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# Performance enhancements of polymer-matrix composites by changing of residual stresses

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#### ABSTRACT

Manufacturing induced residual stresses of polymer–matrix composites (PMC) reduce the tensile load at which first ply failure occurs. Thermomechanical treatments offer the potential to change these residual stresses, but their application is hindered because the shape stability of PMC components is limited at treatment temperatures, which must be above the glass transition temperature of matrix. This study describes successful localized treatments performed on a stress concentration induced by a circular hole in a laminate. Localization of the treatment allows significant property improvements at much lower treatments loads. Investigation of the influence of moisture pick up on treatment effectiveness revealed it reduces the benefits of treatment to a large extent. Calculations of local stresses between single fibres and predictions of laminate properties based on laminate plate theory using a quadratic failure criterion confirm the experimental results and provide confidence in the physical explanations offered for the measured effects.

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#### 1. Introduction

Most of applied polymer-matrix composites (PMC) designs consist of many plies, which are dictated by the stiffness and strength requirements of the component or product. The single ply is characterized by anisotropic behaviour and very large property differences such as stiffness and thermal expansion between the fibre and the matrix. In high performance PMCs, these characteristics give rise to large internal residual stresses even immediately after manufacture, in the unloaded case at room temperature [1–5]. These stresses are fundamentally due to curing at elevated temperature. When injection technology is used instead of Prepreg technology, there is an additional source of residual stresses: matrix shrinkage [6]. During usage of a laminate, these internal stresses are changed by moisture pickup and the resulting volumetric expansion of the matrix. These effects depend on environmental conditions, time of exposure, diffusion coefficient and geometric parameters [5,7]. Finally, the loading itself can induce additional inhomogeneities in the stress/strain distribution, particularly in those plies with reinforcement-oriented transverse to the primary loading direction [5]. The superposition of all of these spatially varying stresses results in a very inhomogeneous stress distribution and creates local stress concentrations in the matrix throughout the whole laminate. These stress concentrations in turn result in a much lower tensile load sustained at first ply failure stress/strain than the load sustained at final failure stress/strain [8–10]. This fact prevents a designer from fully exploiting the potential weight savings of PMCs, because in many applications, the components should be designed so as to preclude ply failure at any location. First ply failure makes the components more vulnerable to subsequent degradation, for example accelerating environmental effects (e.g. moisture pick up) as well as fatigue crack growth [11–16].

In the past this problem has been accepted, due to the lack of a simple solution. Approaches to change the internal stresses of PMC by applying thermomechanical treatment have been very successfully applied to laboratory scale specimens or simple components [17–26]. However, applications to large or complex components have been limited, primarily due to problems concerning components' shape stability during relatively high stress loading at temperatures above glass transition. This is a fundamental step in the application of thermomechanical treatments.

In this study, thermomechanical treatments were performed so as to localize the treatment to failure critical areas, thereby reducing the overall load on a component during treatment. Additionally, the influence of residual stress change by moisture pick up on untreated and differently treated specimens was investigated. The intent was to get a real view of potential property improvements under various service conditions by alteration of residual stresses. The results of this study should also provide insight into the potential property improvements which may result from





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future modification of matrix materials to contain nanoparticles [27–30]. Such matrix materials are thought to have less thermal expansion and/or less moisture pick up than conventional ones.

#### 2. Experimental

#### 2.1. Materials and specimens preparations

All investigations in this study were performed with a composite consisting of continuous carbon fibres T800 in a thermosetting matrix Cycom 5245C, former trade name NARMCO 5245C, (Resin type: Modified cynate ester), because most characteristics of this PMC needed for predicting first ply failure and micromechanical calculation of local stresses and for achieving thermomechanical treatment were available from previous studies [21,24,25]. The fabricated laminates had a quasi-isotropic lay-up [(+45°)0°)–45°/ 90°)<sub>2</sub>]s. A fraction of them were completely cured (curing cycle to manufacturer's specification + post-curing), the remainder were produced using a shortened curing cycle (reduction of curing time = 30%) without post-curing.

