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Finite Element Analysis and Resin Rich Area Characterization of Spread Tow Carbon Fibre Fabrics from Synchrotron X-ray Tomographic Microscopy

Siriganya Kampanthong[1], Phakkananan Pakawanit[2], Sutatch Ratanaphan[1,3*] and Sontipee Aimmanee[4 *]

[1] Department of Tool and Materials Engineering, King Mongkut's University of Technology Thonburi, 126 Pracha Uthit Rd., Thung Khru, Bangkok 10140, Thailand
[2] Synchrotron Light Research Institute, 111 University Avenue, Muang District, Nakhon Ratchasima 30000, Thailand
[3] Center of Excellence in Theoretical and Computational Science Center (TaCS-CoE), Faculty of Science, King Mongkut's University of Technology Thonburi, 126 Pracha Uthit Rd, Thung Khru, Bangkok 10140, Thailand
[4] Department of Mechanical Engineering, King Mongkut's University of Technology Thonburi,126 Pracha Uthit Rd., Thung Khru, Bangkok 10140, Thailand
*Corresponding author's e-mail address: sutatch.ratanaphan@mail.kmutt.ac.th and sontipee.aim@kmutt.ac.th

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ABSTRACT

The fact that most of the finite element method (FEM) on micromechanics of carbon fibre-reinforced polymers are generally constructed based on the two-dimensional image correlation obtained from optical or field emission scanning electron (FE-SEM) microscopes, interpretations of stress or strain distributions and resin-rich area characterizations are limited due to the lack of the dimensional views. To alleviate this issue, we obtained the three-dimensional structures of the spread tow carbon fibre/epoxy composites by using the synchrotron X-ray tomographic microscopy located at synchrotron light research institute, Thailand. For FEM analysis based on a region of interest, the materials properties of the constituents in the composites were assigned according to the X-ray spectrum contrast differences. Specifically, matrix and carbon fibre had different elemental compositions. Thus, the synchrotron X-ray imaging result showed different grey values, which could differentiate the matrix and reinforcement region for the FEM process. The load is applied onto a side of 3-dimensional models to obtain the results in the form of maximum stress values, displacement, etc. It was found that stress or strain distributions were consistent with the loading experiment. Resin-rich areas were detected in volumetric values, indicating that 3-dimensional modelling from synchrotron XTM imaging results can represent the characteristics of STCFRP samples. FEM results showed the effect of fabric pattern in the stress distribution and implied that mechanical properties by weight of spread tow carbon fibre reinforced polymer. STCFRP fabrics are more than commonly used CFRP fabrics. Statistical analyses of phase-contrast X-ray computed micro-tomography reveal distinctive gradients as well as localized correlations between carbon fibre and matrix phases. Based on these differences, a highly efficient algorithm for fibre tracking using statistical distributions of phase contrast has been proposed in this study.

INTRODUCTION

Due to several advantages, carbon fibre reinforced polymer composites (CFRP) are often used in the automotive industry and automobiles.[1] Spread tow technique on a carbon fibre tow will enhance the advantages of fabricating a spread tow carbon fibre reinforced polymer (STCFRP) composite material since it can make the fibre tow thinner, resulting in a reduction in the thickness per ply and an increase in the strength-to-weight ratio. Establishing the fibre and matrix volume fraction is essential to estimate and contrast the mechanical characteristics of carbon composite materials. Several research examined the relationship between the macroscopic parameters of the composite, such as tensile strength, compressive strength, shear strength, and fatigue properties [2]. X-ray Tomographic Microscopy (XTM) with a high solution procedure provides the imaging results at the microscopic level to separate the matrix and the fibre region by the contrast of the intensity [3]. Furthermore, the XTM technique gave us the image stack file that could generate a three-dimensional model to examine visual data such as the volume fraction of the matrix and fibre area or the 360 degrees visualization to examine the direction or distribution of the fibres. To ensure that the quality of XTM data is accurate and adequate to generate three-dimensional models to apply the Finite Element Analysis, the image results generated from XTM were calibrated using two-dimensional image results from Field Emission Scanning Electron Microscope (FE-SEM). In this experiment, the advantages of XTM and FE-SEM will be combined and generated the 3-dimensional models that can represent resin rich contents and mechanical behaviors of STCFRP under the load test. Moreover, the analysis of the resin-rich area, which relies on 3-dimensional modeling techniques, offers a non-destructive approach that has the potential to decrease material wastage. By employing this method, it becomes possible to accurately identify and quantify the resin-rich regions within the composite material without causing any damage to the sample. Manufacturers and researchers can gain valuable insights into the material distribution and optimize their manufacturing processes to achieve higher efficiency and cost-effectiveness.

