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CONTRIBUȚII LA STUDIUL
PROPRIETĂȚILOR MATERIALELOR
COMPOZITE ARMATE CU ȚESĂTURI ȘI A
EFFECTULUI SOLICITĂRILOR CICLICE
ASUPRA ACESTOR PROPRIETĂȚI

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MATERIALELOR COMPOZITE ARMATE CU
ȚESĂTURI ȘI A EFECTULUI SOLICITĂRILOR
CICLICE ASUPRA ACESTOR PROPRIETĂȚI**

**CONTRIBUTIONS REGARDING THE
PROPERTIES OF FABRIC REINFORCED
COMPOSITE MATERIALS AND OF THE EFFECT
OF CYCLING LOADINGS ON THESE PROPERTIES**

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Introduction

Achievements in the field of composite materials gradually increase with the necessity of their being used in various fields, such as: aerospace, automotive, naval industry, sports industry, energetic industry, constructions, medicine. The increased usage of composite materials in the aforementioned areas is owing to their characteristics, superior in most cases to those of the traditional materials, to the reduction of the energy consumption required for their manufacture, to their increased resistance to corrosion, tensile strength, attrition, resistance to high temperatures, low density, controllable thermal conductivity, good deformability, remarkable dimensional stability, low thermal expansion, resistance to impact and also to many other characteristics that, unlike those of traditional materials, are projectable [1].

Unlike traditional materials, composite materials are made up so as to have their properties perfected, their design being acquired based on the characteristics of the components used in the new composite material. Thus, according to the field intended for their use, composite materials must meet certain qualities (properties) required by the intended use.

The most important qualities of traditional materials (metals, ceramics, polymers) can be enhanced by fibre-reinforcement. Composite materials can be reinforced with short, long, continuous fibres and/or fabrics for improving their mechanical properties, as fibres take a significant part of loadings, thus increasing the material or structural strength. The fabric type used in composite materials is of utmost importance, as each fabric presents different properties; e.g. carbon-fibre fabric presents excellent electrical properties and it is used in the aero industry, for both civil and military aircrafts, glass fibre fabric has good anticorrosive properties and high resistance to humidity, being therefore used in the construction of maritime ships, aramid fibre fabric presents anti-shock properties and it is used in military applications. Such fibre-reinforced composite materials have replaced the traditional structural materials such as wood and steel in many applications, especially due to their positive ratio of mechanical strength and density, to their chemical resistance and to a versatile design.

Matrix is the second element of composite materials. Owing to the excellent mechanical, chemical and electrical properties, epoxy systems are the materials most often used in the category of thermoset polymers [2]. The properties of solid polymers – mechanical, electrical, and thermal – rest to a great extent on the hardening agent used, as its type and the volume ratio of resin and hardener mixture affects viscosity at pre-polymer state and, consequently, its malleability at the stage of effectively placing the components into the matrix, in shapes in which composite materials or structures are going to take.

As it is still novel, especially from the perspective of its outstanding development, the field of composite materials is marked by deficiencies of the mathematic patterns associated with the description of these materials. The researcher or engineer specializing in composite materials is confronted with difficulties induced by the basically infinite number of combinations of matrix and reinforcement elements, of the manufacturing networks and techniques, each of these elements having major impact on the end properties of the materials formed.

In this context, the present study has been designed along two main lines – fairly divergent from one another – on the one hand, testing the laminate model for fabric-reinforced composites

with polymer matrix, using lamina properties as data entry, and, on the other hand, dynamic testing of the formed materials in order to identify their behaviour to fatigue. The idea of the study has been largely influenced by the results of previous research carried out at the Research and Development Centre for Thermoset Matrix Composites of “Dunărea de Jos” University of Galati by my colleagues, Dr Vasile Bria, Dr Marina Bunea, Dr Igor Roman and Dr Victor Ungureanu.

The study has been designed so as to ensure repeatability of the results, the main aspects concerning the formation (in identical conditions) of the materials to be tested (be they laminae or laminates). There have been formed (fabric-reinforced) laminae with epoxy matrix for 18 types of fabrics and three different epoxy resins. The laminated materials have been formed by using nine layers of fabric for each one of the three epoxy resins.