From these laminates, specimens of 36 mm by 280 mm were cut. A certain number of specimens were notched *before* thermomechanical treatment by drilling a central hole of a diameter of 6 mm (notched specimen) while being water-cooled. The remaining specimens were similarly notched *after* thermomechanical treatment. Before thermomechanical treatment and testing in dry condition or storage in defined climate, specimens were stored in a dry environment at 70 °C for three days and than at 90 °C until constant mass was reached. A certain number of untreated and treated specimens were moisturized by storage in a constant climate of 70 °C/95% relative humidity for 200 days. The moisture pickup was measured by weighing.

#### 2.2. Thermomechanical treatment of specimens

A load frame designed for uniaxial creep testing at elevated temperatures up to 250 °C and tensile loads up to 100 kN was used for thermomechanical treatments. In general, these treatments were all based on the following steps, namely quick heating up to treatment temperature, uniaxial loading at this temperature, cooling down under load and finally releasing from the treatment load. Different treatments were applied to completely cured specimens (Treatment A) and to partially cured specimens (Treatment B). In the first case, the treatment loading was performed above the glass transition temperature for only a short period of time (details

Table 1

Conditions of thermomechanical treatments

in Table 1). In the second case, the specimens were held under reduced load at curing or post-curing temperatures until complete cure occurred. The load was then increased to a level higher than that applied in Treatment A (details in Table 1). The treatments were applied to both notched and unnotched specimens. Unnotched specimens were notched following treatment. The determination of proper treatment conditions was described in detail in [21,24,25].

#### 2.3. Determination of properties before and after treatment

Uniaxial tensile tests were performed according to DIN 65559, in order to determine the number of transverse cracks as increasing load was applied to the specimens. During these tests, specimens were loaded stepwise in increments (500 N for untreated and 1000 N for treated specimens) and monitored via acoustic emission (AE) measurements until rupture occurred. After receiving the first AE signals, the specimens were removed from the load frame and prepared for radiographic examination. The specimens were contrasted by storage in a contrast fluid (zinc iodide dilution containing 1200 g zinc iodide, 160 ml H<sub>2</sub>0, 200 ml wetting agent Agepon, 200 ml isopropanol) in a vacuum chamber for 4 h. The contrasted specimens were then radiographed and the resulting images examined for indication of transverse cracks. The specimens were installed back into the load frame and loaded to the next load step, removed and radiographed again. This procedure was repeated until delamination occurred. After the onset of delamination the specimens were loaded up to final rupture and notch strength was recorded.

#### 3. Results

#### 3.1. Moisture pick up of untreated and treated specimens

Fig. 1 illustrates the moisture pick up of untreated and differently treated specimens versus storage time. It can be seen that treated specimens had less moisture pick up than the untreated ones, but there is essentially no influence between various types of treatment. The continuing moisture pick up over time is typical for the type of matrix used.

#### 3.2. Tensile properties

Fig. 2 compares average laminate strains at first ply failure measured by AE on untreated and differently treated specimens in both

Type of treatment/step		Unnotched specimen			Notched specimen		
		Temperature (°C)	Stress (MPa)	Duration (min)	Temperature (°C)	Stress <sup>a</sup> (MPa)	Duration (min)
A	Heating up	23-210	0	15	23-210	0	15
	Loading	210	250	5	210	133	5
	Cooling down	210-23	250	15	210-23	133	15
	Unloading	23	250-0	1	23	133–0	1
В	Heating up	23-180	0	15	23-180	0	15
	Loading	180	222	115	180	117	115
	Heating up	180-190	222	5	180-190	117	5
	Loading	190	222	115	190	117	115
	Heating up	190-200	222	5	190-200	117	5
	Loading	200	222	115	200	117	115
	Heating up	200-210	222	5	200-210	117	5
	Loading	210	222	235	210	117	235
	Loading	210	306	5	210	167	5
	Cooling down	210-23	306	15	210-23	167	15
	Unloading	23	306-0	1	23	167–0	1

<sup>a</sup> Average stress at the smallest cross section.

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