INFLUENCE OF HEATING TIME DURING MOLTEN STATE TESTING OF FLAX/PP COMPOSITES USING THE BIAS EXTENSION TEST

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ABSTRACT

This study examines the effect of heating time on the stress distribution of flax fiber-reinforced polypropylene composites during a bias extension test. A new fixture was designed and manufactured to facilitate the preheating of the oven prior to placing the sample. A finite element model was also developed to simulate the test and compute the stress distribution. Several tests were conducted, including differential scanning calorimetry, thermal tests, and thermo-mechanical bias extension experiments. The thermo-mechanical tests were performed with and without preheating to compare the preheating effect on the test results, and it is shown that the heating time significantly affects the stress distribution.

1 INTRODUCTION

In response to encouraging the use of environmentally friendly and sustainable materials [1], using natural fibers as reinforcements in thermoplastic composite parts that offer the possibility of recycling has increased significantly. As a cost-effective and lightweight material [2], a significant share of the bio-based reinforcements used in the European automotive industry is devoted to flax [3]. Besides, other industries are considering flax fibers as a potential replacement for E-glass [4-8]. Flax fiber-reinforced thermoplastic composites, and among them, spread-tow structured composites, are commonly used in producing parts with advanced structures [9]. As the name implies, spread tow composites are made by spreading fibers into thinner, flatter reinforcements, creating a structure with slight waviness and less matrix, leading to a composite containing a high fiber content and better performance per unit weight than conventional reinforcements [10].

Thermoplastic composites can be shaped into the desired geometry by forming techniques at high temperatures. The method applied here forms components by melting a consolidated product and pressing it into the chosen shape. Performing tests and characterizing the material in similar thermal and mechanical loads to the industrial process is essential to guarantee reliable forming [11]. As one of the dominant deformation modes of composites during forming is in-plane shear [12], such tests should address their in-plane shear behavior. The bias extension test is one of the most common experiments in this field that can be used to characterize the mechanical behavior of composites subjected to large shear deformations [13].

Several studies have been conducted to characterize the in-plane shear behavior of flax fiber-reinforced thermoplastic composites at room temperature in their solid state [14-16]. Other studies have used the bias extension test to evaluate the in-plane shear properties of braided fabrics with flax and thermoplastic commingled yarns at various temperatures and strain rates [17-19]. Lahl et al. provided an example of using the bias extension tests on natural fiber thermoplastic composite laminates. Using this test, they evaluated the in-plane shear properties at different temperatures and strain rates. As forming depends highly on temperature, the tests were conducted at the same temperature that the material undergoes during the forming process. Later, they used these parameters to model the forming behavior of seat shells [11].

However, the characterization of these materials at high temperatures can be more challenging compared to the ambient temperature. A successful bias extension test requires having a temperature balance throughout the sample concerning the desired test temperature [20]. Reaching this temperature requires time, and since natural fibers are less thermally stable than synthetic fibers, the heating time can result in the decomposition of the specimen, which limits their processing time and temperature [11]. On the other hand, non-uniform temperature distributions can cause undesired thermal stresses within the sample, which is ignored in most studies.

This study compares the effect of the heating time on the stress distribution in thermoplastic composites during a bias extension test. A new fixture is designed and manufactured for this aim, as explained in Section 2. With this fixture, it is possible to preheat the oven before placing the sample. Multiple tests are performed to compare the effect of preheating on the test results. Thermocouples are connected to the sample in each test to record the temperature history. Since one of the requirements for accurately determining the material characteristics is having a reliable numerical model, a Finite Element (FE) model, incorporating both the sample and the fixture, is developed in Section 3. The results of linking the experimental results with the finite element model are presented in Section 4. The last section of the paper summarizes the conclusions.

2 FIXTURE DESIGN AND EXPERIMENTS

In the commonly used bias extension tests, the sample is installed in the oven before the heating stage starts. In this case, the sample experiences either a temperature gradient or thermal stresses if the mechanical load is applied before reaching a uniform temperature distribution. On the other hand, giving more time to reach a uniform temperature distribution throughout the sample may cause material degradation. Accordingly, a new fixture is designed in this study to overcome these problems by preheating the oven to minimize the amount of time that it takes for the sample to reach the desired temperature. This helps to ensure that the temperature is uniform and that the sample is not exposed to extreme temperature fluctuations. Figure 1 shows the designed fixture. As seen, part A can be mounted in the oven before starting the heating stage, while part B can be placed after reaching the test temperature.

