



# The influence of the laminate thickness, stacking sequence and thermal aging on the static and dynamic behavior of carbon/epoxy composites



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## ABSTRACT

In this work, we investigate the influence of laminate thickness, stacking sequence and thermal aging on the damping behavior of carbon/epoxy laminates. The dynamic behavior of quasi-isotropic and unidirectional laminates of different thickness, are measured by means of Laser Doppler Vibrometry and Dynamic Mechanical Analysis. Obtained results before and after aging at 175 °C for 250 and 500 h, are compared with static strength properties. The Laser Doppler Vibrometry measurements of the modal damping factors, show general increasing tendency for the unidirectional composites after aging, but decreasing tendency for quasi-isotropic plates. The DMA damping measurements reveal an increase of the damping in longitudinal direction after aging, but a decrease in transverse direction. Comparison of results of dynamic measurements with static properties, confirm that mechanism of damping in composite structures depends mainly on stacking sequence. Moreover, modal damping of unidirectional composites depends mainly on the properties of the fiber–matrix interface, while damping of the quasi-isotropic laminates is affected by long-term properties of the matrix. Thus, to ensure long term damping performance of the carbon/epoxy components subjected to vibrations, one should take into account the stability of the matrix and fiber–matrix interfaces and also the alignment of the reinforcing fibers.

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## 1. Introduction

Fiber reinforced polymer composites are increasingly being used in a wide range of load bearing applications. The long term application, requires the use of accelerated aging regimes to validate and guarantee the useful lifetime of the components [1]. The long term usage of the composite materials, require not only stability of the strength properties but the predictable damping properties as well, which are crucial for the noise, vibration and harshness (NVH) analysis. The NVH analysis is gaining more and more attention from designers, who these days make efforts to minimize the detrimental influence of the vibrations by usage of carbon/epoxy composite materials. Carbon/epoxy composites are the example of high-end materials, known for their low density and considerable stiffness and strength to weight ratio, that have recently entered the transportation sector [2]. This group of materials, e.g. fibrous laminates, are also known for their higher damping capabilities compared to metals traditionally used in vehicle design [3], which results in

improved vibration behavior. The energy dissipation capabilities are influenced by many factors, which in the case of laminates include fiber and resin types, fiber volume fraction or the number and orientation of layers. Maher et al. [4] reported the significant effect of the angle orientations of the outer laminate plies, on the modal parameters of the composite. The interaction between reinforcement and matrix materials in the interface region also contributes to the process, being caused by friction between the phases. However, the most influential factor that increases the damping properties of laminates is the stacking sequence [5]. The composite damping mechanisms and analytical models for the prediction of damping, are described in detail in a review article [6].

The stability and predictability of changes of damping properties of carbon/epoxy composites are crucial during the designing of structural parts. In engineering applications, structural parts made of carbon/epoxy materials, frequently work at high temperatures, which leads to thermal aging. Recently there have been reported several experimental results on the influence of thermal aging on composite strength properties. The short review on the influence of the thermal aging on carbon/epoxy composites is presented in the next chapter. However, there is still a lack of experimental results, regarding the effect of thermal aging on the damping properties.

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The objective of this work is to investigate the influence of thermal aging on the dynamic properties of carbon/epoxy composites after accelerated aging profiles, typical for engine compartment applications in the automotive industry. The dynamic behavior of complex composite structures is described by modal damping ratio. Modal damping ratio is a fraction of critical damping, defined for each vibration mode independently. This value can be calculated based on modal analysis results and is expressed as a function of frequency. In presented research, modal damping properties investigated by means of Laser Doppler Vibrometry (LDV) and calculated using half-power method are compared with DMA results and static tension strength tests for different stacking sequences. The results showed that changes of the modal damping in unidirectional laminates correlates with the alterations of material damping and strength properties in longitudinal direction. Composite properties in longitudinal fiber alignment depend strongly on the fiber–matrix interface. On the contrary, damping properties of the quasi-isotropic laminates correlate with the material loss factor and strength properties in transverse direction, which depends on matrix properties. We demonstrated, how the stability of damping properties of carbon/epoxy laminates, depends on the reinforcement direction, during operation in the harsh environments.

## 2. Thermal aging of the carbon/epoxy composites

Durability of carbon/epoxy composite structural components, working in engine compartments, is significantly affected by thermal aging. In engineering practice, to investigate high temperature durability of these components in the automotive industry, one can use accelerated aging tests. Experimental investigations of the carbon/epoxy and carbon/vinylester composites after accelerated aging, shows a two-step trend of the variation of the mechanical properties [7]. During the first stage, called the consolidation phase, improvements in the tensile mechanical properties are observed, which are related to the post-curing process. The second, degradation stage, is characterized by significant decreases of mechanical properties, due to the oxidation of the epoxy matrix and degradation of the fiber–matrix interface. The oxidative aging of the epoxy resin, consist of three phases [8]: “A first phase, dominated by the matrix viscoelastic behavior and by stress relaxation at high temperature”; “a second phase, characterized by thermo-oxidation effects”; and a third phase, with dominating development of microcracks. The thermal treatment causes irreversible structural changes in polymer matrix, like: enlarged air bubbles and cracks [9,10]. These defects, caused by matrix shrinkage, influence the strength properties [11], as well as moisture and oxygen diffusion process.

