. The results of this study are hoped to bridge and narrow down the gap exists within the body of knowledge of fatigue life in relation to natural fiber composite materials.

1.3 Objectives

The objectives of this study are as follows:

1) To detect the defect in kenaf reinforced epoxy composites via IR thermal imaging technique.

2) To experimentally determined fatigue behaviour and fatigue damage mode in kenaf reinforced epoxy composites.

3) To predict the fatigue damage in kenaf reinforced epoxy composites via IR thermal imaging technique and thermography analysis.

1.4 Scope of study

The scope of the study encompasses the detection of the defects in kenaf reinforced epoxy specimens via NDT name as IR thermal imaging technique and extent to which the IR thermal imaging technique can determine fatigue damage. All of these aspects would finally lead to the estimation of fatigue life in kenaf reinforced epoxy composites, by determining the potential parameter and variables that influence the fatigue life. The goal is to know the fiber percentage towards resistance of tension-tension fatigue in kenaf reinforced epoxy composites hence, proposing kenaf reinforced epoxy composites fatigue life model.

This study limitation is as follows;

- NDT used in this study are thermal imaging technique and optical microscope observation
- only applicable on natural/cellulose fiber reinforced composites materials not for metal,
- concern only fatigue failure of constant cyclic loading not amplitude loading,
- excludes computational analysis such as finite element, proposed empirical modeling, analytical assumption and costing analysis.

1.5 Thesis layout

This thesis is designed in six chapters, encompassing introduction, literature review, theories, experimental procedures, results, analyses, discussion and conclusions. Chapter 1 briefly introduces the breakthrough achieved from this study on fatigue life estimation in composite materials before elaborating the problem statement, objectives and study scope. Chapter 2 presents literature review on previous and recent relevant research on composite materials. In particular, these include sections that highlight the perception on predicting the fatigue life of composite materials. Besides, previous researches on predicting fatigue life of composite materials using NDT are also incorporated. Chapter

3 is intended to explain the research methodology, describing details of the specimen preparation and test description. Chapter 4 mainly reports on findings and results. These include analyses and discussions. Chapter 5 describes fatigue life estimation of kenaf reinforced epoxy composites using thermal imaging techniques. The final Chapter 6 concludes the study and presents recommendations for future study.

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