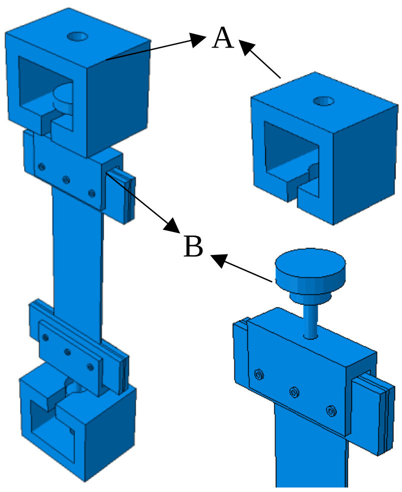

Figure 1: Designed fixture including, (A) fixed parts that are installed in the oven before starting the heating stage and (B) removable parts of the fixture with the sample.

First, Differential Scanning Calorimetry (DSC) analysis was performed to determine the melting temperature of the testing material and its specific heat capacity. Measurements were conducted at a heating rate of 10 ºC/min from -50 ºC to 250 ºC, followed by cooling at the same rate. The obtained peak value for the melting temperature was used as a reference for the molten state test temperatures, and the specific heat capacity values were used as inputs for the thermal FE model.

Since the temperature of the samples changes from room temperature to test temperature during the heating stage of the bias extension test, a series of tensile and shear tests based on ATSM D3039 and ATSM D3518 standards were conducted at room temperature to determine the elastic material properties of the composite, which were then used as inputs in the FE model.

Before conducting the thermo-mechanical tests, a thermal test was performed to record the temperature history of several locations on the specimen. Later, three test cases were conducted to investigate the effect of preheating. During the first thermo-mechanical case, the oven was preheated for two hours before starting the test to achieve the uniform distribution of the desired temperature. Then, the sample was quickly placed in the oven by opening the door and positioning it inside. In this case, the loading started with a 10-minute delay to compensate for the temperature drop during the sample placement and to reach a uniform temperature distribution throughout the sample. The tests were performed without preheating in the second and third thermo-mechanical cases, where the sample was placed in the oven from the beginning, i.e., at room temperature. In the second case, the mechanical load was applied after the desired temperature was reached within the oven (after about 20 minutes), while in the third case a similar waiting time to case 1 (10 minutes) was considered before the application of the mechanical load. This procedure evaluates how the sample behaves during the heating stage. The results are compared and analyzed to understand the effect of temperature on the sample's mechanical properties. Table 1 presents the different test cases.

Type of test		**Target**
DSC		Determining the melting temperature and specific heat capacity
Mechanical tests at room temperature		Determining elastic properties at room temperature
Thermal test		Determining the temperature history and boundary conditions for the thermal FE model
Thermo-mechanical tests	**Case 1**	Bias extension test with preheating (waiting to reach a uniform temperature of 170 ºC)
	Case 2	Without preheating (waiting to reach a uniform temperature of 170 ºC)
	Case 3	Without preheating (waiting for the same time as in case 1)

Table 1: Summary of the performed experiments.

An Instron universal testing machine with a TA Instrument climate chamber was used for the thermo-mechanical tests (Figure 2). The samples were cut from a four-ply W8SVR® Woven composite sheet (W8SVR GmbH, Germany), made from polypropylene matrix, and reinforced with spread tow woven flax fibers (40% fiber volume fraction) with an average thickness of 2.2 millimeters. During the experiments, the fibers were oriented 45 degrees with respect to the applied load, and the displacement rate was 10 mm/min. In each case, several type-K thermocouples were connected to the sample to measure the temperature history. The measurements were logged using the 8000 Micro-Measurements data logger with a data sampling frequency of 1 Hz.

