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Experimental study of environmentally Friendly composite materials Behavior in aeronautical applications

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ABSTRACT/RESUME

Abstract: *This study is a part of a work in progress on the mechanical behavior of laminate in various modified epoxy matrices. we begin with a laminate six folds with fiberglass taffeta, and epoxy matrix cross linked by aliphatic Amine called MEDAPOXY STR, this resin is in the form of a kit of two elements ,a monomer and a hardener with the report weight of 0,67%. The treatment of the results shows that the reticulation of monomer by this Amine is incomplete what provokes a plastic domain on the mechanical behavior of the matrix, concerning composites made from these elements, the results show that the elastic domain is dependent on the elasticity of fibers used and not on the matrix. The control by ultrasound can be considered as another way to measure and follow-up of the parameters elasticity of the elaborate laminate.*

I. Introduction

Composite materials have important benefits over traditional materials particularly in aeronautics applications. The advantages include the improvement in life, aging, and ease of shaping that allows obtaining an optimal aerodynamic shape and particularly a minimum weight. These factors are of great importance when it comes to reduce fuel consumption see Figure 1 which is in accordance with the first certification standard for CO_2 emissions from aircraft adopted by ICAO (international civil aviation organization).

Air transportation contribution represents almost 2% of global CO_2 emissions, thus the need to explore possibilities for aviation emission reduction.

Indeed these need justify the use of composites, in particular thermosetting polymer, in the automotive, marine, aeronautics and other industries, knowing that for 40 years the market share in these sectors has increased significantly, this evolution is the origin of several innovations of materials made in laboratories of AIRBUS and BOEING. During the last twenty years, the constitution of the aircraft structures has increased to more than 50% of the total weight of the aircraft structure. Material composite rate in the Airbus A330 and the Boeing B737 represents about 10% of its total structure weight. While on the current aircraft (B787, A350) represents 53% of the weight of the structure reducing fuel consumption by 15%.

Despite all these advantages, we cannot ignore the disadvantages of these materials that can be a fire hazard as well as high.

The performances of composite materials are influenced by the properties of the matrix used; the latter maintains the form desired and the protection of the reinforcements against the external attacks. The epoxy resins present good physicochemical performances [1].

Different studies about the behavior of this type of resin have been realized specially mechanical behavior in static and fatigue by M. M. Shokrieh and all [2], and the behavior of recycled carbon fiber (CF) reinforced epoxy composite has been experimented using the microwave irradiation by Y tominaga and all [3].in the same way, the resin considered to be tested has been reinforced by glass fiber to see its influence in the mechanical composite characteristics.

The reticulation of this matrix is carried out by several famiies of the acids amines, the choice of hardener is carried out according to several parameters bound by the method of working, as well as the field of use, consequently, viscosity, the gel time and the characteristics in a solid state will be changed [4]. In the literature, the characterizations of laminates are based on types and architecture of reinforcements [5], and of the epoxy matrix properties [6, 7], in this context, the creep behavior is strongly influenced by the viscoelastic properties of resin and the characteristics of fibers. In the case of the GFRP the creep limit is 0, 3 F U (F U = tensile strength), in the case of the CFRP and aramid (AFRP) the rupture limit to the creep of 0, 70 F U according to recommendations' of the ACI 440.4R04 [8].

The objective of this work is based on the physic mechanical study of a laminate based on epoxy matrix solidified by aliphatic amine and glass fiber of the type E used in aircraft.

