Monitoring techniques for carbon fibers reinforced plastics used as complex structures

A Savin\textsuperscript{1}, R Sturm\textsuperscript{2}, Z Bergant\textsuperscript{2}, M D Stanciu\textsuperscript{3}, R Steigmann\textsuperscript{1} and G S Dobrescu\textsuperscript{1}

\textsuperscript{1}National Institute of R&D for Technical Physics, Nondestructive Testing Department, 47 D. Mangeron Blvd, Iasi, 700050, Romania
\textsuperscript{2}University of Ljubljana, Faculty of Mechanical Engineering, Aškerčeva c. 6, 1000 Ljubljana, Slovenia
\textsuperscript{3}Transylvania University, Faculty of Mechanical Engineering, 29 Eroilor Blvd, 500036, Brasov, Romania

E-mail: asavin@physiasi.ro

Abstract. Carbon fiber reinforced plastics (CFRP) have become an indispensable part of modern life. CFRP materials continue to be used in a large number of applications ranging from aerospace systems to automotive, industrial and consumer products. CFRP have evolved both in reinforcement and matrix. The epoxy resin was the most usually matrix for CFRP. The reliability of these materials is essential, especially when it comes to safety-relevant components. The service life of CFRP components is affected by aging processes or improper use. Nondestructive evaluation methods which can be applied for examination carbon/epoxy laminates, manufactured by autoclave processing method with different ply stacking sequences \([0]_4\) and \([(45/0)_2]\), are presented. The samples were realized from 4 plates of carbon-epoxy manufactured at the Faculty of Mechanical Engineering, University of Ljubljana, Slovenia. The paper present influence of thermal treatment and stacking sequence on damage delamination's due to impact about mechanical properties. Ultrasound and electromagnetic nondestructive evaluation methods are used for good localization of damage and characterization composite materials and other techniques can provide complementary information once the damage location is known.

1. Introduction

The fiber reinforced polymers (FRP) are composites with reinforcing fibers embedded in a polymeric matrix. These are responsible by the distribution of material loading, the matrix being only a protection for fibers [1]. The FRP have heterogenous and anisotropic properties, behaving linear elastin in the limit of applied forces. The most usually fibers are glass fibers (GFRP) and carbon fibers (CFRP), which can be assembled in filiform geometry, are continuous and can have between 4 and 10 μm diameter. Between them, CFRP has higher value of stiffness and maximum stress. In the structure of reinforcement, the fibers can be assembled in unidirectional, bidirectional and multiaxial fabrics. CFRP have become an indispensable part of modern life. CFRP materials continue to be used in a large number of applications ranging from aerospace systems to automotive, industrial and consumer products. CFRP have evolved both in reinforcement and matrix. The epoxy resin was the most usually matrix for CFRP. The reliability of these materials is essential, especially when it comes to safety-relevant components. The service life of CFRP components is affected by aging processes or improper use. Damages such as cracks in matrix, delaminations, dry areas, or fiber breakage can lead to...
component failure. Due to the heterogeneous nature of composites, the shape of defects is very often different from those typically of metallic material and the fracture mechanisms are more complex. Damage in composites due low velocity impact occurs over a large of time and length scale.

It has been shown in literature that if after tests with low energy impact, a space-time scale of damages that appears is realized, it can be observed that fibers failure and matrix cracks are at the same level with the quasi-static residual stress states after impact. This study shows that for the monitoring of damage evolution in composite material, a “local-global” frame must be thought [2].

Residual quasi-static stress states after impact or as a result of the equilibrium state following the manufacturing process can be the cause of the subsequent damage events. The order of progression plays an important role in the general characterization of the composite, so that the monitoring becomes an essential component of the analysis of the composite’s structural health.