3 FINITE ELEMENT MODEL

A finite element model of the sample and the fixture was developed to investigate the effect of the heating time on the temperature and stress distributions. Mesh sensitivity analysis was done to ensure that the solution is independent of the mesh size, resulting in a model of 133628 elements. As in a previous study of the authors, the experimental and numerical temperature distributions were compared to calibrate the finite element model and determine the correct thermal boundary conditions [20].

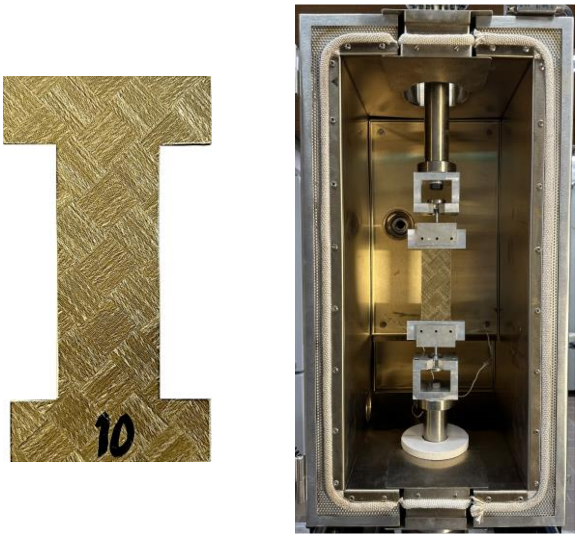

Figure 2: (left) Flax/PP test sample; (right) manufactured and assembled fixture.

For the mechanical part, an orthotropic elastic behavior ($\boldsymbol{\sigma}^e = \boldsymbol{D}^e \boldsymbol{\varepsilon}^e$) was assumed between stress ($\boldsymbol{\sigma}^e$) and strain ($\boldsymbol{\varepsilon}^e$), where $\boldsymbol{D}^e$ is defined as:

$$\boldsymbol{D}^e = \begin{bmatrix} \frac{1}{E_1} & \frac{-\nu_{21}}{E_2} & \frac{-\nu_{31}}{E_3} & 0 & 0 & 0 \\ \frac{-\nu_{12}}{E_1} & \frac{1}{E_2} & \frac{-\nu_{32}}{E_3} & 0 & 0 & 0 \\ \frac{-\nu_{13}}{E_1} & \frac{-\nu_{23}}{E_2} & \frac{1}{E_3} & 0 & 0 & 0 \\ 0 & 0 & 0 & \frac{1}{G_{12}} & 0 & 0 \\ 0 & 0 & 0 & 0 & \frac{1}{G_{13}} & 0 \\ 0 & 0 & 0 & 0 & 0 & \frac{1}{G_{23}} \end{bmatrix} \quad (1)$$

where E_i, G_{ij}, and ν_{ij} are Young's modulus, Shear modulus, and Poisson's ratio, respectively.

A linear function with temperature was defined for the tensile and shear modulus to represent the temperature-dependent behavior as $E_i = E_{i,0} + E_{i,1}T$ and $G_{ij} = G_{ij,0} + G_{ij,1}T$, where $E_{i,0}$, $E_{i,1}$, $G_{ij,0}$ and $G_{ij,1}$ are constant material properties. These constants were defined by comparing the variations in force and shear angle (in the middle of the specimen) of the experiments with the equivalent numerical model. Then, the developed model was used to compute the stress distribution for the different test cases corresponding to various heating approaches.

4 RESULTS

This section presents the results of the experiments provided in Table 1. A DSC instrument was used to determine the melting temperature of the samples. Figure 3 shows the variations of normalized heat flow to the applied temperature. According to the results, the peak value for the melting temperature of the tested material is 170 ºC. Therefore, the thermo-mechanical tests were conducted at this temperature.