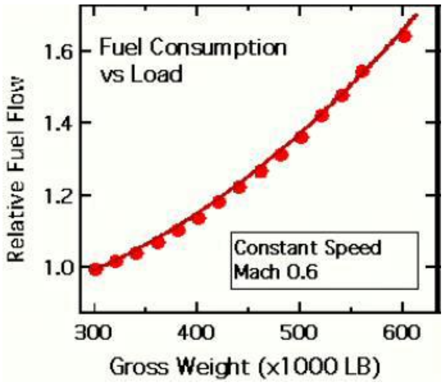

Figure 1.*fuel consumption versus aircraft gross weight*

II. Materials and technics

II.1. Used materials

Epoxy resin is used as the matrix The matrix , the monomer is of the DGEBA type whose the equivalent epoxy weight [9] (The epoxy equivalent is defined as the weight of resin in grams, which contains one gram of the equivalent of epoxide) by chemical proportioning equal 186,38 g/mol, cross-linked by aliphatic amine with a report/ratio weight of 0.67% given by the manufacturer , the set name is MEDAPOXY STR.,The reinforcement used is a class E glass fiber; its weaving is of type taffeta, the characteristics of the wick in warp and weft direction are identical. The weight is of the order of 500 g per square meter.

II.2. Elaboration of material

The specimens of STR matrix molded in machined aluminum mold to give the dumbbell shape in accordance with ISO 527, then, after remolding, the cross-linked test pieces subjected to a thermal treatment of 80 C for 8 hours in order to increase the degree of crosslinking [6].

In most cases curing reaction of the resin with the hardener, is done either by homopolymerization (action of initiators), or by copolymerization (with crosslinking agents of different types) [10].The synthesis of an epoxide polymer is obtained following a crosslinking reaction.

The crosslinking occurs as a result of successive reaction of epoxide and aliphatic amine with basic character and high reactivity ,this isothermal curing reaction is usually complicated and is as a consequence of the interaction of the chemical kinetics of curing with other physical processes, causing significant changes in the macroscopic physical properties of the reactants ,these processes determine different transition phase (gelation, glass transition,) this epoxide has a three-dimensional microscopic structure obtained by chemical reactions between the epoxy prepolymer and a hardeners [11].

The laminated plates are worked out by the infusion method (complete replacement of the void by the resin), the plate remains in depression by the pump until the cross-linked of the matrix; then, it is put into an oven at 80 °C for eight hours. The specimens are cut in rectangular form elaborate plates, in accordance with the standard ASTM D 3039. The samples are provided with aluminum heels;

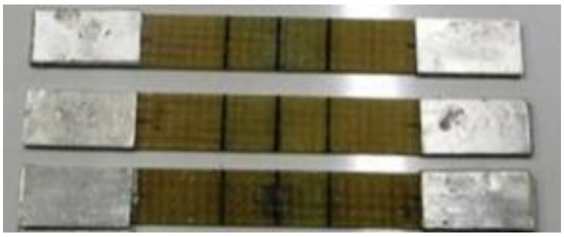

Figure 2. *Prepared tensile specimens*

III. Experimental devices

Thermal analyzes DTA and TGA were performed using the instrument STA NETZSCH (simultaneous thermal analyzer). It measures the thermal effect, and the mass changes under the effect of the temperature variation [12]. This technique is validated under the standard NF-EN 31357-2 (plastic Analyis calorimetric differential). The analysis is carried out on two types of samples. The first type is cross-linked without undergoing heat treatment, while the second type is cross-linked with the preceding of heat treatment. The speed of heating during the tests of the thermal analysis is of 10 °C/min. for these tests, we took as reference an empty capsule.

The tensile tests of STR matrices are carried out on a universal machine of Zwick/Rolle equiped with a sensor of force of 10 KN and with an extensometer. A Computer with a software testXpert version 12.0 controls this machine.

Concerning the laminates, the tensile tests are carried out at ambient temperature using a universal machine of type Zwick/Rolle 250, equipped with a sensor of capacity 250 KN and an extensometer; this machine is controlled by computer using the software TestXpert version9.0.

