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X-ray microtomography applications for quantitative and qualitative analysis of porosity in woven glass fiber reinforced thermoplastic

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ABSTRACT

A three dimensional representation of woven glass fiber reinforced thermoplastic composite has been obtained by means of X-ray microtomography. Various methods of image segmentation were applied to classify microtomography results. Segmented images were used to perform complete qualitative and quantitative analysis of porosity present in material. Several image processing tools were developed to precisely define porosity distribution on the global, layer and single yarn scale. A 3D mesh representing fiber, resin and porosity geometry has been obtained to better visualize localization of various forms of porosity and serve as a base for future mechanical simulations.

A problem with yarn impregnation has been identified in one principal direction of material and has been precisely described regarding to orientation of yarns and location in thickness of material along with topographic description of resin deficiency in single yarn cross-section. Additional verification was provided by observation of the samples by Scanning Electron Microscopy.

1. Introduction

In recent years polymer matrix composite materials have become much more popular on the markets previously inaccessible due to high costs of project development and manufacture. An analogy to the history of polymers themselves can be made, where the use of structures incorporating these materials was strongly inhibited by the lack of knowledge of materials' mechanical behavior. Limited control over material parameters lead to overconstrained designs and poor performance. In time, increase in comprehension of materials' chemistry, physics and mechanics has lead to development of new technologies. Countless experiments and simulations have allowed to adjust parameters of production to obtain optimal results and create more automatized processes. Now textile reinforced polymer matrix composite materials are entering this higher, more robust level of development. It is possible because of the invention of methods allowing to process thermoplastic matrices. Thermoplastics are easier to recycle and could also be bio-sourced. When these properties are coupled with performance delivered by textile structures from glass, carbon or natural fibers, such composite materials may be regarded as a foundation for innovative and sustainable development.

Further studies of manufacture processes and mechanical behavior of thermoplastic composites will allow to better design structural parts and tailor their production to obtain optimal performance to cost ratio. Along with process automatization, enabled by development of process indicators, these two issues constitute a key to acceptance of woven composites in large scale industries, especially automobile and aeronautical. Thermoplastics reinforced with glass fiber are of particular interest to these two sectors because of their favorable properties when subjected to high speed impact as will be shown in further article being in course of preparation and based on experiments performed on the same material as described here.

Precise description of woven composites has to fully encompass their complexity. Such simplification as is present in description of classical homogenous or orthotropic materials is not possible because it may lead to poor comprehension of materials mechanics. For example, porosity and voids can act as strain concentrators, but giving only the global porosity percentage without indicating its distribution in material is not very informative as has been shown in voids–fatigue life correlation studies in [9]. This study showed correlation between void location and fatigue failure. In our study local characterization of voids has been performed in higher resolution, including microporosity. It has been also

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performed for masked, 3D subregions like single textile yarns. Thorough characterization in three dimensional space could also serve as a diagnostic tool for manufacture process. The importance of porosity characterization is further detailed in ASTM D3171-11 norm [2].

Classically, porosity content has been measured by Archimedes method. It is a very convenient method for testing bulk amounts of material. Its main drawback is that all closed pores are not taken into account, especially micropores. It can also yield non-physical, negative results as it is particularly susceptible to density and principal phase composition information. Another choice for porosity estimation is microscopy, optical or Scanning Electron Microscopy (SEM). It requires a well polished surface to perform good automatic classification. Textile reinforced polymer materials can be very difficult to preparate – polishing grains tend to locate themselves in the matrix and have to be removed e.g. by sonic cleaning. The resulting image features real, manufacture-related porosity as well as that introduced during preparation. There are many other methods for porosity detection like ultrasonic detection and active thermography [12]. In current study though a full characterization of all of the phases was preferred and thus X-ray microtomography has been used.

Today X-ray microtomography provides a complete three-dimensional representation of composite structures. Microtomography studies help with identification of various mechanical parameters, monitoring of crack propagation or characterization of phase composition for different types of materials [4–6,17,18]. With constant development of image processing tools, not only two-phase (material + air) materials like ceramic and metallic foams but also multiphase composites can be examined [8,10,11,14,17].