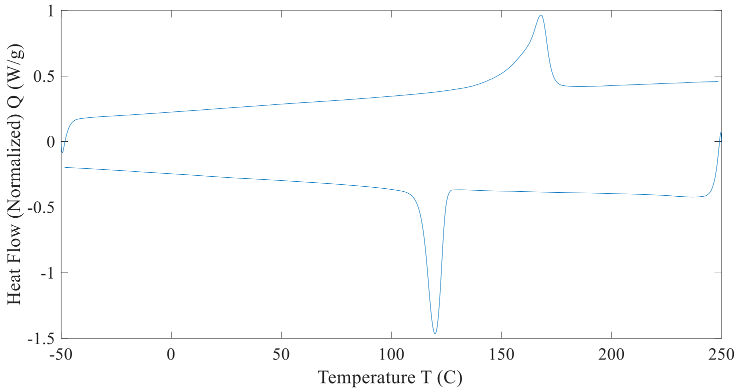

Figure 3: DSC measurement of a heating-cooling cycle of a flax/PP composite laminate.

In the following step, a simple thermal test was conducted to obtain information on the sample's thermal history during the bias extension test. Three thermocouples were connected to the sample to measure the temperature of the mid-sample, at five millimeters above the lower fixture and at five millimeters lower than the upper fixture. As shown in Figure 4, the sample takes approximately 10 minutes to reach 170 ºC. This time was used as a reference for the thermo-mechanical test with preheating, where we waited for 10 minutes to reduce the temperature gradient (case 1). Additionally, in case 3, the waiting time before applying the mechanical load was also considered as 10 minutes to make a comparison with case 1.

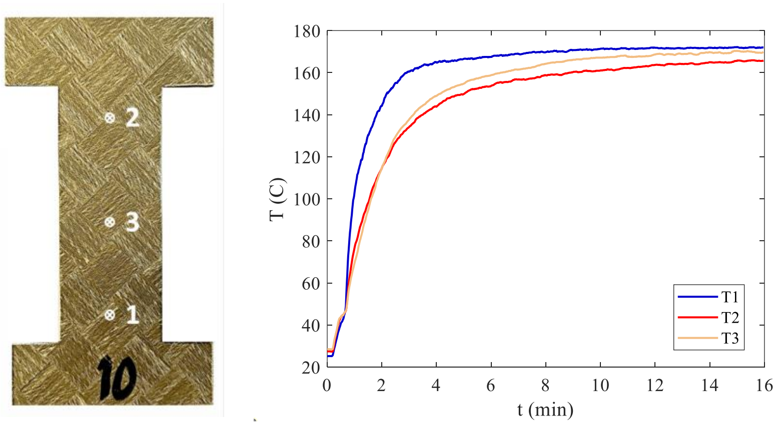

Figure 4: Temperature variation of the specified points on the sample after placing it in the preheated oven.

Table 1 presents the three thermo-mechanical test cases that were conducted to study the effect of preheating time. Based on the observations in case 2, the sample obtained high deformation due to the high level of degradation and failed immediately after being subjected to the mechanical load. Thus, in the following steps, the results for cases 1 and 3 are compared, which are referred to as "with preheating" and "without preheating" cases, respectively.

Figure 5 compares the finite element and experimental force-shear angle variations for both cases. The shear angle in the middle of the specimen in both the numerical and the experimental methods is calculated based on the theoretical shear angle equation:

$$\gamma = \frac{\pi}{2} - 2arccos\left(\frac{(H-W)+u}{\sqrt{2}(H-W)}\right) \tag{2}$$

where H and W are the specimen's height and width before deformation, respectively, and u is the applied vertical displacement [13]. This comparison is used to extract the material behavior at high temperatures in the elastic region. As shown in Figure 5, there is a good alignment between experimental and numerical results. In addition, by comparing the two cases in Figure 5, it is evident that the amount of force required to maintain the same shear angle changes significantly due to the heating process.