The mass rate of the reinforcement to be characterized separately is determined by the method of the loss weight with calcination, according to standard NF T 57-571 (applicable to the tablecloths, wire and laminates of glass). The test-tube is of prismatic form; the latter is weighed first once at the ambient temperature (My). Then, put in a furnace at 600°C during 1 hour in order to burn the resin. The reinforcement remaining is then weighed (MF). The mass rate of reinforcement is given by the following formula:

$$T_m = \left(Mf \;/\; Ma\right) \qquad (1)$$

The creep tests were carried out on the universal Machine 250 KN for a load of 50% of maximum stress during 100 hours in mode of relieving and creep what makes it possible to carry out the model semi logarithmic for the prediction.

This model is largely used to describe the tension time relationship FRP under a constant load and proved reliable like an effective means to characterize and to predict the behavior depend on the time of FRP [13, 14], the general form of the model semi logarithmic curve is:

$$e = e_0 + A \; Ln\left(t\right) \qquad (2)$$

Where ε is the total deformation of creep and ε0 is the initial elastic strain, A = coefficient related to the level of the matter and the stress.

e_0 obtained by the constraint and the durable modulus of elasticity, it is important to determine the values of A by the method of the adjusted curve

The cyclic tests are carried with an intensity increase step by step; the first stage starts with 50 MPa, for each cycle increases the force in max of 2 MPa until failure. The method of nondestructive testing of laminated composite plates, by ultrasound wave frequency performed with ultrasound test bench (figure 2) using the acoustic waves in the transverse and longitudinal directions.

The method used is called contact method; the transducers are in direct contact with both sides of the plate to be analyzed. The longitudinal wave is emitted by the emitter transducer (2 MHZ) and picked up by a receiver transducer (4MHZ) on the other side, with the flight time in the sample and knowing the distance between the transducers; the propagation velocity of the wave in our material is calculated adopting formula (3). The mechanical properties are in relation to the density and longitudinal and transverse speed [15].

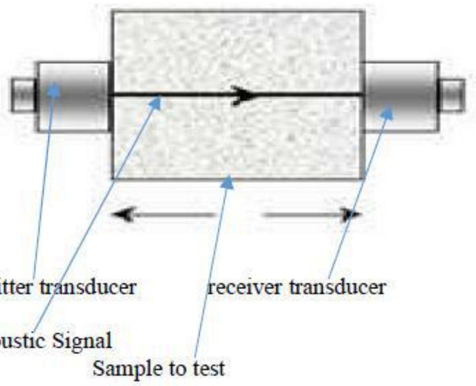

Figure 3.*specimen test by ultrasound*

$$V_L = \sqrt{\frac{E.\left(1-O\right)}{r\left(1+Ö\right)\left(1-2Ö\right)}} \qquad (3)$$

$$V_T = \sqrt{\frac{E}{r\left(1+2Ö\right)}} \qquad (4)$$

With :
- V_L : vitesse de propagation de l'onde longitudinale;
- V_T : la vitesse de propagation de l'onde transversale ;
- ρ : masse volumique du matériau ;
- E : module de Young ;
- √ : coefficient de poisson

From these formulas, one as follows deduces the properties from elasticity:

$$E = r \; \frac{3V_L^2 - 4V_T^2}{V_L^2 - V_T^2} \qquad (5)$$

$$V = \frac{1}{2}\frac{V_L^2 - 2V_T^2}{V_L^2 - V_T^2} \qquad (6)$$

IV. Results and discussion

IV.1. Characterization of the matrix

The results of DTA and the TGA of the samples without and with posterior heating enables us to determine the temperature of vitreous transition Tg and the mass decrease ΔM/M0, the results are summarized in the table below.

Table 1. *Results of thermal analysis*

	Without heat treatment	With heat treatment
t_g	99.6	137.12
ΔM /M0	-1.40%	-1.04%

The thermal analysis results show that the glass transition temperature [16] of the heat-treated matrix (post-curing) is higher than that of the untreated matrix.

