DETERMINATION OF POROSITY LEVEL OF OUT OF AUTOCLAVE CARBON/EPOXY ADVANCED COMPOSITES USING ULTRASONIC C-SCAN (PULSE-ECHO METHOD)

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by

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LIST OF ABBREVIATIONS

OOA	Out of Autoclave
MMC	Metal Matrix Composite
CMC	Ceramic Matrix Composite
PMC	Polymer Matrix Composite
UT	Ultrasonic Testing
MPI	Magnetic Particle Inspection
NDT	Non-Destructive Testing
2D	Two Dimensional
3D	Three Dimensional
dB	Decibel
TOF	Time of Flight
AMP	Amplitude
PTFE	Polytetrafloroethylene
CFRP	Carbon Fibre Reinforced Plastic
GFRP	Glass Fibre Reinforced Plastic
TiMMC	Titanium Metal Matrix Composite
TIRP	Thickness Free Reflector Plate
BSE	Back Surface Echo
SNR	Signal to Noise Ratio
UEPM	Ultrasonic Energy Propagation Movie
UPGMA	Unweighted Pair Group Method with Arithmetic Mean
FD	Fractal Dimension
ATR	Automatic Target Recognition
PEEK	Polyetheretherketone
BSS	Blind Signal Separation
SDAC	Spectral Distance Amplitude Correction

WPT	Wavelet Packet Transform
SEM	Scanning Electron Microscopy
FFT	Fast Fourier Transforms
RGB	Red, Green, Blue
ASTM	American Standard Test Method

LIST OF SYMBOLS

MHz	Megahertz
kHz	Kilohertz
MHz	Megahertz

TAHAP KELIANGAN OLEH PENGUJIAN PENGIMBASAN ULTRASONIK (KAEDAH GEMA DENYUT) UNTUK LAMINAT KARBON/EPOKSI DIPERBUAT MENGGUNAKAN KAEDAH LUAR AUTOKLAF

ABSTRAK

Teknik pemeriksaan menggunakan ultrasonik C-scan adalah yang paling sinonim dalam industri pembuatan bahan laminat komposit termaju. Tidak ada piawaian yang boleh diterima pakai secara meluas atau prosedur untuk pemeriksaan C-scan ultrasonik untuk bahan yang berasaskan komposit termaju yang mana boleh mengakibatkan kebolehpercayaan dan kebolehkesanan kaedah ini telah terhad. Pengkajian tentang kaedah dan pengujian bahan komposit termaju telah dijalankan sebagai tindakbalas kepada masalah di atas. Kajian ini telah menghasilkan tiga prosedur untuk mengendalikan kelengkapan C-scan melalui kaedah denyut gema, penghasilan panel rujukan yang mana panel rujukan ini adalah dihasilkan melalui kaedah yang diperakui kualitinya dan melakukan analisis lanjutan dengan penggunaan perisian seperti Matlab yang bertujuan untuk menganalisa kecacatan dalam bentuk imej 3 dimensi. Tesis ini memberikan gambaran keseluruhan objektif dan aktiviti penyelidikan yang telah dijalankan. Sebagai penemuan, penilaian antara ujian musnah dan tanpa musnah (pemprosesan imej) menunjukan kebolehbandingan antara satu dengan yang lain. Sampel O1 adalah sampel yang terbaik dikalangan bahan OOA disediakan dalam experimen ini dengan menghasilkan kecacatan samada keliangan ataupun lompang dengan peratusan yang rendah. Ianya melibatkan pemampatan vakum dengan menggunakan teknik beg vakum yang normal dan dibantu dengan dua (2) sumber vakum dengan kehadiran filem polytetrafluoroethylene yang ditempatkan pada permukaan acuan bagi melancarkan perjalanan resin dipermukaan acuan semasa proses penyediaan sampel. Analisis selanjutnya perlulah dilakukan dalam usaha menilai secara kualitatif.

DETERMINATION OF POROSITY LEVEL OF OUT OF AUTOCLAVE CARBON/EPOXY ADVANCED COMPOSITE USING ULTRASONIC C-SCAN (PULSE-ECHO METHOD).

