Structural Characterization and Mechanical Behaviour of Carbon Fiber/epoxy Composite for Aeronautical Field

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In the present study EP142-C510-50 woven carbon fiber composite was investigated from structural/ mechanical point of view aiming manufacturing parts for aeronautical industry. The composites were obtained by hand lay-up process. Prior to mechanical tests, the cure reaction results of composite resins specimens were analyzed by using DSC technique. The EP142 epoxy matrix was cured/polymerized at 160°C. After molding, the laminates were cut into specimens for 3 points flexure, tensile and fatigue tests. The mechanical tested samples observed by scanning electron microscopy revealed their fractured and microstructure aspects. A nondestructive US method allowing elastic constant determination on a composite plate was proposed during this study.

Keywords: carbon/epoxy composite material, mechanical properties, structural characterization, aeronautical industry

Composites are materials widely used lately in the aeronautical industry to manufacture several parts as flaps, aileron, landing-gear doors and others [2]. According to the literature, polymer matrix composite materials constituted by continuous carbon fibers embedded in a thermosetting epoxy resin were first developed to satisfy high standards required in aircraft design [3]. Compared to metals the epoxy/carbon composites offer similar or better mechanical properties by mixing in these two distinct phases, fibers and resin, and adopting different fiber configurations. In the fabrication of aeronautical components and structures, the specific mechanical performance of the employed material, i.e., property/ density ratio, is a major requisite since fuel saving and payload improvements constitute fundamental criteria for the operational optimization of the fleet. Despite the several advantages of the polymeric composites over the metallic materials like high strength-to-weight, stiffness-to-weight ratios, high fatigue strength and high corrosion resistance [1,3,4], the former is more susceptible to mechanical damages when they are subject to great efforts of tension, compression, flexure and impact, which can lead to interlayer delamination [5]. With further application of external load, the delamination propagates through the interlayers leading to catastrophic failure of the composite structure. The present work takes also in account the major disadvantage of the cost related to the processing/ manufacturing of composite materials. We propose a much cheaper technology by use of an oven, of performing the samples from the plates or by molding, for the testing campaign. Next research results will contain a detailed structural and mechanical characterization of this kind of composite materials, obtained by using autoclave technology, available at INCDT COMOTI. In this study the tensile and 3 points flexure tests are used to determine both strengths and modulus values, the main purposes being quality control, data evaluation and comparative testing. The fracture surfaces analysis, resultant of the tests, are used to investigate the failure mode analysis. In this work, there were used rectangular specimens of polymeric composites without end-tabs in accordance to SR EN ISO527-4:2000. Generally, rectangular specimens are required for the composite material characterization, because the "dog-bone" type tends to split in the region where the width changes, still we have to take in account that the grips of tension test frame introduce stress concentration in the specimen. In the 0°/ 90° prepreg used in this study, tensile strength is a mix of strength of the reinforcement oriented at 0° governed by the tensile strength of the fibers, and the 90° ones, when the specimens fail by crack propagation through the matrix and/or the fiber/matrix interface. US method was investigated and two elastic constants were determined by calculations, using Snell-Descartes law along with relations that correlate the scroll time through the plate, propagation rate of longitudinal waves in the composite plate on direction chosen, and taking in account the composite material density.

Experimental part

Composites molding/manufacturing

EP142-C510-50 prepreg from Gurit Co (Swiss was used for the composite preparation. EP142-C510-50 is a woven carbon fiber fabric crowfoot 1/3 pre-impregnated with 50% modified epoxy/cyanate ester blend EP142. The molding process for the composite samples preparation used a Binder oven device, the pressure for the composite system being provided by the moment used for the (aluminum or steel) moulds closure. For the EP142-C510-50 prepreg (introduced in the oven at 60°C) it was used a curing cycle with a heating rate of 3 ± 0.2 °C.min^{-1} up to 160°C, holding at this temperature 75 min. (mono-nest mould) or 150 min (plate mould). The cooling rate used was 4 ± 0.2°C.min^{-1}. The thickness of the cured laminate ply was 0.26 mm.