The high costs of CFRP have been surpassed by their strength-to-weight ratio and feasibility. The CFRP structures in aviation/aerospace industry makes compromise between advantages – weight reduction, the mechanical properties can be tailored by layout design, high resistance to corrosion at interfaces with metallic joints and disadvantages as high recurring costs, barely visible defects appearance and their propagation, difficulties in repairing, raising the requirement of testing during the fabrication of structures as well as in service monitoring. The criteria for materials acceptance moved the practice of non-destructive evaluation into a new position of reliable detection and sizing of flaws, using conventional strain gages [3], ultrasound using different types of sensors and methods, PZT sensors, thermography and acoustic emission [4] and also sensors for electrical properties [5], wireless sensing with low sampling rate [6], wireless strain and temperature sensors, especially knowing that carbon fibers have electrical properties and these properties are modified by the effect of impact with low energies [7]. Due to the numerous types of layups and layouts in configuring the composites in function of layers number, orientation of fibers and woven, type of matrix, etc, the full effect of design over the behavior of the composites at impact isn’t yet elucidated, different designs leading to the development of different control methods [6, 8]. Failure initiation in such assemblies is often associated with crack initiation in the adhesive, or in composite layers close to the adhesive bonding caused by efforts made differently by the bonding components [9].

The paper presents NDE methods which can be applied for examination CFRP laminates, manufactured by autoclave processing method realized at the Faculty of Mechanical Engineering, University of Ljubljana, Slovenia. The paper present influence of thermal treatment and stacking sequence about mechanical properties of composites. Ultrasound (US) and electromagnetic (EM) nondestructive evaluation methods are used for characterization composite materials and other techniques such as Dynamic Mechanical Analyzer DM can provide complementary information.

2. Samples and experimental set-up

2.1. Studied samples
CFRP properties can be controlled by proper selection of substrate parameters by fiber orientation, volume fraction, fiber spacing and layers sequences, etc. CFRPs are obtained by choosing carbon fibers as reinforcement and plastics as matrices. The CFRP structure may undergo modifications both in the process of obtaining (by forming voids or the variation of process parameters) as well as in operation when delaminations due to the impact with high and low energies, possibly accompanied by the breakdown of carbon fibers, may occur; local overheating that can damage the matrix locally. For the optimal design of composite structures, the mechanical properties of the materials must be known.

CFRP materials involved into study for the first stage are represented by 4 plates, made from 8 plies of plain weave fabrics GG200P, manufactured by autoclave processing method with different ply stacking sequences (two for each) respectively [0]_8 and [(45/0)]_8s [10]. From composite plates having dimensions 295x205x1.91mm³ (figure 1), samples with 50x10x1.91mm³ were cropped, in order to determinate the elastic and shear moduli along the directions 0° and 45° using Dynamic Mechanical Analyzer DMA 242C – Netzsch Germany, with 3 points bending testing fixture.
Figure 1. Studied samples: (a) CFRP orthotropic [0], and quasi isotropic [(45/0),], plates; (b) surface of sample; (c) cross section - optical microscope 10x.

The mechanism of changing elastic and viscoelastic properties (storage modulus $E'$), behavior at temperature modification in time (loss modulus $E''$, loss factor $\tan\delta$, glass transition) were analyzed. Monitoring the activation energy for glass transition can become a technique for detecting materials changes after environmental exposure and ageing [10]. The main directions noted at realization (SF) (figure 1) correspond to X axis - direction 1; Y axis - direction 2 (SF plane X, Y in the plane of the composite), axis Z - direction 3 perpendicular to the plane SF.

Figure 2 presents the principle diagram of a route followed in obtaining the samples, respecting the technical-economic stage of the obtaining process.

Figure 2. Principle diagram.

The mechanical properties of the CFRP depend on the nature of the material components, the fiber volume fraction, the matrix, the sequence and orientation of the sheets, etc. The matrix and the fibers fixed together at the traction work as a single material, transferring the loading between them. The mechanical properties also depend on the volume constraints. The rule of mixture [11] states that the elastic modulus of a composite depends by the volumic content of fibers $V_f$. The fiber volume fraction was determined as recommended by the Standard Test Method for Ignition Loss of Cured Reinforced Resins ASTM D2584 [12], volume fraction is 0.44±0.03 respective 0.45±0.03. The thickness of the composite is around 1.91mm and volume ratio 0.45. Diameter filaments of fibers are 6.8μm, number of filaments in yarn 3K. The plain-woven fabric consists of two sets of interlaced yarns where the lengthwise is “warp” and the crosswise “weft”. The characterization of the material from elastic properties for tension and compression tests was realized in [10].
2.2. Experimental set-up

Poisson coefficients \( v_{12} \) and \( v_{21} \) were obtained by measuring phase velocity of waves using ultrasound method using Lamb wave, \( A_0 \) mode, using sensors with contact Hertzian described in [13]. In figure 3 is presented principle scheme of the measurement equipment with Hertzian contact sensors system.