ABSTRACT

Various inspection techniques are available for inspection and quality control in monitoring services of composite structures, which ultrasonic inspection techniques C-scan is the most widely used in industrial manufacturing advanced composite materials. At this time, there are no standards that can be applied universally or procedures for inspection ultrasonic C-scan for advanced composite based materials which may cause the reliability and traceability of these methods has been limited. In response to this need, the study of methods and testing of advanced composite materials has been carried out. This assessment has produced three procedures 1. to operate the equipment C-scan through the pulse echo method, 2. producing the reference panel which reference panel is produced by a method recognized for its quality and usability of advanced composite panel produced aims to simulate defects. 3. Perform advanced analysis with use software such as Matlab®, which aims to analyze the defects in the form of three-dimensional images. This thesis gives an overview of the objectives and research activities to be carried out. As a finding, evaluation performance between destructive and image processing technique shown that comparable results between each other. Sample indicated O1 was determined as a best sample among others with lowest defect (i.e. porosity and void) percentage. It involved in no debulking process with normal vacuum bagging technique assisted by two (2) vacuum sources with polytetrafluoroethylene film on mould surface to enhance flow during fabrication. Supporting analysis should be carried out in order to analyze qualitatively.

CHAPTER ONE

INTRODUCTION

1.1 Background

The latest generation of prepreg out autoclave has been introduced by prepreg material producers, this latest development of prepregs has shown that it is possible to produce quality autoclave parts using vacuum bag assembly. Without the use of autoclave, the prepreg material developed can reduce the cost of acquisitions and operations, and is compatible with various types of low cost curing process, including conventional ovens, heating blankets, and heated appliances (R.Harshe, 2015).

The use of prepreg out of autoclave (OOA) does not accelerate the production process of advanced composite products. Due air trapped removing during processing, is it a time-dependent process. The curing cycle of OOA technique is usually longer. Typically, after completely conducted debulking process, vacuum must be held for a longer period before starting the curing process. The duration of this vacuum holding depends on the size and complexity, from as low as 4 hours for 0.4 m^2 to 16 hours for 72 m^2 (Cytec Industries Inc.,2015). In addition, external factors such as relative humidity also play a very important role in controlling void content in the lamina. In general, epoxy resin tends to absorb moisture in the air and trapped humidity is very difficult to remove under VBO processing. The effect of relative humidity on empty content of processed laminate VBO has been systematically studied (Nutt et.al 2010).

Method of inspection using ultrasonic C-scan was identified as a primary screening technique for determining the quality of an advanced composite material before it can be used in the field. The principles of ultrasound and disability carbon /

epoxy composite can occur during the fabrication process have been widely discussed (Brodal et al. 2011; Maeva et al. 2011; Shen & Dongri 2012; Shuck 2010; Venegas et al. 2012; Wright & Kingdom 2006; Zhangb et al. 2010) Among the defects that often occur in polymer-based composite materials are voids that exist in the laminate and the product during the manufacturing process. Defects such as voids and porosity often occurs due to the manner and appropriateness occur during the manufacturing process. Defects such as may result in a reduction in interlaminar shear strength (ILSS) of 5 % to 7 % for every 1 % increase voids are detected (AA 1997; Bowles & Frimpong 1992; Costa et al. 2001; Ghiorse 1993; H Jeong 1997; Schnars & Henrich 2006; Wisnom et al. 1996). With this reduction, it would lead to a reduction in strength as a whole. Generally, void exist in or between the layers of fibers used even in resin system itself. There are four mechanisms that can cause the formation of voids and porosity: (Loos & Springer 1983)

(i) the air trapped during the accumulation layer.
(ii) a slowdown in the resin.
(iii) volatiles released from the curing process.
(iv) internal pressure build-up of resin cure shrinkage.