Structure evaluation

The carbon fiber/epoxy composites specimens were investigated post process. DSC analyses were performed on a Netzsch DSC 204 F1 Phoenix device, in order to study the polymerization degrees of the composites. Microstructure observations were performed by FE SEM (Field Emission Scanning Electron Microscope) technique by using a Zeiss equipment, ΣGMA model. Micrographs of the surface and cross section of the studied composites

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were also observed by optical microscopy (OM) to evaluate how homogeneous was the lamination and to observe the specimen in detail after the mechanical tests. The morphological evaluation was performed in an Olympus BH equipment.

The fiber and matrix contents were determined by acid digestion of the polymer matrix, according to the ASTM D 3171 normative [ref]. The composite specimen was weighed and then immersed in a hot sulfuric acid solution to remove the composite epoxy matrix. Afterward, the residue (carbon reinforcement) of the composite specimen was weighed and expressed in volume fraction, according to (1):

$$\frac{m_m}{m_f} = \frac{\rho_m}{\rho_f} x(\frac{1-f}{f}) \quad (1)$$

where:

m_f and m_m are the carbon fiber and matrix weights (g);
ρ_f and ρ_m - the carbon fiber and matrix densities (g/cm^3),
f - the fiber volumetric fraction (%).

Mechanical characterization

Tensile test

Measurements of tensile properties of carbon fiber fabric composites were performed under SR EN ISO 527-4:2000 standard [6] using a minimum of five specimen/test condition (dimensions: 250 mm of length x 25 mm of width x 2 mm of thickness). The specimens were prepared without bonding end-tabs to the specimens no gripping problems being detecting during preliminary testing. The tensile tests were done in an universal testing machine Instron 8802 with hydraulic grip, a 250kN cellule force, the extensometer device was attached on the specimen to measure displacements in longitudinal direction. A constant cross-speed of 2 $mm.min^{-1}$, at room temperature was used, humidity RT 45% (4h conditioning), 5 samples/test condition. The geometry of the tensile tests is given below in the figure 1.

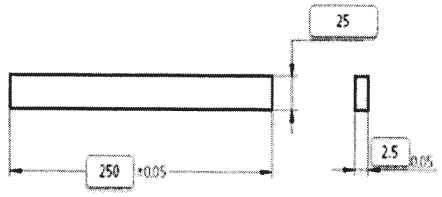

Fig. 1. Geometry and dimensions of the specimens tested in the longitudinal tensile tests

The 3 point flexural tests were carried out in accordance to standards (3-point loading), using a minimum of five specimens /test condition dimensions 80mm x10mm x4mm, according to ASTM D790 [7] and 100mm x15mm x2mm, according to SR EN ISO 14125:2000) [7, 8]. These tests were performed in an universal testing machine (Instron8802) at constant cross-speed of 2mm/min and respectively 5mm/min., at room temperature, using an appropriate device for flexural test.To calculate the flexural strength it was used in (2):

$$FS = \frac{3PL}{2be^2} \quad (2)$$

where:

F S - flexural strength [MPa];
P - rupture load [N] ;
L - support span [m];
b - width of specimen [m] ;
e - thickness of specimen [m].

Fatigue tests

Fatigue tests were performed on a hydraulic fatigue machine at constant load amplitude. Fatigue tests were carried out according to ASTM D3479 [9] at different maximum stresses, the ratio $S_{max}=\sigma_{min}/\sigma_{max}$ was 0.1, where σ_{max} and σ_{min} are the maximum and the minimum applied stresses, respectively. The fatigue frequency was 8 Hz.

US technique

Ultrasound technique was used as an original way to determine elastic constants in composite materials plates. The method of determining the elastic constants of thin composite plates is based on the determination of the scroll time for the ultrasonic pulse to pass through the plate at various angles of incidence. The composite plate is caught between two ultrasonic transducers with variable angle. The longitudinal waves are sent / received at adjustable angles. A transducer is transmitter and the second placed in mirror position is the receiver, the signal received being amplified and displayed on a digital oscilloscope. The transmitter is excited by a transducer transmitter-receiver (Pulser-receiver) and works at a frequency of 2 MHz. All these details are given below in the figure 2.