![Figure 3. Generation of Lamb waves: (a) scheme; (b) detail Hertzian contact.](image)

The samples were tested at 100Hz frequency, the amplitude of force being 6N. The sensor was in contact with composite material using conical buffer rods made from 7075-T6 aluminium magnesium alloy and the point curvature radius of the pick is 3mm. The transducers were connected to Pulser Receiver 5073PR, the waveform and the propagations times being controlled by digital oscilloscope Wave Runner 64Xi. On relatively small distances between two buffer rod can be visible both \( A_0 \) (antisymmetric) and \( S_0 \) (symmetric) modes. \( S_0 \) mode is dispersive, practically total attenuated, and the \( A_0 \) mode is propagated.

For pressed on an elastic plate with the force \( F \), the radius of the Hertzian contact, \( a \), is given by

\[
a = F^{1/3} (DR)^{1/3}
\]

where \( D = 3 \left( \frac{1 - v_1^2}{E_1} + \frac{1 - v_2^2}{E_2} \right) \), \( v_1 \) and \( E_1 \) represent the Poisson coefficient and the elastic modulus of buffer rod and \( v_2 \) and \( E_2 \) represent the Poisson coefficient and the elastic modulus of the plate.

The group velocity of Lamb waves in \( A_0 \) mode is

\[
C_{g} = \left( \frac{D}{\rho h} \right)^{1/4} \frac{\omega}{\sqrt{\gamma}}
\]

where \( D \) is flexural rigidity of the plate; \( h \) plate thickness, \( \rho \) density; \( \omega \) angular frequency of the wave. The US propagation velocities were determined according to [14].

The elastic properties of composites were determined as following: storage modulus \( E' \), loss modulus \( E'' \), loss factor tan\( \delta \) has been determined by Dynamic Mechanical Analyser - DMA 242C-Netzch. DMA was set-up to measure the real component of the elasticity modulus \( E' \), the imaginary components \( E'' \) and \( \tan \delta = \frac{E'}{E} \).

The EM evaluation methods can be also utilized for evaluation of CFRP. The carbon fibres have an average conductivity and are embedded into an electrical insulating matrix. The sensor creates inside of plates an EM field that propagates with wavelength

\[
\lambda = \frac{2\pi}{\omega \mu_0 \sigma_i}
\]
where $\mu_0 = 4\pi \times 10^{-7}$ H/m is the permeability and $\sigma_t = 10^2$ S/m is the transverse conductivity of composite. For a lift-off 20μm, EM sensor with metamaterial lens connected to a Network/Spectrum/Impedance Analyzer type 4395A Agilent USA Analyzer and displacement system XY [15] a raster scanning with established steps was obtained.

Voids inside of resin or delamination of fibres can be change the structure of composite that leads to accompanied by important modification of transversal conductivity [16]. The entire composite can be modelled as a succession of anisotropic electric conductive plans, in thickness, having the direction of anisotropy axis according to the composite layout. Present of discontinuities are realised using electromagnetic sensor with metamaterial lens of electromagnetic (EM) sensors [15,17]. Information provided by EM sensor can be presented in amplitude and phase.

3. Experimental results; discussions

The most widely used method for determining the degree of porosity is that of ultrasound, with a mostly linear correlation between attenuation with US and porosity [18]. The quantification of porosity level in composite materials is strongly dependent on the segmentation method and parameter used [19]. The porosity level in CFRP composite materials is essential, being in close correlation with mechanical properties such as shear resistance. A level of about 3-5%, or below, for materials used in structural application might be acceptable [20].

For the detection and characterization of porous areas, Phasor XS equipment coupled with phased array with 32 sensors with a pitch of 0.5mm, central frequency 5MHz was used [21]. In order to determine the porous areas, the transducer was placed on a delay line of the same material as the transducers wedge. The results obtained in the testing of the studied thin composites are shown in figure 4.