METHODOLOGY

1. Spread Tow Carbon Fibre Reinforced Polymer – Sample Preparation

The Spread-Tow Carbon Fibre Reinforced Polymer (STCFRP) samples included eight layers spread-tow carbon fibre fabrics, (0/0/0/0) s. The specimens from the vacuum infusion process contain four types of spread tow carbon fibre from Alpha Composition, i. e. 200GSM and 205GSM twill weaves, 205GSM and 280 GSM plain weaves respectively. Epoxy resin mixture consisted of 35LV(A) 7257(B) with a 3:1 ratio from Neotech Composite. All specimens were well-prepared to 3x5x10 mm and 3x1x10 mm. sizing by a rotary microtome and sanitized by the ultrasonic water cleaner for examination through a Field Emission Scanning Electron Microscope (FE-SEM) and X-ray Tomographic Microscopy (XTM) at Synchrotron Light Research Institute (SLRI).

2. Resin-Rich Area in Carbon Fibre Composite

A Resin-Rich Area (RRA) refers to a region within a composite material where the concentration of resin is higher than the concentration of reinforcing fibres. In the context of carbon fibre composites, a resin-rich area would be a region where the concentration of resin is higher than the concentration of carbon fibres. Improper layup procedures used during fabrication, inefficient curing procedures, or incorrect tooling design are all possible causes of resin-rich areas in carbon fibre composites. Inadequate compaction pressure during fabrication can also lead to the formation of resin-rich areas, as can poor fibre wetting due to insufficient resin flow. Thus, carbon fibre composites should be manufactured carefully and subjected to strict quality control procedures to prevent resin-rich regions from impairing the material's durability and performance.

In carbon fibre composites, resin-rich regions can lead to decreased mechanical strength, decreased stiffness, and higher weight, Because the excess resin adds unnecessary weight without contributing to mechanical properties such as the mechanical stiffness and strength. Moreover, resin-rich regions can also compromise the composite's dimensional stability and heat resistance, limiting their suitability for certain high-temperature applications. To ensure high-performance carbon fibre composites, meticulous attention to resin distribution and manufacturing processes is crucial.

3. X-ray Tomographic Microscopy (XTM)

Microscopic image results were scanned by an XTM from the 1.2W Beamline at Synchrotron Light Research Institute, Nakhon Ratchasima using YAG-Ce scintillator sCMOS camera with 5.5 megapixels (2560 x 2160) as a detector and 10X optical projection magnitude. A scintillator will gather and transform the variations in the passage of energy waves responding to an object's interior structures when it is subjected to X-rays into visible light. After that, it will go via a minuscule lens system and be captured by the sCMOS camera as digital signals. To create "sinograms," which are then utilized to reconstruct computed tomography, CT slices, a collection of X-ray pictures will be processed. By stacking the CT slices that are produced, the item may be examined, and its internal components can be seen. The experiments at the BL١،٢W will be carried out based on the synchrotron radiation X-ray tomographic microscopy (XTM) represented in **Figure 1**

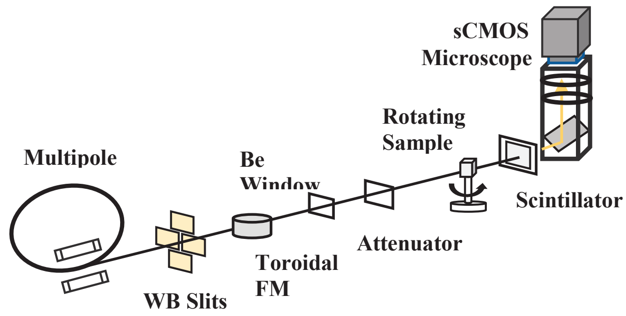

Figure 1. Schematic of X-ray Tomographic Microscopy (XTM).