The first chapter of the thesis outlines general aspects of composite materials, pertaining to their importance in various applications. With consideration to the importance of this chapter, the current state of research has also been tackled, with regard to theoretical and experimental studies of the mechanical, thermal, and electrical properties of polymeric composites.

The second chapter outlines the main research objectives.

The third chapter reflects the methods of preparing and forming the fabric-reinforced composites (lamina and laminates) with epoxy matrix. It also presents the characteristics of the materials used in the study – fabrics and polymers – for the formation of composites.

The next chapter deals with the analysis of the thermal properties of the formed materials. Emphasis is laid on specific heat and coefficient of linear thermal expansion, as these properties represent, in the case of polymeric matrix composites, a defining criterion for their application.

The fifth chapter consists of aspects related to the mechanical properties of laminae, with an analysis of the influence of the fabric and matrix used. The data presented is the basis for the analysis of the mechanical properties of laminates.

Chapter 6 outlines the results obtained during the static and dynamic mechanical tests on laminates, next to a comparative analysis of the experimental data with data provided by applying the laminate model.

The last chapter presents the general conclusions, alongside with prospective research.

Chapter 1. Current State of Research

Composites are made up of two or more materials which produce together the desired properties that cannot be acquired with any of their constituents [3,4, 5]. Composite materials are ensembles of at least two individual materials which need to ensure the establishment of interface and sometimes even crystallographic bonds [6, 7, 8, 9]. One of the components, the ductile one, named matrix, ensures the load transfer towards the latter, called fibre, which is most often hard. The phenomenon is based on the difference in the elasticity of the components [10, 11, 12].

Polymeric materials have been used since prehistory. Polymers are found in nature, in all living systems and in materials such as wood, paper, leather, natural fibres [13,14,15]. While natural polymers preserve their intrinsic importance, synthetic materials are mostly used nowadays. The first man-manufactured polymers appeared in the latter half of the 19th century, by chemical modification of natural materials [16, 17, 18].

Polymers and reinforced plastic materials are used in various significant applications, from household appliances to aerospace products [19,20]. Polymers have a wide variety of physical and mechanical properties suitable to a large number of technical applications. The chemical structure, average and distribution relative molar mass, chain conformation, morphology, additives and fillers define the properties of both individual and hybrid materials, and allow specific application for sophisticated designs of materials [21].

These materials are light, strong and relatively cheap, but they can be difficult to manufacture in complex geometries [22]. The light design becomes increasingly important in various industries, especially in aerospace industry, wind energy and automotive applications [23, 24, 25].

Fibre-reinforced polymers have become some of the most important materials for engineering applications, due to high rigidity, performance to fatigue, good chemical and thermal resistance and low costs [26, 27, 28]. Other properties which deem them applicable in all industrial areas are: low density and high strength. Owing to their advantages, fibre-reinforced composites are increasingly used to replace the traditional metallic materials, being widely used in aircrafts, ships, defence, automotive structures, sports equipment, ground transportation, constructions, etc. Unlike monolith metallic materials, fibrous composites are customarily used as laminated products with various layer orientations, each layer being made up of fibres and matrix constituents [29, 30, 31]. This is why the ways in which fibrous composites fail are much more complicated. With a view to exploring the potential of composites in structural designing, understanding their failure mechanisms becomes extremely important [1, 2, 3, 4]. In these composites, the fibres are the main component mechanically speaking, while the matrix material binds the fibres together, acting as loading transfer environment between fibres, and protects the fibres from the environment (e.g. humidity, etc.) [32, 33, 34].

The mechanical behaviour of a laminate depends to a great extent on the directions of the fibres; consequently, the laminate must be protected in order to meet the specific requirements for each and every application, with a view to obtaining maximum advantage from these materials. The structural and optimization analysis of the correct and effective procedures is of the essence in fulfilling this task [35, 36]. The shared objective of optimal designing of laminated composite structures [37] is that of determining the layer thickness, orientation and the number of layers which provide the structure minimal mass, which has to correspond both to the imposed constraints [38,

39] and to the adopted failure criterion [40]. An integrated model for the optimal design of the mass of laminated composite plates under dynamic loading is presented in Figure 1,

Research carried out at both national and international levels for various layered composite materials reinforced with glass fibre, carbon or Kevlar has focused on the study of the effects of fibre orientation and lamina positioning on the mechanical properties of these material, at different loading combinations [41, 42]. Also, there have been conducted studies on the behaviour of polymeric composites in tensile and bending, for determining their rigidity, elasticity and other mechanical properties [43, 44].