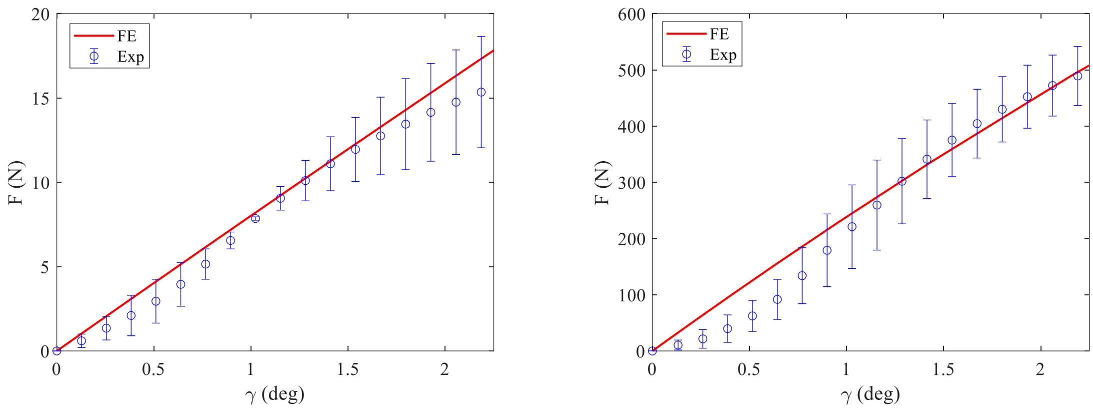

Figure 5: Finite element and experimental force-shear angle variations for specimens (left) with preheating and (right) without preheating during the mechanical loading in the elastic zone.

Figure 6 illustrates a comparison between the temperature distribution within the fixture and the sample for cases with and without preheating. The second case shows that the sample does not reach the uniform temperature of 170 ºC. Moreover, the difference between the minimum and the maximum temperature along the sample in the case of preheating is significantly lower.

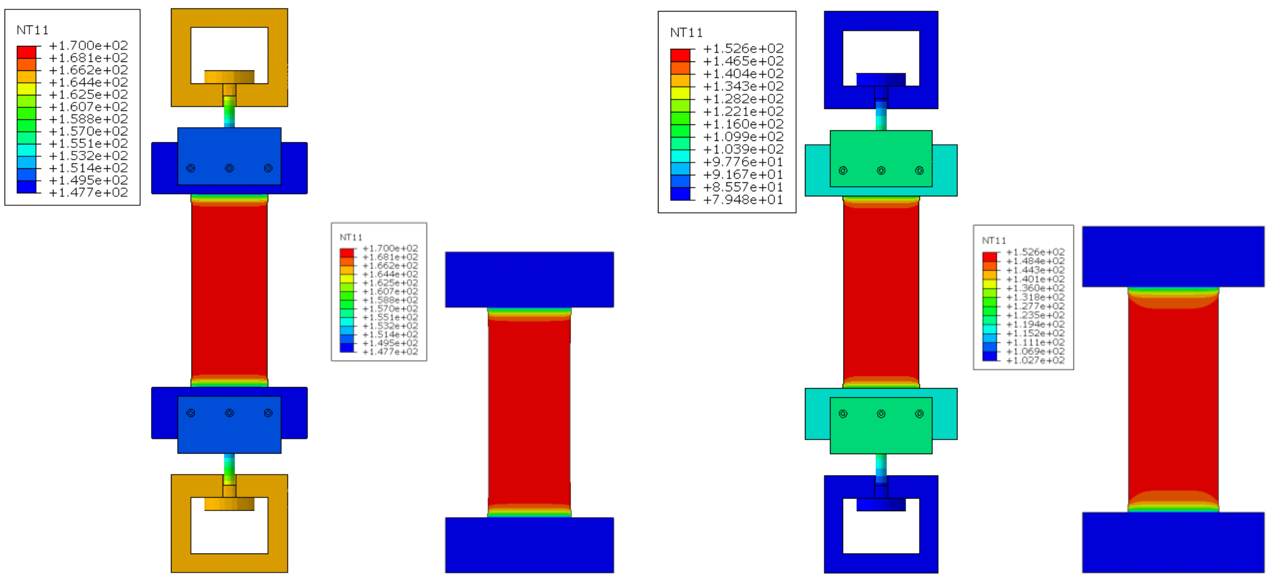

Figure 6: Temperature distribution in the fixture and the specimen (left) with preheating and (right) without preheating during the mechanical loading in the elastic zone.

In the following step, the temperature distribution was utilized to simulate the stress distribution within the sample. Figure 7 shows the equivalent stress distribution for the cases with preheating and without preheating. The higher amount of stress for the case without preheating can be related to the presence of thermal stresses due to a larger temperature gradient, as well as different elastic material properties since the sample's temperature is lower.