1.2 Problem Statement

Generally, Cycom 5320 is a newly developed Carbon/Epoxy prepreg material by Cytec (Cycom 5320) for OOA application. Since this is a newly developed material, it required a determination of the most suitable bagging technique that produced high quality of composite laminate. In order to determine the quality of the laminate using pulse-echo ultrasonic C-scan, there is several problems have been highlighted as below:

i. Porosity detection on the composite laminate especially on the laminate surface. Most of the quality inspection were conducted using non destructive and destructive method which is costly.

ii. Insufficient solution of defect detection

iii. Existing image and signal processing is not adequate to determine the overall composite quality

    a. The problem of detection of localized porosity through the thickness in composite materials
    b. Image processing : Most problem has been highlighted is the speckle region in the image. (Image noisy and blurred)
    c. Signal processing : limitation of that highlighted is attenuation of ultrasonic waves is due to absorption and scattering phenomena especially for localized porosity.

Since the preparation of composite sample using prepreg material has been done in the environmental controlled room (controlled from dust, temperature and humidity), assumption has been made that there is no others defect affected sample during fabrication process except porosity/void defect.

1.3 Objectives.

The general objective of this research is to design, manufacture and test representative parts in order to evaluate and compare the capacities of autoclave and out-of autoclave manufacturing. The are the specific objectives that need to be achieved such as below:

i. To produce carbon/epoxy composite panels using OOA technique by means of vacuum bagging and oven cure.

ii. To compare the manufactured carbon/epoxy composite panels using UT (pulse echo method) with Matlab®.

iii. To analyse and correlate the porosity level using non destructive and destructive test.

1.4 Scope of Study

In this study, without any necessary tools, the non-destructive inspection of composite laminate will depend on the experiences in analysis the C-scan images. Most of the practice done will involve with destructive test to determine the defect, impurities or discontinuities in composite laminate such as determination of void content using burn off, acid digestion technique and mechanical test. In order to analyze the composite laminate quantitative and qualitatively, some of the researcher introduced data analysis algorithm. With this technique, comparison of signals and images been done. It also helps in eliminate noise signal. Matlab® will be used in order to analysis raw data from ultrasonic C-scan test. TOF and AMP data was analyzed accordingly in order to determine the thickness, porosity level and location. Besides that, density was measured in order to compare with the analyzed data. 3D images were produced and analyzed accordingly especially to determine the region that saturated with porosity. In this research, newly developed prepreg under tradename of Cycom 5320 was used as the main material which dedicated for OOA moulding and it has been produced by Cytec Industries Inc. As a comparison purpose Cycom 970 has been used. Cycom 970 is a prepreg that establishly used for autoclave moulding. Variation of bagging techniques were successfully conducted.

CHAPTER TWO

LITERATURE REVIEW

2.1 Out of Autoclave Technology (OOA)

The out of autoclave (OOA) or vacuum bag only (VBO) process depends largely on the extraction process or the physical and volatile air removal that aims to achieve low porosity which is time-dependent processes (C.Ridgard, 2016). The strategy to minimize the amount of curing period have been discussed in several scenarios by researchers and manufacturers especially involve in-tool and characterization of prepreg and adhesive film used which has freestanding postcure for monolithic and sandwich structure particularly after demoulding process (C.Ridgard, 2016).

Fibre placement known as the most popular fabrication route nowadays. R.W. Koon et. al (2016) investigated the fibre placement in order to investigate the quality of high rate automated fabrication for composite structure which is developed by Lockheed Martin Skunk Works® for MTM45®. They are comparing the quality of the product which is produced by autoclave and fibre placement technique. As finding, machine design and process parameters contributes a significant impact on improving the quality and cost for OOA fibre pacement process.

A.Haro et.al (2016) reported on rapid OOA composite manufacturing for aerospace grade prepregs which involved time-intensive and cost. Due to high demand for aerospace primary and secondary structure in the future with void free quality as well as performance wise, they introduced rapid process known as ‘Quickstep’ process (Figure 2.1) which involved with rapid heating and cooling with moderate compaction pressure. Two types of commercially available prepregs were utilized in the study for comparison among ‘Quickstep’, vacuum bag (VBO) and autoclave. Their system demonstrated more than 50 % total cycle time reduction

compared to VBO and autoclave with less that 1 % void content which is comparable with autoclave method.