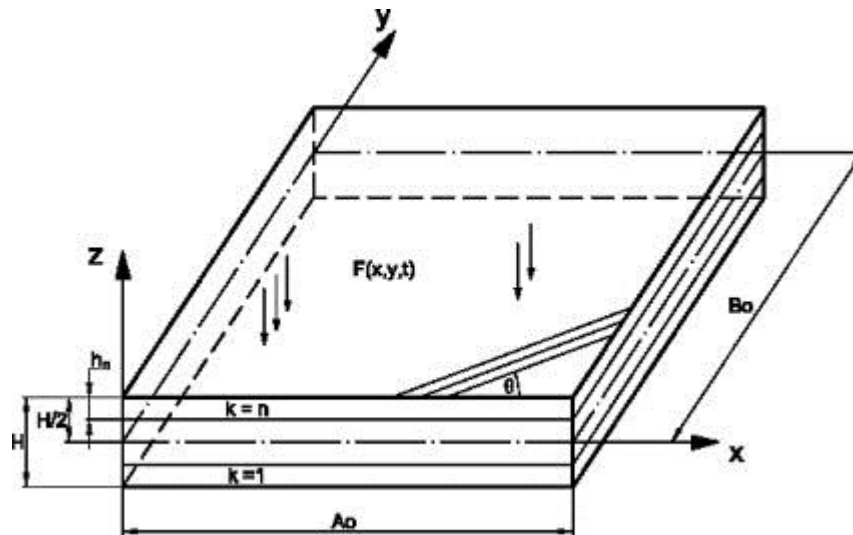


Fig. 1. Geometry and loading of the layered composite plate [45].

Carbon-fibres reinforced composites present excellent mechanical properties but high fragility. Hybridization with self-reinforced polypropylene (APRS) [46] is a promising strategy of improving the tensility of carbon fibre-reinforced polypropylene (CFRPP) [47].

In hybrid composites, layers may include two or more fibre types, e.g. carbon fibres and glass fibres or glass fibres and aramid fibres, etc. [48]. Hybrid composites provide extensive possibilities of controlling strength, stiffness and, last but not least, costs. An extremely promising application of these materials is associated with the so-called thermostable structures, whose size does not modify at thermal changes (heating or cooling) [49].

A low mass of structure conjugated with high rigidity and good fatigue durability has led to the advancement from metallic to composite structures. This aspect brings forth new concerns related to the certification of the new components as failure mechanisms and durability requirements of the composites [50] significantly different from those of the traditional materials (metals) [51].

As well-known, one of the greatest disadvantages of fibre-reinforced laminated composites is the discontinuity of the material properties between adjacent layers [52]. This defect is critical for the lifespan of the laminated composites, as these properties' discontinuity may lead to severe stress concentrations and complex stress states at the interface level, even on conditions of simple plane loading [53].

Chapter 2. Research Objectives

After perusing the bibliography on forming, characterizing and testing fabric-reinforced composites with epoxy matrix, a few very important conclusions have been drawn. Perhaps the most important of all concerns the fact that obtaining a valuable composite is an ample process that starts with design, continues with selecting the materials and the manufacturing technology and is concluded with the characterization and testing of the material. The aim of this research work is that of analysing the properties of fabric-reinforced polymeric composites and of determining the way in which using one resin or another produces changes in the (mechanical, thermic or electromagnetic) behaviour of the formed material. To the same extent, another aim of this research is concerned with checking the functioning of the laminate model for fabric-reinforced composites, setting out from the study of fabric-reinforced lamina (i.e. of a fabric placed within a polymeric matrix).