With a full $tg_{¥}$ crosslinking temperature of the EDGBA resin between 150°C and 200°C, the rate of the reticulation progress is given by the relation based on the diagram of the temperature, the transition time [17].

$$\Delta\tau = \frac{t - \tau_0}{\tau_0} * 100 \qquad (7)$$

$$\tau = \frac{t_g}{t_{g_{¥}}} * 100 \qquad (8)$$

From the equations (6) and (7), this increase in the crosslinking percentage is calculated as follow

$$\Delta\tau = \frac{\frac{t_g}{t_{g_{¥}}} - \frac{t_{g_0}}{t_{g_{¥}}}}{\frac{t_{g_0}}{t_{g_{¥}}}} * 100 = \frac{t_g - t_{g_0}}{t_{g_0}} * 100 \qquad (9)$$

By applying the preceding formula, we have for this matrix: $DT = 37,67\%$, effectively in the ordinary cases, the tertiary amines are encumbered sterically, and some functional groups remain without crosslinking (inhomogeneity at the microscopic scale), the treatment by temperature makes it possible to facilitate molecular mobility and decreases this steric hindrance [18].

Figure 4 presents behavior in traction of dumbbell test pieces containing the STR matrix, an elastoplastic behavior of the matrix is observed characterized by a Young modulus of 2.53 GPa and a deformation of 3.6%

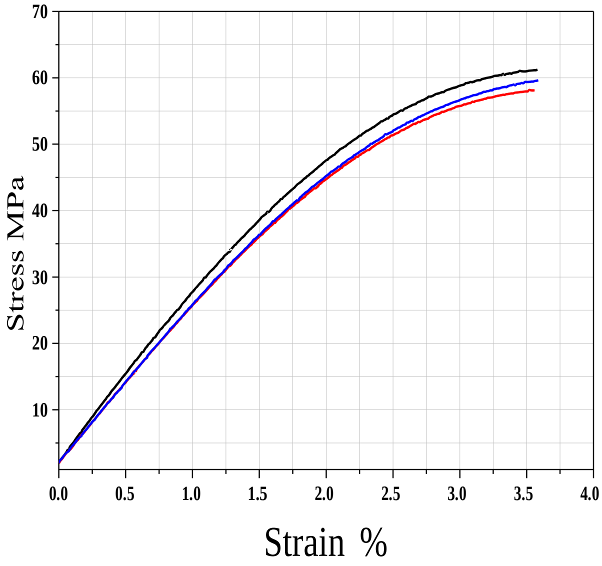

Figure 4. *Tensile Behavior of the matrix STR*

IV.2. Characterization of reinforcement

The reinforcement used is a glass fiber oiled of type E with an armor of the taffeta type.

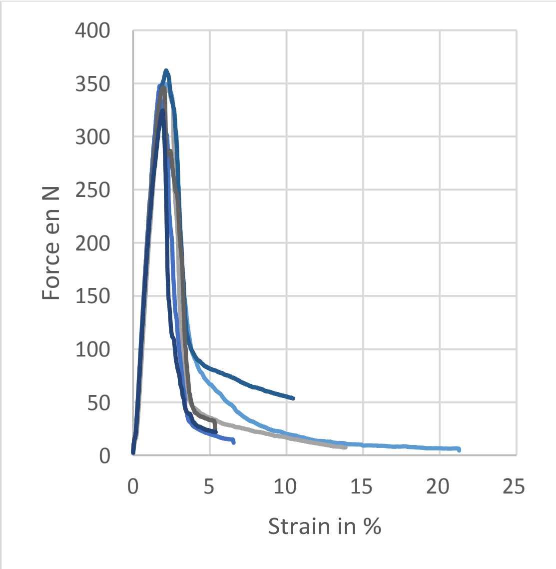

Figure 5. *Tensile Behavior of the wicks in glass fiber*

The calculation of the shrinkage consists in measuring the deformation of the wicks in the two directions, starting from the tensile test; the deformation is presented in the form of shrinkage. Various measurements show that the values of the undulations are equal in the two directions. The average value of the shrinkage is about 2%.