![Figure 4. The B scan image of a region of composite with porosity: (a) sample [0]ₘ; (b) for sample [(45/0)₂]ₘ.](image)

Lamb waves are dispersive waves, the wave number respectively the phase velocity and the group changes depending on the frequency. For Lamb waves, the propagation speed, the voltages and the intensity of the waves change and depending on the thickness of the material, obtaining different types of waves for each mode. When the thickness of the materials is small, compared to the wavelength then only the zero wave modes, respectively $S_0$ (symmetric), $A_0$ (anti-symmetric modes) appear. For longer distances (about 29mm), the $S_0$ mode is more dispersive, the $A_0$ mode being practically attenuated being the propagated one. The average propagation velocity of ultrasound through the material are $C_l = 2754 \pm 20$ m/s and $C_t = 1945 \pm 20$ m/s for sample [0]ₘ and $C_l = 2840 \pm 20$ m/s and $C_t = 1970 \pm 20$ m/s for sample [(45/0)₂]ₘ.
The values represent the average of 50 determinations at different points of the samples, the dispersion being calculated with the standard method.

Figure 5 shows the group velocity distribution for mode $A_0$ in propagation directions measured from $10^\circ$ to $10^\circ$ for a circle. It can be observed that the samples having quasi-isotropic behavior have an approximately constant angular velocity distribution around 2800 m/s.

![Figure 5. Angular distribution of propagation speed.](image.png)

Thus, for the direction $0^\circ$, the value of $E_1$ determined by the calculation of the propagation speed of the Lamb wave is of 23.4 GPa, near value determined by static and dynamic test, figure 6.

CFRP with two type of stacking sequence realised was investigated using DMA. The parameters obtained from the tests are ideal for vitrification information, referred to $T_g$ (glass transition), resulting from the cross-linking reaction. A multi-frequency measurement regime was used to obtain the apparent activation energy for the glass transition process. The analysis was performed to provide a better understanding of the consequences of an interrupted process of hardening in the autoclave. DMA shows the temperature range and the state in which a curing process of the composite that has been interrupted can continue. Storage modulus $E'$, is correlative with stored energy. For plates having stacking sequence [(45/0)$_2$] it has a value between 21 and 22 GPa. The value of $E'$ slowly decreases until $90^\circ$ when a transition temperature $T_g$ is reached to the vitreous state around 94°C. This sudden drop after reaching $T_g$ determines the maximum normal operating temperature of the material, during which the steps are still reversible. The thermal destruction of the resin begins when the temperature exceeds the maximum temperature that defines the high elastic area (180°C).

![Figure 6. DMA measurement.](image.png)
The loss modulus $E''$ represents the dissipated energy in the form of heat in dynamic mechanical analysis experiments. The DMA analysis shows that the glass transition is reversible for these samples, up to the temperature at which the maximum reaches 118°C.

For eNDE method characterization of composites structure, EM sensor with metamaterial lens was used. Results are presented in figure 7, and it can show that the advances of eddy current combined with evanescent waves detection improved image of structure of composite.

![Figure 7. The answer of the EM sensor at the scanned structure composite.](image)

4. Conclusions
Using an ultrasound and electromagnetic method of nondestructive evaluation combined with DMA is possible to characterize the composite material for evaluation performance of this. That permits to detect possible voids, nonalignment of carbon fibers, lack of resin and possible delamination. The detection of a defect is limited by thickness, and indeed in this study that appear necessity to used complementary study for improved quality of material.