Refraction plane waves Snell-Descartes law and a link relation with the scroll time for the ultrasonic pulse to pass through the plate are used to determine the elastic constant of the composite. Longitudinal wave propagation velocity is expressed in terms of density ρ of the composite material and the spring elastic constant on the direction of propagation Cθ (3).

$$c_{L1} = \sqrt{\frac{C_{1\theta}}{\rho}} \quad (3)$$

The main directions for measurements are indicated in the figure 3. Direction 1 corresponds to the dominant orientation of the fibers (if that exists). In a contrary case, it is chosen and marked the reference direction 1. The second direction is in the plane of the plate, perpendicular on the first direction, and the third one is perpendicular to the plate.

Therefore if the incident wave is perpendicular on the composite plate, the elastic constant C33 can be determined. For other angles, the constants are given

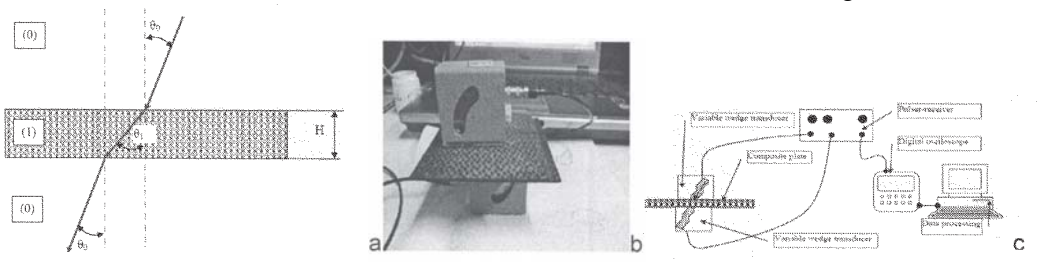

Fig. 2. a) Ultrasonic waves propagation scheme through composite plate, b) US measuring device, gripping mode of the traducers, mounting plate composite between angular transducers and c) principle sketch

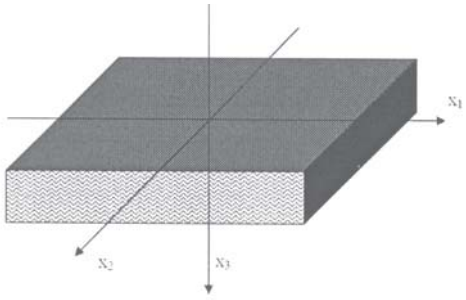

Fig. 3.The main directions in the description of a composite plate

through relations between the constants on main directions [9]. The composite plates are build up from 7 prepreg layers orientated in the same direction. The carbon fibers orientation seems to be 5° lagged from the plate border. The surface is mirror state.

Results and discussions

Structural characterization

Results regarding the volume content of carbon fiber and epoxy for the studied laminate showed a approximately 50 % in volume of each (fiber and resin).

DSC

Sample was heated with a rate of 10 ° C / min, in the temperature range 20-400° C, using constant flow 40mL/ min nitrogen. Figure 4 below shows the DSC curves obtained for the E142-epoxy system. The area above the peak at the exothermic region was used to determine the fractional conversion of the epoxy resin by assuming that the heat evolved during cure is directly related to the disappearance of the epoxy groups during the reaction.