During these tests, the slip of aeronautical fibers in the wick is due to the oiling, which protects them

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from brutal shearing. The majority of fibers break with the maximum loading, except for some fibers that slip between them. What explains the appearance of a residual force until the total rupture that varies from deformation of fibers equal to 5% and a force close to 100 N.
The linear part of the curves in the directions chains and screen is identical. We deduce that the wicks used for the two directions (warp and weft) are identical or present the same mechanical characteristics.
This deduction is confirmed by the comparison between these results and measurements carried out of shrinkage.

IV.3. Characterization of the laminate

The density is determined according to the method of standard NF T 51-561. The results obtained from the measurements on three samples give us the average value of 1.9314 g / cm3. To calculate the mass ratio, the method of the loss on the calcination, according to standard NF T 57-571 "equations (1)" is useful; the values obtained are summarized in the table below

Table 2. *Values of the mass ratio*

Composites containing matrix STR		
Mm	Mf	Rf
7.2147	5.6527	78.35
5.7451	4.5179	78.64
6.3221	4.9413	78.16

the volume fractions are obtained by applying the law of mixtures in composites, knowing the densities of the composite (ρc) and the fiber which is already calculated (ρf = 2,56g/cm3).after calculation, Fv = 59. 13%.
The tensile tests are carried out at ambient temperature on a universal machine of type Zwick 250.
The elasticity of the laminate STR is between 0 and 2%. This value represents the shrinkage of the reinforcement used, the obtained Young's modulus of 17.85 GPa. The plastic range is characterized by two phenomena as shown in Figure 5, namely, delamination and the rupture starting from deformation of 3%. The elastic deformation of the matrix is less than the shrinkage of the fibers used which gives us a value of deformation around 2%, which remains within the range of elasticity of the laminate composite; the matrix is not in its elastic range.
The creep test protocol is to apply 50% of stress of breakage during 100 hours, below figure 7 present the deformation versus time.

With determination coefficient R2 = 0.9488 logarithmic model creep behavior is given by