4. Field Emission Scanning Electron Microscope (FE-SEM)

After the scanning process by X-ray Tomographic Microscopy (XTM), another set of specimens with 3x5x10 mm dimensions were carried out by TESCAN MIRA3 Field Emission Scanning Electron Microscope with the magnitudes of x20 x50 and x200, SEM HV 30 kV at Synchrotron Light Research Institute, Nakhon Ratchasima. Before the scanning process, all specimens were sterilized by ultrasonic and then coated with gold and affixed onto the scanning stub.

5. Image Data Acquisition and Finite Element Method (FEM)

After all the image data of STCFRP specimens were collected from XTM, The Octopus Reconstruction software from beamline 1.2 at SLRI was used to reconstruct the gathered image data from the XTM procedure, and subsequently, VG Studio Max (version 2022.3) software was used to generate the visualizable 3-dimensional models according to the same iso-value and create the model for a finite element analysis by the structural analysis module. FE-SEM 2-dimensional imaging results were measured and calibrated by ImageJ software.

The finite element analysis of four 280 GSM plain weave STCFRP samples was examined on the 100 x 100 x 100 cubic voxel models with the same distance distribution of 100 voxels in both axes as representative volume elements, which 1 voxel size equals to 0.0007 mm. The Representative Volume Elements, RVEs contain fibre and resin [4]. The top, and bottom planeswill be selected as a region of interest ROI. The bottom side of the RVE will be fixed and the 10kN load will be applied only on the top side along Y-axis.

Table 1. T300/T700 carbon fibre and epoxy resin's mechanical properties were obtained from TORAY and Neotech Composite's technical datasheets.

Tensile Modulus (Pa)	T300/T700 CF	2.30 e+11
	Epoxy Resin	3.81 e+09
Poisson's Ratio	T300/T700 CF	0.2
	Epoxy Resin	0.3

RESULTS

1. High-Resolution X-Ray Tomography and Image Processing

The imaging results of X-ray Tomographic Microscopy, XTM provided the results in the form of stack files consisting of a thousand 2-dimensional TIFF (Tag Image File Format) images. The difference between the intensity of the fibre and resin region depends on the absorption coefficient and density of carbon fibre and epoxy resin. Carbon fibre has a higher density and atomic number than epoxy resin, which means that it absorbs more X-rays than epoxy resin. As a result, the carbon fibre appears brighter or has a higher intensity than the epoxy resin in XTM images. The darker area in **Figure 2** represents the resin-rich area.

2. Field Emission Scanning Electron Imaging Results

To verify the 3-dimensional model generated from XTM imaging results, initially, a high-efficiency imaging method is required. FE-SEM provides good image quality with different magnitude values. [7] In this experiment, the magnitudes of 20x, 50x, and 200x were selected to represent the characteristics of fibre and resin. **Figure 3** represents both parallel and perpendicular sides of the 205GSM plain weave sample. FE-SEM image result clearly shows the fibre's characteristics in microscale. **Table 2** indicates that the fibre's diameters are consistent with every sample due to the 7μm measured by ImageJ software. Furthermore, the magnitude value of 200x in **Figure 4** shows the difference between plain weave and twill weave's lay-up structure. The plain weave tow bunch's structure is in a lenticular shape, meanwhile the twill weave tow bunch's structure is a long quasi-elliptical shape.[6]

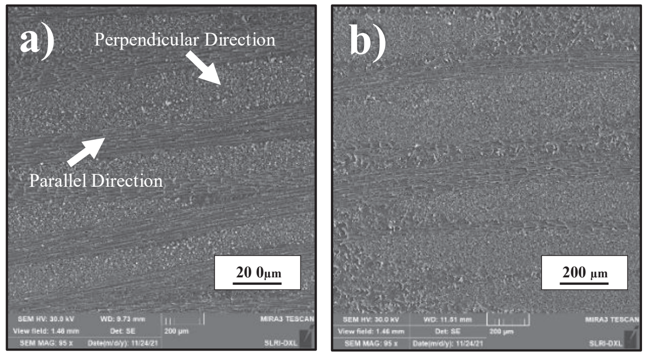

Figure 4. FE-SEM Images of both-perpendicular and parallel directions of carbon fibres of a) 205GSM twill weave b) 205GSM plain weave sample.