The long term aging of carbon/epoxy composites, shows that elevated pressure significantly reduces screening times for materials subjected to temperature aging in oxidative environments [12]. Although, it must be remembered that due to the diffusion controlled oxidation mechanism, the rate of degradation depends on the stacking sequence [13]. The thermo-oxidation of the composite results in damage mainly on the skin of the specimen [14] which may not affect the mechanical properties, if the load is transmitted by inner plies. In [15] the author reports an unchanged slope of the stress–strain curves for specimens with longitudinal fiber alignment, while the mechanical strength decreases during aging time. Additionally, the linear relationship between mass loss and mechanical strength decrease is observed due to the oxidation. The thermo-oxidation process changes the strength properties of epoxy resin. In article [16] the authors present results of the study of oxidation at 150 °C, under vacuum and ambient air, by means of the Ultra Micro-Indentation (UMI). Elastic modulus, measured by UMI, increases with time of isothermal aging under atmospheric

air, which corresponds with concentration of oxidation products. Other studies of the variation of mechanical properties resulting from thermo-oxidation are presented in [17]. Oxidative-aging at 177 °C results in increasing mode I strain energy release rate  $G_{IC}$ , and decreasing mode II strain energy release rate  $G_{IIc}$ . These phenomena are caused by matrix degradation which reduces the resistance of the material to matrix-controlled failure modes, like: delamination and transverse cracking. These results prove the leading role of matrix and matrix–fiber interface on aging behavior. Studies of compressive properties reported in [18] shows that the ultimate strength and modulus decrease with aging time for both: plain and multi-hole laminates. Decreases in the compressive strength and modulus are greater for perforated laminates, due to the accelerated oxygen diffusion. The mechanical properties of carbon/epoxy composites are affected additionally by the elevated pressure [19]. The thermal degradation under elevated pressure accelerates the degradation process. Thermal aging influences not only static material strength but fatigue behavior as well. In [20] the authors report experimental investigation and model of degradation of fatigue strength of carbon fiber reinforced rings. Modification of the fatigue strength is modeled by means of the Arrhenius equation, in the same way as we predict the changes of viscoelastic properties of PDMS materials during the aging time [21].

Thermal aging affects short-term behavior, but what of the long-time visco-elastic behavior? In article [22] the authors investigated viscoelastic properties of carbon/epoxy composites by measurement of indentation print shape after UMI. Measurements were done by means of Confocal Interferometric Microscopy (CIM). The UMI measurements gave load vs. displacement curves to gain information about local short-time behavior, while the CIM observations provided some information about material relaxation. For studied resins, the UMI showed significant stiffening of the epoxy matrix with oxidation level. The CIM showed that aging affects the material viscoelasticity, which is visible in the time of recovery.

Despite a large amount of articles on thermal aging, there is still an open research question: how thermal aging influences the damping behavior of carbon/epoxy composites and how it is related with composite layup and strength properties? The experimental results of LDV, DMA and static strength tests for different laminays after elevated temperature aging are discussed in the next sections.

## 3. Materials and methods

All tests were carried out with the carbon/epoxy laminates from CG TEC Carbon & Glasfasertechnik GmbH (Spalt, Germany). The laminates used in this study had different lay-ups. Unidirectional lay-ups ( $UD_{4T}$  and  $UD_{8T}$ ) 1 and 2 mm thick, were chosen to investigate the influence of the thermal aging on dynamic and static properties in tension with 0, 90 and 45° directions. The Quasi-Isotropic  $[0/45/-45/90]_{25}$  laminate (QI) – 2 mm thick was taken to investigate the influence of aging on dynamic and static strength properties. The fiber volume fraction of each laminate is approximately 56%. Samples were cured at 140° for 90 min under a pressure of 5 bars with temperature increase between ambient and the cure temperature at 0.5–0.8 °C/min. Temperature decrease between cure temperature and ambient was equal to 1 °C/min. All laminate plates were controlled in case of any defects that could have influenced the measurements. The glass transition temperature ( $T_g$ ) of investigated composite is about 120 °C. After processing the UD and QI laminates were isothermally aged in an oven at 175 °C for 250 h. Thermal treatment above ( $T_g$ ), but well below the temperature of thermolysis, results in thermal rejuvenation [23], which ensure that physical aging history is removed. In order to gain an additional insight into the changes of composite properties, part of

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