5. References

[1] Morphy RD 1999 Behaviour of fibre-reinforced polymer (FRP) stirrups as shear reinforcement for concrete structures Master’s Thesis (Canada: University of Manitoba).
[2] Pearson JD, Zikry MA, Prabhugoud M and Peters K 2007 Global-local assessment of low-velocity impact damage in woven composites Journal of Composite Materials 41(23) 2759-2783
[3] Menendez JM and Guemes JA 1999 Strain measurements inside thick CFRP laminates at the vicinity of bolted joints. InSmart Structures and Materials: Sensory Phenomena and Measurement Instrumentation for Smart Structures and Materials 3670 184-194
[4] Kober J, Prevorovsky Z and Chlada M 2016 In situ calibration of acoustic emission transducers by time reversal method Sensors and Actuators A: Physical 240 50-56.
[5] Savin A, Steigmann R, Bruma A and Šturm R 2015 An electromagnetic sensor with a metamaterial lens for nondestructive evaluation of composite materials Sensors 15(7) 15903-15920.
[6] Savin A, Iftimie N, Steigmann R, Rosu D, Dobrescu GS, Grum J and Barsanescu PD 2018 Effective Methods for Structural Health Monitoring of Critical Zones of Scalable Wind Turbine Blades Strojniški Vestnik/Journal of Mechanical Engineering 64 (11) 680-689
[7] Šturm R, Grimberg R, Savin A and Grum J 2015 Destructive and nondestructive evaluations of the effect of moisture absorption on the mechanical properties of polyester-based composites Composites Part B: Engineering 71 10-16
[8] Yin W, Withers PJ, Sharma U and Peyton AJ 2008 Noncontact characterization of carbon-fiber reinforced plastics using multifrequency eddy current sensors IEEE transactions on instrumentation and measurement 58(3) 738-743.
[9] Totry E, González C and LLorca J 2008 Failure locus of fiber-reinforced composites under transverse compression and out-of-plane shear Composites Science and Technology 68(3-4) 829-839

[10] Bergant Z, Savin A and Grum J 2018 Effects of manufacturing technology on static, multi frequency dynamic mechanical analysis and fracture energy of cross-ply and quasi-isotropic carbon/epoxy laminates Polymers and Polymer Composites 26(5-6) 358-370

[11] Morgan P 2005 Carbon fibers and their composites (Boca Raton - CRC press)

[12] ASTM D2584 2002 Standard test method for ignition loss of cured reinforced resins (West Conshohocken: ASTM International)

[13] Grimberg R, Savin A, Steigmann R, Stanciu MD and Grum J 2010 Determination of Elastic Properties of CFRP Using Lamb Waves Resonant Spectroscopy. In 2nd International Symposium on NDT in Aerospace Hamburg, Germany, 22-24 Nov 2010

[14] Grimberg R, Savin A, Steigmann R, Bruma A and Barsanescu P 2009 Ultrasond and eddy current data fusion for evaluation of carbon-epoxy composite delaminations Insight-NonDestructive Testing and Condition Monitoring 51 25-3

[15] Savin A, Steigmann R, Bruma A and Šturm R 2015 An electromagnetic sensor with a metamaterial lens for nondestructive evaluation of composite materials Sensors 15(7) 15903-20

[16] Grimberg R, Savin A and Rotundu CR 2001 Eddy current microscopy applied to graphite-epoxy composite. Sensors and Actuators A: Physical. 5 91(1-2) 73-75

[17] Savin A, Bruma A, Steigmann R, Iftimie N and Faktorova D 2015 Enhancement of spatial resolution using a metamaterial sensor in nondestructive evaluation Applied Sciences 5(4) 1412-1430

[18] Krautkrämer J and Krautkrämer H 1990 In Ultrasonic Testing of Materials (Berlin: Springer) pp 528-550

[19] Kastner J, Plank B, Salabeger D and Sekelja J 2010 Defect and porosity determination of fibre reinforced polymers by X-ray computed tomography In 2nd International Symposium on NDT in Aerospace (Hamburg, Germany)

[20] Koumoulos EP, Trompeta AF, Santos RM, Martins M, Santos CM, Iglesias V, Böhm R, Gong G, Chiminelli A, Verpoest I and Kiekens P 2019 Research and Development in Carbon Fibers and Advanced High-Performance Composites Supply Chain in Europe: A Roadmap for Challenges and the Industrial Uptake Journal of Composites Science 3(3) 86

[21] Savin A, Barsanescu PD, Vizureanu P, Stanciu MD, Curtu I, Iftimie N, Steigmann R 2016 Damage detection of carbon reinforced composites using nondestructive evaluation with ultrasond and electromagnetic methods. In IOP Conference Series: Materials Science and Engineering 133(1) p. 012013

Acknowledgments
This work was partially supported by Romanian Ministry of Education and Research, under grant UEFISCDI PN-III-P1-1.2-PCCDI-2017-0239/60PCCDI 2018 and Nucleus PN 19 28 01 02.