Figure 2.1 : Quickstep QSE250 Machine and Curing Chamber (A.Haro et.al, 2016)

2.2 Ultrasonic C-Scan Testing

UT is the most popular nondestructive inspection method to examine the homogenous and heterogeneous materials. Frequency of 20 kHz to 20 MHz commonly applied in examining homogenous materials (i.e. noncomposite). When conducting composite materials evaluation, the operating frequency is usually below 5 MHz which is lower due to increment of attenuation inside materials and reduced the detection capability for tiny flaws. (Network & Guide 2013). Generally, UT can be used for varies material (metals, plastic and wood) in determining defect on surface and subsurface. Besides that, it also can be used in order to determine the thickness of materials via sound velocity and attenuation measurement (Krautkrämer & Krautkrämer 2013; Worlton 1956; Silk 1984; Willcox & Downes 2003; Birks et al. 1991)

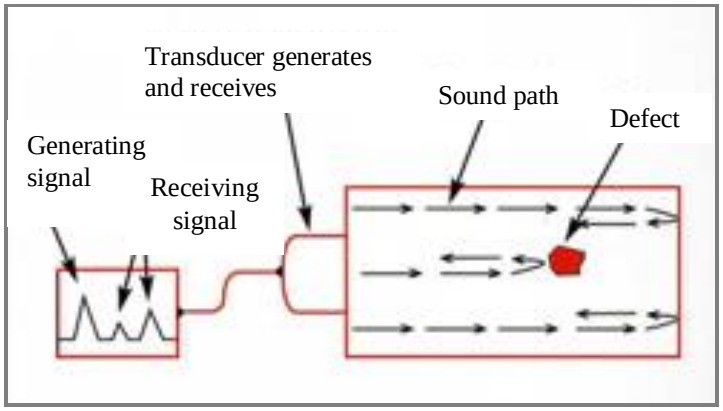

Figure 2.2: Ultrasonic testing principle (Krautkrämer & Krautkrämer 2013)

During inspection, high frequency pulses of sound wave are transmitted through the thickness of composite and sound reflecting time is measured. It is useful in determining void and delamination inside composite materials (Sfarra et al. 2013). Attenuation, signal scattering and absorption are the difficulties occurred in composite inspection especially in inspecting a thick and complex composite products.

2.2.1 Pulse Echo

Determination of the defected location in materials were measured using time required for UT pulse which generated by transducer to travel through the thickness of material and reflect back from defect location (i.e. voids, delamination and impurities) to transducer. Time interval is recorded as a few microsecond. There is two way transit time which is penetration and reflection the travel path. The relationship stated in Equation (i)

$$d = \frac{vt}{2} \; or \; v = \frac{2d}{t} \qquad \text{(i)}$$

where [d] is the distance from the surface to the discontinuity in the test piece, v is the velocity of sound waves in the material, and t is the measured round-trip transit time.

2.2.2 Attenuation Measurement

Propagation of UT wave is influenced by the microstructure of the materials. Modulus of elasticity and density probably influence the UT wave velocity which normally governed by the phases and defect inside materials. Sum of absorption and scattering (UT attenuation) depends on damping and scattering from the grain boundary in the materials. Knowledge and data of thermophysical parameter is useful in order to characterize the produced attenuation signal which is difficult to quantify (Ru 2006) (Bastianini, Tommaso, et al. 2001).

The change of signal attenuation and conducting a simple qualitative measurement can be carried out which determine easy way compared to real measurement. This is known as relative measurement. Measurement of the relative attenuation measurements can be conducted by examining the exponential decay of multiple back surface reflections. Mechanical properties and variation in microstructure properties produced small changes in terms of attenuation and wave velocity (Bastianini, Tommaso, et al. 2001).

2.2.3 Defect Detection Using Ultrasonic C-Scan For Homogeneous Materials

Kapadia (2013) wrote about his work in deciding the utilization of frequency for imperfection location as a part of homogeneous materials. As a finding, the frequency regularly utilized is as a part of the scope of 20 kHz to 20 MHz (Network & Guide, 2013).

Summerscales (1990) reported they utilized of straight versatile break (linear elastic fracture) mechanics as a premise for the definition ought to be recognized to decide the measure of sub basic imperfections. It is because of the life of homogenous materials, for example, metal is controlled by the nucleation and development of splits or harm in the material (Summerscales 1990) . Li et.al (2010) had concentrated on the examination of various NDT system which is thermography