In microtomography of polymer composites low contrast between fibers and resin makes it hard to automatically classify the results, i.e. to segment resulting images into different phases: fibers, matrix and porosity. Segmented images can be further quantified to determine volume percentages of each of the phases. Several approaches have been used to achieve this. Usually the simplest method by grayscale thresholding and watershed transform is being used [11,13,19]. It performs quite well when the contrast between phases is well pronounced but in current study it was deemed insufficient. Another approach has been used: images were segmented by learning algorithms developed for data mining. Segmented two-dimensional images can be further used to render a three-dimensional volume [1,3]. Rendering allows to visually inspect the results. In our study segmented images were transformed into surfacic mesh, separate for each phase. This has added more flexibility in exploration of class distribution in 3D space and opened a way to mesh optimization and artifact elimination.

The aim of this work is to provide indicators of porosity on different scales. We begin with standard characterization on the global, bulk level followed by characterization in textile and resin layers and concluded with analysis of single yarns. These are examined lengthwise and on cross-sections. To achieve such level of detail a set of image processing algorithms and procedures were developed and applied to the results of X-ray tomography. Obtained 3D mesh reconstruction is planned to be used for further mechanical simulations, possibly with comparison to the results from synchrotron radiation microtomography.

2. Material and methods

2.1. Material

The material was supplied by *Ecole des Mines de Douai*, France. A laminate of thermoplastic reinforced with 8 plies of glass fiber 2/2 twill weaved textile (Tex = 266 g/km) was manufactured by consolidation under pressure during a confidential process. Two materials were considered: P1 with bulk porosity 0.45% measured by Archimedes method and P2 with bulk porosity 1.0% [8]. Six specimens of dimensions 3 × 3 × 10 mm were cut from different locations from manufactured sheets as shown in Fig. 1. To obtain a representative volume element (RVE), the samples were cut side by side by using low-speed specimen cutter set to progress speed of 0.005 mm/s. Low speed ensured minimal amount of introduced damage. Specimens were then submerged in ethanol solution and cleaned ultrasonically to remove possible debris. After cleaning they have been left to dry during 8 h in the oven set to 40 °C. It has been previously verified that ethanol does not impact the integrity of this material.

2.2. Experimental method

2.2.1. X-ray microtomography and image processing

Microtomography scans were obtained at *Laboratoire Matéis* of *INSA Lyon*, France using an X-ray microtomograph [15]. The parameters of acquisition were as follow: Focus-to-Detector Distance (FDD) 577 mm, Focus-to-Object Distance (FOD) 11 mm, X-ray beam acceleration voltage 80 keV. The effective CCD detector area was 1920 × 1536 which with 50 × magnification gave resulting voxel resolution of 2.5 × 2.5 × 2.5 μm. The principle of microtomography is described in [11]. With this type of setup only absorption microtomography was available. Samples were installed on the goniometer as shown in Fig. 2 and scanned with cone-shaped beam in two passes to encompass full length of each sample.

Resulting sinograms were then reconstructed. Obtained were images with coefficients of attenuation of material. Coefficients were related to grayscale: 0 (white) – fibers, 255 (black) – porosity. Further image processing has been mostly performed with ImageJ/Fiji [16] software package and specially in-house developed programs, macros and plugins in Java and in Python.

First, the results were segmented. Unfortunately, due to low contrast caused by relatively low resolution and noise, a standard method based on thresholding followed by median filtering, that is successfully used for processing scans of ceramic and metallic foams [10,13], for current study was deemed unsatisfactory. Instead, a solution based on learning algorithms developed for data mining has been implemented.

A learning algorithms ImageJ/Fiji [16] plugin Trainable Weka Segmentation [7] has been used. Algorithm for segmentation used following training features: Gaussian blur, Hessian, membrane projections, Sobel filter, difference of gaussians. The algorithm of choice was based on FastRandomForest. It divided every image into three classes: matrix (thermoplastic resin), fibers and porosity. The tone range for all results obtained from microtomography was homogenous, which means that coefficients of attenuation were represented by the same pixel values for all scans. On that principle the classifier trained on one representative slice could have been used to classify all of the results.

Thresholding method usually divides image into two classes, like it has been done in other publications [10,11,13,17]. For current study, a thresholding tool was programmed to segment image and provide resulting phase count for 3 or 4 classes. Fourth class was used for masking content during local quantification. Thresholding has been followed by median filtering to remove 1 pixel size noise ("salt and pepper noise") appearing during thresholding of gradient borders between classes and various artifacts in the matrix. The gradient is a result of approximation of values caused by insufficient resolution of the scans. Applied implementation of median filtering has not introduced any new gradients.

A comparison of the results obtained from each of segmentation methods is presented in Fig. 3. A statistical study has been made to