Curing conversion processes using DSC analysis was estimated using the following (4):

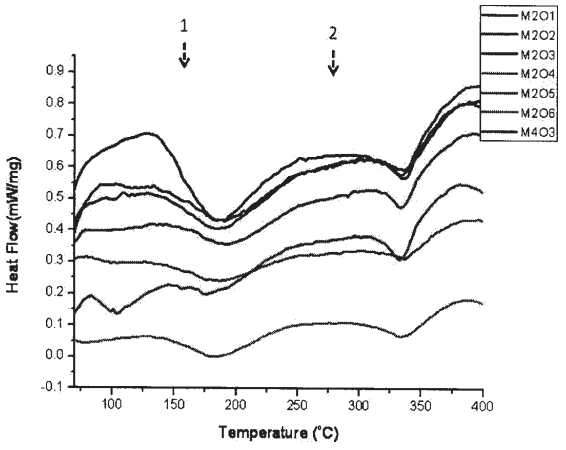

Fig. 4. DSC curves of the E142-C510-50 prepreg at the heating rate of 10°C.min^{-1}; region1-curing process, region2-degradation process

Table 1

DSC RESULTS OBTAINED ON E142-C510-50 PREPREG SPECIMENS AT THE HEATING RATE Of 10°C.min^{-1}

Specimen	Enthalpy (J/g)	Tmax (°C)
M_2O_1	46.3	190
M_2O_2	32.9	192
M_2O_3	53.3	186
M_2O_4	24.5	185
M_2O_6	27.3	182
M_4O_1	*10*	*198*
M_4O_2	*23*	*200*
M_4O_3	86,5	184

$$\alpha = \frac{\Delta H_T - \Delta H_t}{\Delta H_T} x100 \quad (4)$$

where:

α – is the degree of chemical conversion, %

ΔH_T - total enthalpy of reaction [J/g]

ΔH_Tt- enthalpy of reaction at t moment [J/g]

One can see that at first view the exothermic peak disappear almost completely in some cases (on some specimen) but on others (M2O1, M2O3, M4O3) we can still observe residual exothermic peaks. Curves contain both the "cross-linking process" corresponding to the resin cure (1), but also the deterioration of the composite (2). DSC results obtained on E142-C510-50 prepreg specimens using the heating rate of 10°C.min^{-1} are given in the table1.

Mechanical characterization

Tensile test

The static tensile properties of the carbon/epoxy laminates 50±2 % were considered satisfying for aeronautic applications, also compared with [10,11, 13] literature results. Figure 5 below, presents a typical tensile curve obtained on 250x25x2 mm plate specimens, at room temperature, with a constant speed /displacement of 2 mm/min..

3 points flexural tests

The figure 6 shows typical, conventional photograph (a) and and SEM images (b, c) of transversal section (fractured regions) of the 3 points flexure tested specimens. The sample was classified as fractured when the first layer failed under the applied stress, and post flexure test the sample was fractured by hand for fracture surface SEM analysis. One can observe that failure occurred by delamination (in all of the composite layers). The figure 6-c, presents a zoom of the fracture (fig.5-b) region, showing the voids which appears at the matrix/fiber interface, inducing its weakness in terms of mechanical resistance.

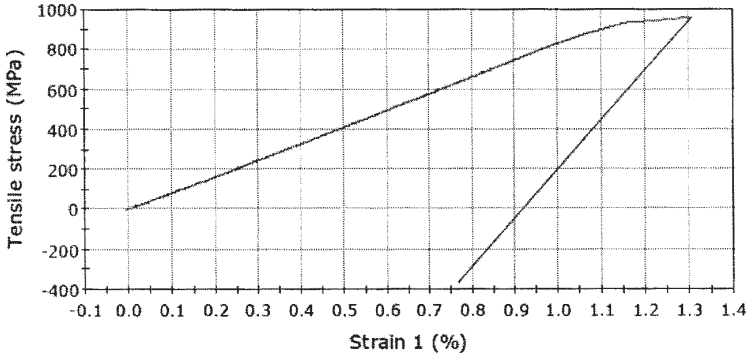

Fig. 5. Typical tensile curve obtaind on the E142-C510-50 prepreg

Table 2
TENSILE PROPERTIES FOR SPECIMENS (E142-C510-50 LAMINATE) STUDIED (2MM/MIN, ROOM TEMPERATURE TENSILE TEST, HUMIDITY RT 45%, 4HOURS CONDITIONING AND 5 SAMPLES/TEST CONDITION); UTS-ULTIMATE TENSILE STRESS,εr (STRAIN AT FAILURE), E (YOUNG MODULUS) AND tr (TIME TO RUPTURE)