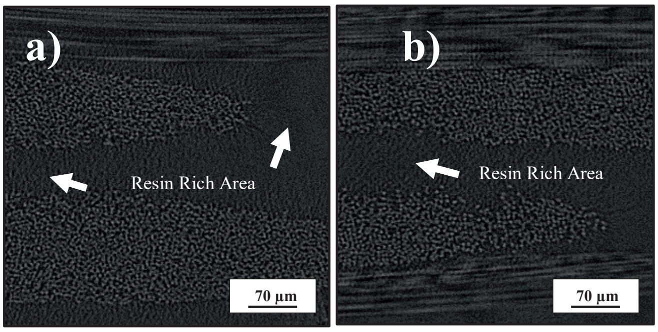

Figure 2. Example of a single image from the tiff stack obtained from XTM with the resin-rich regions a) 205 GSM Plain Weave and b) 205 GSM Twill Weave

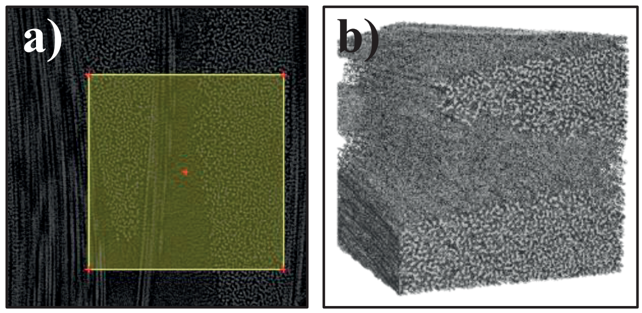

Figure 5. a) single .tiff image of 205 GSM twill weave image stack obtained from XTM b) 600 x 600 x 600 cubic voxel generated from VG Studio Max software.

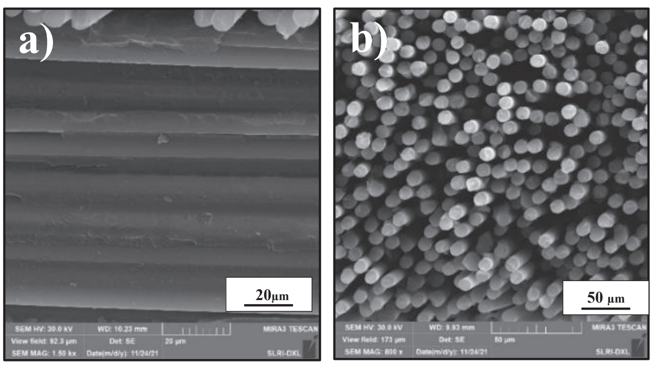

Figure 3. FE-SEM Images of a) the fibres along the fibre's direction b) the cross-sectional perspective of the 205GSM plain weave sample.

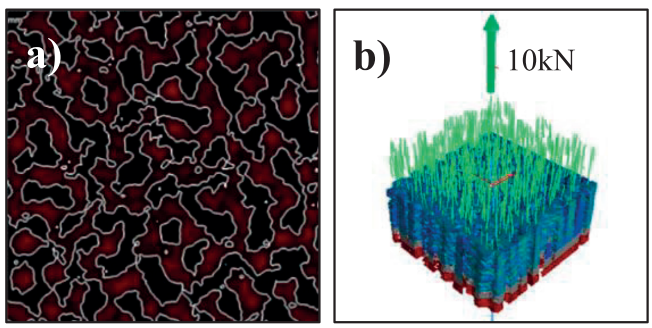

Figure 6. Imaging surface determination of 100 x 100 x 100 cubic voxel of 280 GSM plain weave a) after applying surface determination b) after applying 10kN load along the Y-axis.