$$e = 0.0066 ln(x) + 1.125 \qquad (10)$$

Figure 6. *Breaking mode by tensile test of laminate*

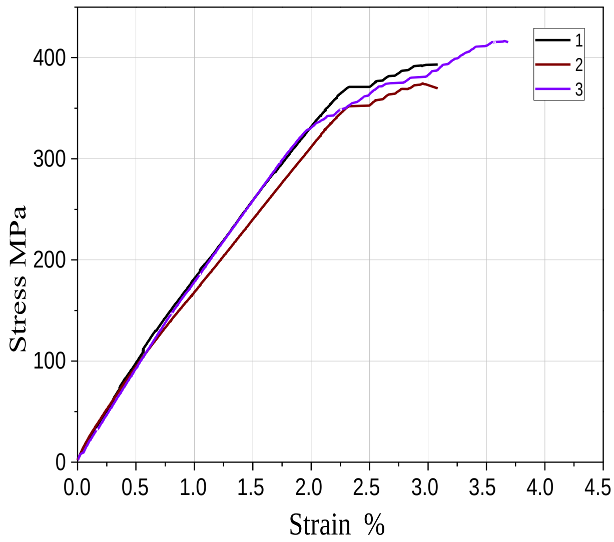

Figure 7. *Tensile Behavior of laminates based STR resin*

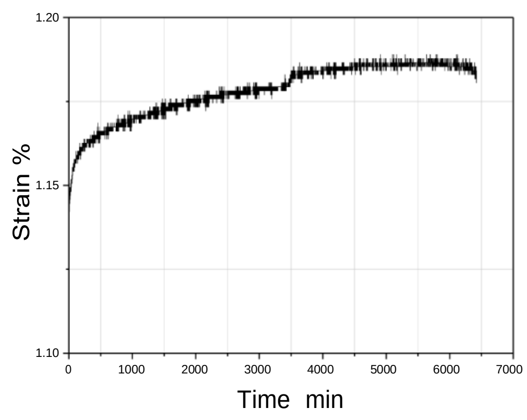

Figure 8. *Creep strain at 50% of ultimate stress*

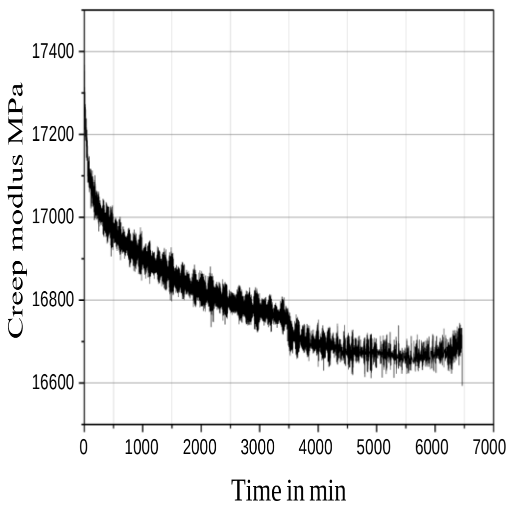

Figure 9. *Creep modulus versus time*

The creep modulus in function of time is shown in Figure 8, the variation of this module is in connection with the evolution of deformation knowing that the constraint is fixed in time, in correlation with the previous formula the evolution of creep modulus in time is deducted by:

$$E = E_0 \ / \ 0.0066 ln \left(t \right) \qquad (11)$$

The results do not show a drastic change in the Young's modulus, but a remarkable decrease of tensile strength, we notice the same deformation than the static test, which shows that the failure mechanism is caused by brutal propagation of micro-cracks or delamination to some level of load.

Four main modes can degrade the state of composites subjected to different stresses in particular load of fatigue [19],cracking of the matrix, detachment of the matrix of fibers, delamination and fracture, some defects accumulate since the beginning of their life whereas fiber fractures limit the life of this composite, resulting in a final failure. It has been observed in our tests that the rigidity of the laminate decreases during the creep and fatigue stresses confirming the conclusion of A Razvan [20].

The test of nondestructive testing of laminated composite plates (see figure 9),is performed with ultrasound test bench using the acoustic waves in the transverse and longitudinal directions, The method used is called contact method, the transducers are in direct contact with both sides of the plate to be analyzed. The longitudinal wave is emitted by the transducer transmitter and picked up by a receiver on the other side. The signal obtained is shown on the following figure 9; the wave speed is directly given by dividing the thickness of the work piece (course path by wave) by the time passed during emission reception.