Sample	UTS (MPa)	εr (%)	E (GPa)	tr (s)
E142-C510-50-PT01	968.92	1.28	84. 81	94.1
E142-C510-50-PT02	954.62	1.31	74.70	93.6
E142-C510-50-PT03	803.38	1.09	77.70	81.6
E142-C510-50-Data sheet	760	-	62	-

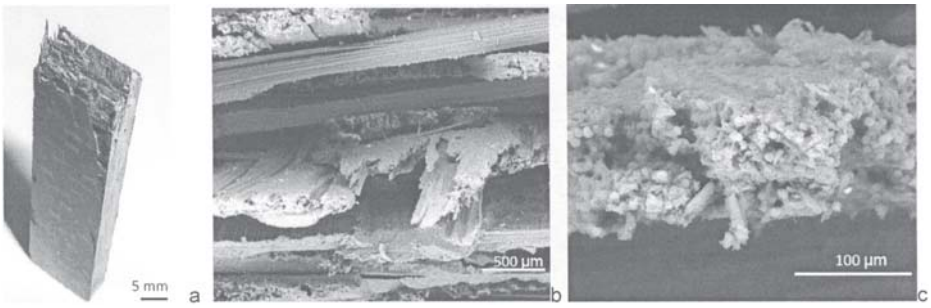

Fig. 6. Photographs (a) and SEM images (b and c) of 3 points flexure failed specimen

Also it is observed that the laminates presented total failure in spite of the different arrangements of the warp and fill of the carbon fabrics.

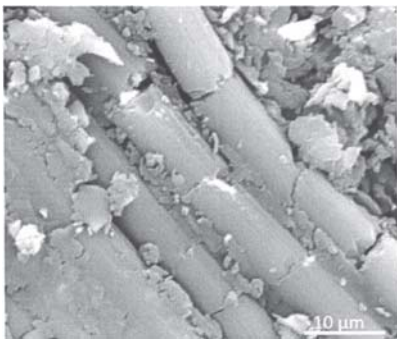

Fig. 7. Step breakage of 90° orientated filaments with respect to the nominal applied stress direction (considered as 0°)

Figure 7 above shows a fractured carbon filament in 90° direction with respect to the applied stress direction. The breakage of filaments caused some cracks in the matrix producing localized debonding. If these cracks had continued along the filament they would have resulted in an increased debonding effect at fiber/matrix interface, eliminating the capacity of these filaments to withstand the loading. In these regions, eventually due to the increased loading, a stress concentration could occur, causing the failure of filaments that are in the nearest neighborhood, which eventually would result in failure of the layer. This fracture mode proposal only for 90° orientated fibers (with respect to the applied stress direction) is in accord with the Bader model [13] which explains failure behaviour of uniaxial fibers composites.

All specimens evaluated, the ultimate rupture occurs within the gage length, between the ends of the specimen. The fracture region shows that fibers has been subject to flexure, on the Figure 8b capture, one can notice that 0° fibers were fractured under the perpendicular applied force.

Table 3, presents the flexure properties of the tested laminate. Considering the standard deviation, it can be realized that the flexural strength values of the E142-C510-50 laminate are close to one another. However, it can be observed a slight increase of the values for some samples. This behavior is attributed to the process structural compactness.

Fatigue tests

The fatigue performances of materials are analyzed by investigating the relationship between the fatigue load (either stress or applied strain) and the fatigue life (or number of cycles to failure). This typical S-N curves are presented in figure 9. Fatigue results obtained during this study were plotted together with carbon fiber fabric/epoxy F155 Hexcel material fatigue results [10-12] on the figure 9, as a curve of maximum fatigue stress plotted against the fatigue cycles.