In order to attain these aims, the following objectives have been set:

- identifying the optimal means of lamina manufacturing so that the existence of a sufficient number of test specimens be ensured;
- finding the ways of mould release of materials so that moulds could be reused (considering that epoxy resins are extremely adherent);
- establishing the forming conditions for each resin type (with special emphasis on gel time);
- preparing the fabrics, which needs to include polymer adherence tests in order to underline the potential necessity of preparing the surfaces;
- extracting the test specimens necessary for lamina pull-off tests
 - tests conducted both on warp (pre-tensioned) and weft direction for the identification of the difference in the values of elastic constants
 - tests carried out for lamina oriented at different angles (30° and 45°) in order to check the applicability of the calculation pattern for the elastic constants of laminae oriented at various angles, in the case of fabric-reinforced lamina;
- actual tensile testing of laminae for determining their elastic parameters;
- statistical analysis of the results in the tensile tests;
- comparative analysis of the tensile tests results in order to identify the contribution of each matrix type;
- comparative analysis of the tensile tests results in order to identify the contribution of each fabric type;
- electromagnetic analysis of laminae – determination of electric conductivity;
- thermomechanical analysis of laminae – determination of linear expansion coefficient perpendicularly on the reinforcement plane;
- formation of the composite plates reinforced with nine layers of fabric;
 - choosing the resin (based on the results obtained for laminae);
 - choosing the forming method;
 - preparing the fabrics;
- extracting the specimens necessary for materials' mechanical testing;
- conducting static tensile tests on fabric-reinforced laminated materials;
- statistical analysis of tensile tests results and determination of the relevant elasticity constants;

Chapter 2. Research Objectives

- determination by calculation, applying the laminate model, of the elasticity constants of the laminated materials;
- comparing the results obtained with results calculated with the help of the laminate model, using the experimentally determined elastic constants of lamina as data entry;
- conducting dynamic tests on laminated materials;
- establishing the cyclical loadings conditions and experimentally determining the elastic constants of the materials after the conclusion of the cyclical loadings;
- conducting tests for determining the electromagnetic properties of the materials;
- analysis of the electromagnetic properties of laminae in order to identify the influence of reinforcement and matrix, respectively;
- conducting tests for determining the thermal properties of the materials;
- analysis of the thermal properties of laminae in order to identify the influence of reinforcement and matrix, respectively.

Chapter 3. Experimental Materials and Methods

Customarily, new materials emerge as a result of the necessity of improving the effectiveness of structure and performance and, as a rule, new materials, in their turn, provide new opportunity for development. Structural materials should display a number of physical, chemical and other properties, but there are at least two main features of utmost importance, i.e. rigidity and strength, which impress the structure the ability to maintain its shape and size under loading or under any other external action [54].

Fibre-reinforced polymers (FRP) are more and more used in various applications, from metropolitan infrastructure to aerospace industry. Their advantages are:

- low density;
- resistance to corrosion;
- longer durability and low maintenance costs;
- high mechanical strength [55].

In order to capitalize on fibres' high strength and rigidity in a monolith composite material suitable for engineers, the fibres have to be bound with the help of a material whose strength and rigidity are naturally much lower than those of the fibres themselves, which is called matrix. The matrices provide the final shape of the composite structure and govern the parameters of the forming process. The best combination of fibres and matrix properties must meet a set of operational and production requirements which are, at times, contradictory. These requirements have been met for all composites already formed (and applied), even though this complex of requirements has not been explained [56].

Material performance is generally assessed by a size of variable value, such as: point shifting, maximum tension, etc. or by a set of variable sizes (i.e. variable value of the size). In the case of composites, variability of certain parameters occurs from the variability of the properties of their constituents, variability of their distribution, structural geometry, variability of the loading conditions and last but not least, variability of the forming conditions. As an orthotropic material, variability may lead to catastrophic failure, mainly when inexactness occurs in fibre loading or orientation, while the traditional approach to safety factors may result in useless and costly conservatism, a serious disadvantage to producing competitive and durable composites [57].

Fibre-reinforced composite materials are used in various applications in high-technology industrial sectors, such as: aerospace, automotive and energetic (wind energy) industry. The high demand on the market determines the constant increase in forming composite materials or structures, which is also supported by very high mechanical strength, low density, and also by the manufacturing pace and the complexity of specific forming structures – when compared to metals – without mentioning the fact that, in the case of composites, complex finishing or coating is not necessary [58].