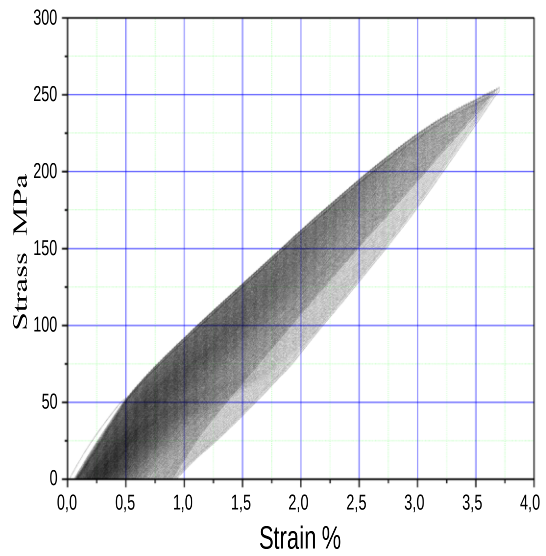

Figure 10. *Cyclic tensile test*

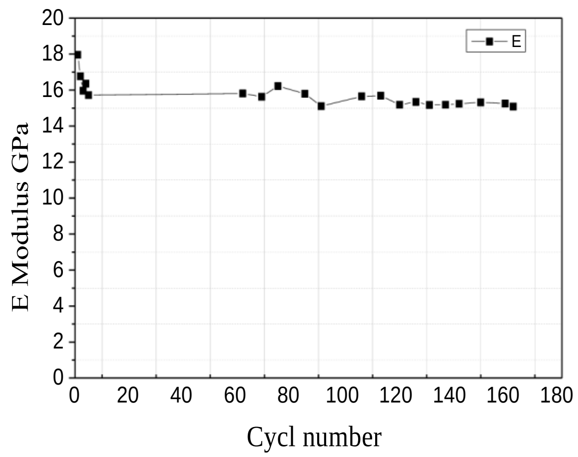

Figure 11. *Young modulus versus time*

From these echoes, the values of transverse speed, longitudinal speed and the Young modulus are calculated by using the equations (3), (4) and (5); the table 3 presents these values.

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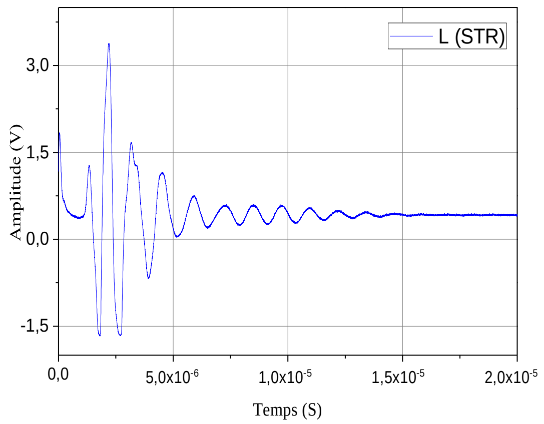

Figure 12. *Propagation of longitudinal wave*

Table 3. *Values of ultrasonic velocity and modulus.*

matrix composites STR		
V_L (m/s)	V_T (m/s)	E (GPa)
1886.55	08.67	17.25

The Young modulus obtained for the laminates based in matrices STR are respectively lower by 0.60 GPa compared to the results of the destructive tests. These differences are due to what follows:
The agent of coupling which can generate attenuations of the ultrasonic waves; the specimen surface quality; the porosity of composite materials.

This measurement technique method is called “method of the building site”.
These variations are lower than 4%, which make the measurements by ultrasound efficient, to determine the mechanical characteristics without destroying the sample to analyze.

V. Conclusions

The aim of this work is articulated around the mechanical behavior of laminate based in epoxy matrices crosslinked by mixture with different hardeners, within this framework, we started with the use of an aliphatic amine characterized by a high reactivity. This mechanism of reticulation allows adopting an elastoplastic behavior to the matrix, the elastic strain is lower than 2%, and this value present the shrinkage glass fibers used in à taffeta form.

The results obtained show the need for heat treatment in the case of a reticulation by aliphatic amines, which are characterized by their high reactivity.

With regard to the mechanical behavior, the elastoplasticity of the epoxy matrix shows that the rate of reticulation is partial, results are in correlation with the type of materials used, effectively, as a thermosetting ,when thermohardening reticulation temperature is reached, its atomic tridimensional form favorites a vitrification.

The Laminates elaborate presents an elastic behavior being able to reach a value of 2%, whereas the matrix in this phase is under plastic deformation, this anomaly is the source of several findings on the creep behavior in the elastic range.

The nondestructive testing of this composite material type based on aeronautical fiberglass and resin epoxy carried out, shows a little variation of rigidity various (about 4%) between classical methods and nondestructive tests, this means can perfectly be used both to detect defects and to characterize the material.

In perspective, we plan an experimental study of this material applied to the repair on the aeronautical laminate composite based in carbon fiber , especially a patch method, and compare the results of its mechanical behavior with Ansys simulation software.

VI. Nomenclature

s - Normal stress (MPa)
e - Normal strain (%)
E - Modulus of elasticity (GPa)
n - Poissons ratio
DT - Température change (°C)
a - Coefficient of thermal expansion (°C)
V_L- propagation velocity of the longitudinal wave
V_T- the propagation velocity of the transverse wave
ρ- Density of material (kg/m^3)

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