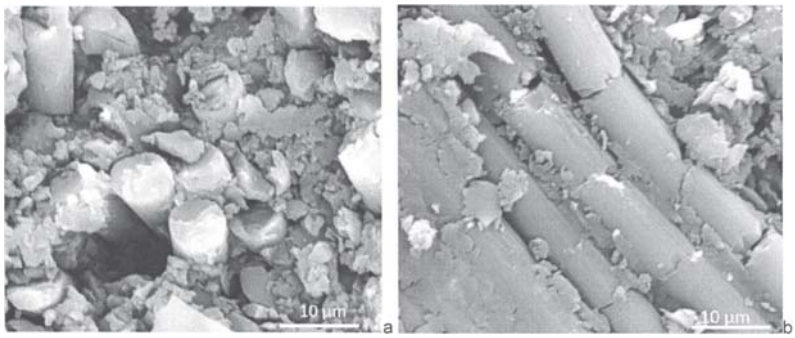

Fig. 8. SEM images of carbon fiber/epoxy laminate after 3point flexure test a) parallel, b) perpendicular on the direction of the applied force

Flexure Specimen	Strain at break (mm/mm)	Flexure Strength (MPa)	Flexure modulus (GPa)
E142-C510-50- M2 01	0.01670	546.99042	35.75
E142-C510-50- M2 02	0.01677	450.27780	26.91
E142-C510-50- M2 03	0.01848	544.11005	30.28
E142-C510-50- M2 04	0.01947	647.00537	37.04
E142-C510-50- M2 05	0.01742	644.05225	40.82
E142-C510-50- M2 06	0.01976	673.37756	39.42
E142-C510-50- M4 01	0,01654	608.64	38.03
E142-C510-50- M4 02	0.01738	586.44934	36.55
E142-C510-50- M4 03	0.01539	451.88214	30.04

Table 3
3 POINT FLEXURE PROPERTIES OF THE E142-C510-50 LAMINATE

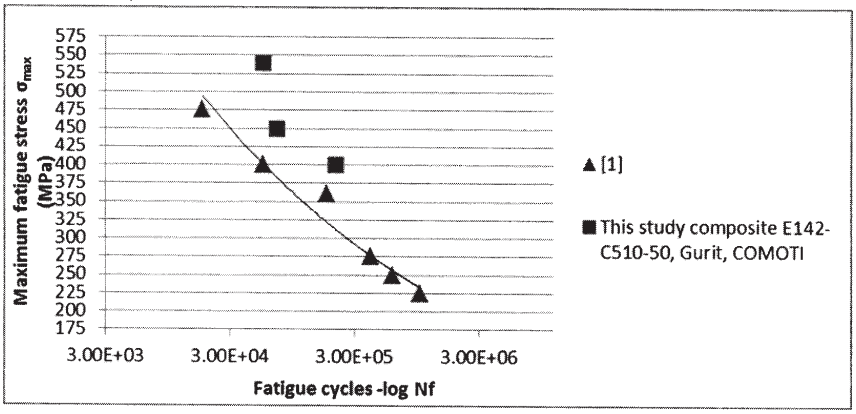

Fig. 9. Fatigue performance of carbon fiber fabric/epoxy F155 Hexcel and E142-C510-50 laminates

Below is presented a summary of fatigue results:

Sample E142-C510-50Pob-02:
$S_{max} = \sigma_{min}/\sigma_{max}=0.1$
$\sigma_{max}=50\%R_m=450MPa=18000N$
$\sigma_{min}=10\%\ \sigma_{max}=45MPa=1800N$
Amplitude: 8100N [(18000N-1800N)/2]

For this specimen the fatigue life translated by the number of cycles during wich the specimen resists to failure was 71 704 ($7,1x10^4$).

Sample E142-C510-50Pob-03:
$S_{max} = \sigma_{min}/\sigma_{max}=0.1$
$\sigma_{max}=45\%R_m=400MPa=16000N$
$\sigma_{min}=10\%\ \sigma_{max}=40MPa=1600N$
Amplitude: 7200N [(16000N-1600N)/2]

For this specimen the fatigue life translated by the number of cycles during which the specimen resists to failure was 210 000 ($2,1x10^5$).