Table 2. The fibre dimensions obtained from FE-SEM image results. Measured by image processing software ImageJ.

STCFRP Sample Types	1st Measurement (μm)	2nd Measurement (μm)	3rd Measurement (μm)	Measuring Angle (deg)	Average Fibre Diameters (μm)
200 GSM Twill	7.005	7.086	7.183	90.00	7.091
205 GSM Twill	7.020	7.044	7.414	90.00	7.159
205 GSM Plain	7.103	7.148	7.160	90.00	7.137
280 GSM Plain	6.933	7.161	7.181	90.00	7.091

Note: The measuring angle value is also shown on the ImageJ. Only 90 degrees measurements will be counted.

Table 3. Fibre volume fraction (Vf) measurement of four types of carbon fibre reinforced epoxy resin samples from XTM generated 3-dimensional models using VG Studio Max software.

STCFRP Sample Types	**RVE Size of 600 x 600 x 600 Cubic Voxel**				
	Total Volume ($Units^3$)	Object Volume ($Units^3$)	Fibre Volume Fraction (V_f)	Resin Volume Fraction (V_m)	VG Studio MAX's Iso-Surface Value
200 GSM Twill	2.16 e+08	1.50 e+08	0.700	0.300	9200
205 GSM Twill	2.16 e+08	1.56 e+08	0.725	0.275	9200
205 GSM Plain	2.16 e+08	1.44 e+08	0.663	0.337	9200
280 GSM Plain	2.16 e+08	1.52 e+08	0.704	0.296	9200

Table 4. Principal Stress obtained from VGStudio Max Structural Analysis module of 280 GSM plain weave samples with different fibre volume fractions.

Fibre Volume Fraction (V_f)	**Resin Volume Fraction** (V_m)	**Principal Stress (MPa)**
0.69	0.31	3.62e+6
0.72	0.28	3.97e+6
0.74	0.26	4.14e+6
0.77	0.23	4.31e+6

Thus, the measurements of fibre diameters from FE-SEM will be used to validate the accuracy of the 3-dimensional models to identify the resin-rich area values by VG Studio Max software. In other words, the fibre diameter obtained from FE-SEM will be compared with the fibre diameter of the generated models to optimize the most appropriate iso-value in this resin-rich area analysis.

3. Resin-Rich Areas Quantification using 3-Dimensional Modeling

The fibre's volume fraction (V_f) ranging of conventional tow of a regular CFRP composite is approximately around 60% [8], this experiment tends to predict both fibre and resin's volume fraction of spread-tow carbon fibre composite. Due to the characteristics of the spread-tow fabric's alignment that causes a more uniform distribution of fibres than the regular tow, the volume fraction of the fibre will be greater than the volume fraction of the regular tow.

The fibre volume fraction, (V_f) result in **Table 3** indicates that the spread tow carbon fibre reinforced by epoxy resin has more than 60% of fibre content calculated from the 3-dimensional models with the iso-surface value of 9200 that referred from the actual fibre diameters of 7 μm. The results implied that with the same iso-surface value, the fibre volume fractions of four STCFRP reached the prediction due to the process of tows making (0.700, 0.725, 0.663 and 0.704 respectively).

CONCLUSION

An investigation of Spread-tow carbon fibre reinforced with polymer, STCFRP using a 3-dimensional model reconstructed from X-ray tomographic microscopy .TIFF images stack indicates that the specimen's characterization can be scrutinized deeply into the fibre and resin content. The resin-rich area can be detected from the intensity which is darker than the fibre region due to the differences between atomic number and absorption coefficient of these two materials. The 3-dimensional projection technique the from 2-dimensional images stack will lead to the non-destructive evaluation of materials in the industrial fields and others. The volume fractions obtained from 600-cubed voxel hypothetically determined that the STCFRP composite may provide higher fibre content to increase the material properties. Also, the results on Finite element analysis of four RVE samples were consistent with the measured fibre contents. Furthermore, the models of STCFRP samples taken by XTM can also provide the distribution of fibres and matrix (known as resin) that the structures can be measured by using a small size of a sample instead of using the whole size of a sample which can decrease the rate of a material wastes.

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