More recently, much attention is granted to hybrid composites, more precisely, to composites with the same matrix but different reinforcement fibres or matrices reinforced with similar fabric type but with different properties, or by complex composites reinforced with various fibre types modified by various agents and matrices made up of two or more polymers. When analysing the mechanical properties of the hybrid composites [59], the general rule of mixtures can be used for describing a certain property of the material, based on knowing the volume ratios of its components and on the properties of these components [60]. A positive or negative effect, in the case of a hybrid composite, is defined as a deviation, either positive or negative, of a certain mechanical property

from a reference material (usually, a two-phase composite). In addition, in what concerns orthotropic composites, a recent study on hybrid composites made up of carbon and glass fibres has shown that the hybrid composite displayed positive effects (increase of the value of measured parameters – strength) both in tensile and compression tests [61]. In the case of composites, either simple or hybrid, wide variability of the results is apparent, being induced, firstly, by variability of the components' properties, variability of the distribution of reinforcement fibres, structural geometry, forming process or testing conditions [62].

Research conducted in the last twenty years has revealed that fibre-reinforced composites with epoxy matrix display the best performances when it comes to mechanical properties. Two fundamental aspects should be underlined: on the one hand, the fact that epoxy resins do not fall under the environmental-friendly category, and on the other hand, the fact that, being thermoset polymers [63], composite structures cannot be obtained by pressing (but only by gluing). Thus, one of the determining advantages of fibre-reinforced thermoplastic matrices is lost – the high forming pace [64]. There is, however, a technical solution – lay-up, which allows using various fibre types and also matrix modification from one layer to the next, while ensuring stability of inter- and trans-laminate polymeric bonds, which, in turn, allow the material to behave as a whole when subjected to various loadings.

Another problem concerns the difficulty of maintaining the regular distribution of fibres during forming, all the more as the lay-up entails mechanical spread of the polymer (pre-polymeric mix) over the fibres using brushes or spatulas. For solving this issue, an attractive solution is that of using bidirectional fabrics instead of orthotropic pre-pegs oriented to various angles (to reduce anisotropy). In this case too, one could mention a drawback – the fact that fabrics generally used as reinforcement elements (carbon, glass or aramid fibres) are unstable, the regular distribution being disturbed by any tension determined by the movement of the brush or spatula. The great advantage of using fabrics is also related to the fact that their use allows obtaining structures with sinuous and complex surfaces in which transversal density of fibres is constant. In general, the mechanical properties of a reinforced composite obtained by lamination can be assessed (as a first approach) by using the well-known laminate model, in which lamina is considered a constituent. In the case of fabric-reinforced composites there is, however, a problem relative to the way in which the elastic properties of laminae could be described – in terms of elastic properties of fibres, mechanical properties of matrix and geometric and engineering parameters of fabric [65].

This study has set out from the idea of fatigue testing of fabric-reinforced composites in order to understand their effects on the general properties of these materials, being further expanded towards experimental validation of two mathematical models widely employed in the analysis of composites: on the one hand, the laminate model when the elastic parameters of laminae are known and, on the other hand, the model of the elastic constants of laminae in a given system of reference, when the values of the respective parameters are known in a different reference system (which is, in fact, a significant component of the laminate model). The novelty element is brought by the analysis of the fabric-reinforced laminae (fabric layers imbued in polymer), pursued for three types of epoxy resins and 18 different types of fabric.

Firstly, there have been formed materials reinforced with a single layer of fabric (laminae) and with each of the three polymers described above as matrix. Thus, 57 such materials have been formed (three for each fabric type described above, one for each type of resin and, in addition, for the carbon-aramid mixed fabric, for each resin another material has been formed, in order to emphasise the difference between warp and weft). All these materials have been formed so as to

determine the elastic parameters of laminae, which may be used in the description of a laminate, thus also procuring necessary data entry for the simulation of a layered material.

For each fabric type, materials reinforced with one layer of fabric (laminae) have been formed using each of the epoxy systems described above as matrix. In order to ensure a sufficient number of specimens, actually, for each pair fabric-epoxy system, two reinforced plates with one fabric layer have been formed. In all cases, the fabric was cut along the length of weft and warp.

For the production of the materials, two glass plates of 650 x 650 mm were used. For the more effective extraction of polymeric composites from the matrix, polypropylene sheets as mould-release agents have been inserted between the glass plates and material. The sheets have good surface quality and enough rigidity to maintain the desired shape. After placing the layer, the matrix is closed, and by applying pressure, the resin surplus and the gases emitted during chemical reactions or introduced during the mix of the two components of the epoxy system are eliminated.