Sample E142-C510-50Pob-04:
$S_{max} = \sigma_{min}/\sigma_{max}=0.1$
$\sigma_{max}=60\%R_m=540MPa=21600N$
$\sigma_{min}=10\%\ \sigma_{max}=54MPa=2160N$
Amplitude: 9720N [(21600N-2160N)/2]

For this specimen the fatigue life translated by the number of cycles during which the specimen resists to failure was 54 441 ($5,4x10^4$).

In the figure 9, one can notice that in the case of carbon fiber fabric/epoxy F155 Hexcel composite [1] for a lower value of the applied stress σ_{max} 475 MPa, compared to the applied fatigue stress to this study composite E142-C510-50 composite (of 540MPa, E142-C510-50Pob-04) the number of cycles is inferior ($1,8x10^4$) to the one obtained during this study ($5,4x10^4$), for a higher applied load. Likewise if we compare, the E 142-C510-02-50Pob-02 where the maximum stress applied σ_{max} was of 450MPa or E142-C510-50Pob-03 where the maximum stress applied σ_{max} was of 400MPa, with the literature results [1] in which the applied stress was 400MPa, the numer of cycles obtained is comparable $7,17x10^4$, respectiv $2,1x10^5$ (this study) versus $5,5x10^4$ [1].

The processing technologies in the two studies (COMOTI versus [1]) are different. Hoever, we can observe that this study mechanical results on composites are comparables to the one reported by other authors [1]. We have to mention also that both types of woven are carbon fiber prepregs [1]) the plies were placed successively in the same direction in the stage of composite samples execution. Thus, on both the main direction of the applied load of 0 ° and also on the perpendicular direction 90 °, the load, the load distribution is almost identical.

US analysis

Plates A and B were placed between the transducers, acoustic coupler being provided with coupled water-soluble silicone. Signals through the plates are compared with the direct signals between traducers and therefore results the scroll time for the ultrasonic signal to cross the plate. Time difference between the two signals is determined with an accuracy of 0.02 µs, using a computer program developed at Politehnica University of Bucharest and is based on signals inter correlations. The results of US tests are presented in the figures 10 and respectively 11.

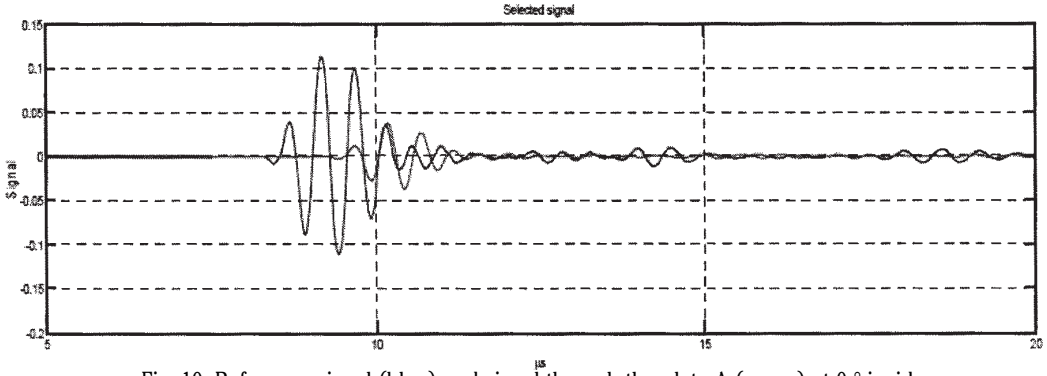

Fig. 10. Reference signal (blue) and signal through the plate A (green) at 0 ° incidence