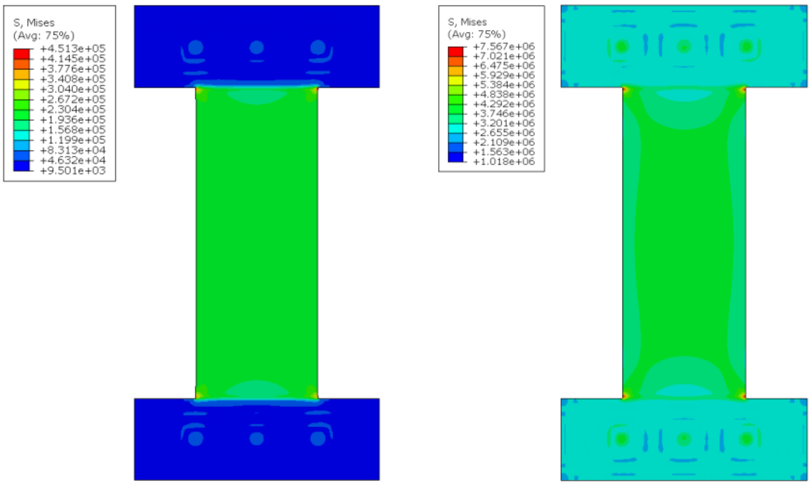

Figure 7: Stress distribution in specimens (left) with preheating and (right) without preheating during the mechanical loading in the elastic zone.

Figure 8 compares the equivalent thermal stresses for the experiments with and without preheating assuming no mechanical load has been applied in the finite element model. As shown in this figure, the preheating reduces the thermal stresses significantly.

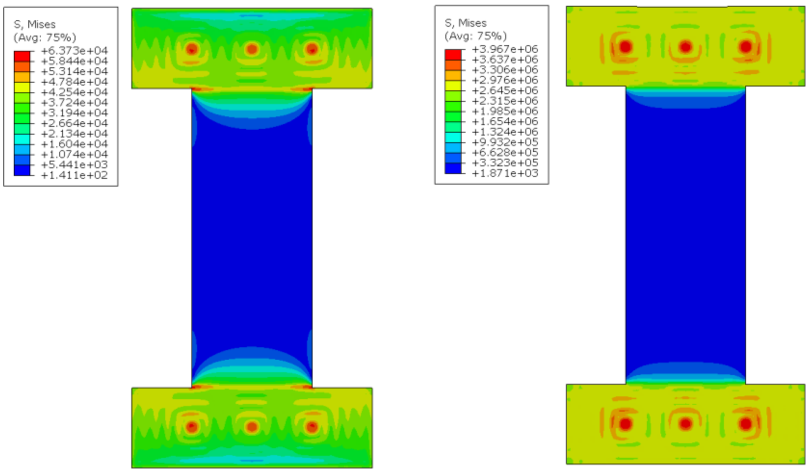

Figure 8: Thermal stresses in specimens (left) with preheating and (right) without preheating.

5 CONCLUSIONS

This study determines the elastic material properties and the effect of preheating time on the bias extension test results of flax/PP composite samples. For this aim, first, differential scanning calorimetry (DSC) analysis was performed to determine the melting temperature. Then, using the bias extension test setup, the temperature history was recorded in a thermal test using three thermocouples by setting the oven temperature equal to the melting temperature (170 ºC). In the next step, the effect of preheating time was investigated by conducting bias extension tests. The results show that without preheating, it takes more than 20 minutes to reach the uniform temperature distribution, after which the sample fails due to severe degradation. However, the proposed fixture in this study made the preheating of the oven possible, and more importantly, it reduced the required time to reach uniform temperature distribution within the specimen to 10 minutes. Moreover, preheating the oven reduces the required time for the subsequent tests in a series, as changing the sample can be done without cooling down the oven. The experimental results are linked with a finite element model to study the thermo-mechanical stress distributions. The finite element simulation results show significantly higher thermal stresses for the case without preheating. Additionally, the extracted variations of load versus shear angle in both experimental and finite element approaches show a good agreement.

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