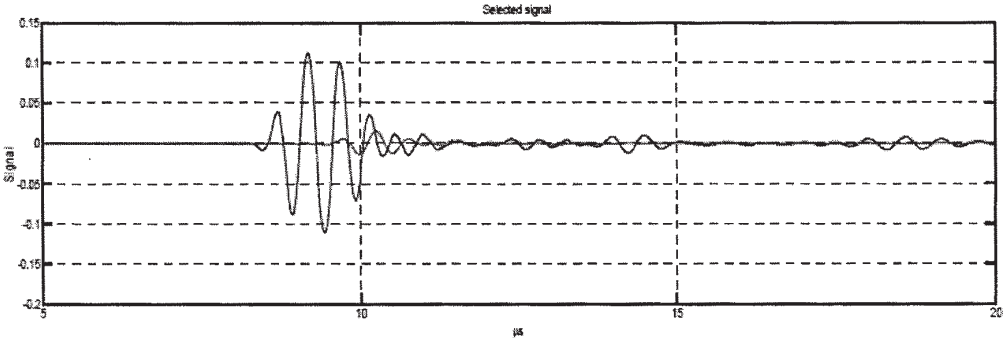

Fig. 11. Reference signal (blue) and signal through the plate B (green) at 0 ° incidence

The density was determined by geometric method: volume calculation from the plate measured dimensions with a precision of 0.01 mm and weighed with a precision of 0.01 g. Densities (ρ) calculated for the two plates investigated are:

a) Plate A: ρ_A = 1.38 mg/mm3 (1380 kg/m^3)
b) Plate B: ρ_B = 1.28 mg/mm3 (1280 kg/m^3)

For the signals in the figure 10 it was determined a long scroll time of 1.02 μs. We determined the longitudinal wave speed in the plate A, in the direction 3, c_{10} = 2.255mm / μs. It results the elastic constant following the normal direction on the plate A.

$$C_{33A} = \rho_A \times c_{10}^2 = 1380 \times 2255^2 = 7.02GPa$$

For the signals in the figure 11 it was determined a long scroll time of 0.84 μs. We determined the longitudinal wave speed in the plate B, in the direction 3, c_{10} = 2.666mm / μs. It result the elastic constant following the normal direction on the plate B.

$$C_{33B} = \rho_B \times c_{10}^2 = 1278 \times 2666^2 = 9.09GPa$$

Determination of constants in the other directions is possible by applying previous relations. More precise experimental measurements using fixtures devices are necessary before other results presentation.

Note: The major advantage of using the curing process of the composite materials, an oven along with molding system it is clearly the price. The method used during the present study is given as cheaper solution to obtain composite parts for less strength demanding applications. Next research results will contain a detailed structural and mechanical characterization of composite materials obtained by using autoclave technology, available at INCDT COMOTI. A comparison between the performances of using the two methods for the polymerization process of the composite materials: the autoclave and the oven assisted molding method, is clearly of great interest and will be performed. The estimated results regarding this future study is to give a value (in %) showing the structural and mechanical strength level difference between the composite materials cured with both methods and also to state optimization solutions for the less expensive but also less performat one (the vacuum assisted molding-oven method).

Conclusions

The mechanical property results exhibit by the carbon fabric reinforced epoxy matrix E142-C510-50 laminate were coherent with expected values, showing that they are adequate materials for application in aeronautical field. Rupture is caused in both tensile and fatigue tests due to interfacial damage. All tensile results are in agreement with literature ones [13,14]. Delamination occurred in this tested samples due also to the lower strong effect of the in-autoclave pressure, replaced by the mould closure pressure, affecting the materials compactness. Regarding the US study results, the present study leads to the following conclusions: it is clear that ultrasound-based method allows determining the three main directions elastic constants. The method is nondestructive, samples can therefore be used in other subsequent determinations. Measurement errors increase with ultrasonic beam tilt, if mechanical clamping devices for transducers are not used. Ultrasonic beam angles should be measured to an accuracy of ± 1°. In the future studies, this method will be developed and improved, the composite plate should be tested in water, the precision determinations would increase compared to the case of angular transducers which transmit ultrasonic waves through Plexiglas.

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