After polymerization of the epoxy matrix from laminae, specimens have been cut for more test types, specific for the determination of the mechanical, thermal, electric properties (Fig. 5).

A laminate is a collection of piled-up laminae arranged so as to obtain the required rigidity and thickness. For example, fibre-reinforced unidirectional lamina can be arranged so as the fibres in every laminate be oriented in identical or different directions [66]. The sequence of layer orientation in a laminate of a fibre-reinforced composite is called lamination scheme [67]. The layers are generally joint together by the same material as the matrix (the one used in lamina formation) but this system is valid for thermoplastic polymers. In this case, lamina (composites themselves) are firstly piled-up in the sequence and orientation designed, and then the package is introduced into a presser and heated up to the temperature corresponding to polymer melting. After cooling, repolymerization ensures the inter- and intra-lamination bonds, providing the structural integrity of the laminate. The lamination scheme and the individual properties of a lamina of the composite material provides excellent flexibility for the designer to adapt the laminate rigidity and strength in order to meet the rigidity and structural strength requirements [1].

A typical composite laminate consists of individual layers, usually made up of unidirectional layers with the same or alternating regular alternance [68]. One layer may also be made up of metals, thermosetting or thermoplastic polymers and fabrics or may have a reinforced three-dimensional spatial structure [69]. A typical composite structure consists of a system of layers bound together. The layers may be made up of various isotropic or anisotropic materials and may have various structures, widths and mechanical properties. By contrast with the typical layers whose basic properties are experimentally determined, the characteristics of the laminates are customarily calculated using information regarding the number of layers, their piling-up succession, geometry and the mechanical properties of laminae, which should be known [70].

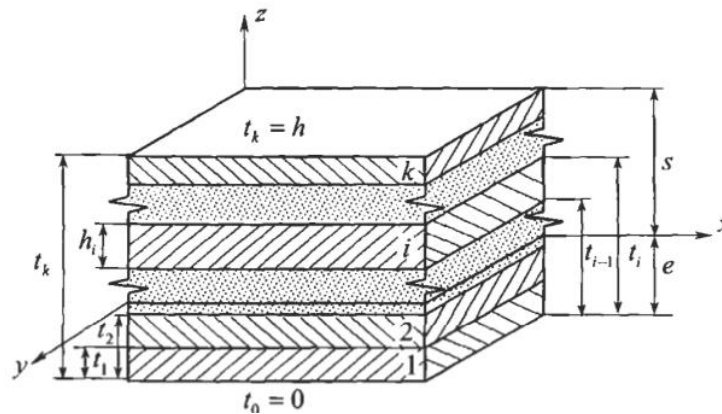


Figure 6. Laminate structure [38]

Having drawn a number of conclusions after the mechanical, thermal and electrical tests on fabric-reinforced laminae, the formation stages and architecture of the laminated composites have been established [71]. For the formation of laminates, fabrics of 650 x 650 mm and fibre orientation at 0° have been cut.

Starting from the expertise acquired during the production of laminae, the method remains the same but, in this case, in order to ensure higher volume ratio of the reinforcement, more fabric layers will be introduced into the matrix. In laminate formation, the unidirectional laminae have been overlapped so that laminae fibres be oriented identically for each lamina, fabrics being imbued with enriched resin. Laminae are glued with the same material as the one forming the laminae matrix. The matrix was formed using a mechanical mixer for homogenizing the resin with the hardener. In what the matrix type is concerned, all composites have been formed with epoxy resin of the EPIPHEN RE 4020 and EPIPHEN DE 4020 hardener, as, from the analysis of the experimental data obtained after laminae testing, we note that this epoxy system provides the best properties and it is most easily handled because of its longer gel time. It is worth mentioning that, fortunately, this epoxy system is also the cheapest of the three used in the former part of the study.

The forming method has been similar to that used in laminae manufacturing, consisting of the placement, layer by layer, of fabrics imbued with pre-polymeric mixture, which ensures the inter- and intra-laminar bonds. The materials, of generous sizes, two plates for each, have been extracted from matrices and left for two weeks, for complete polymerization. After this period of time, before extracting the specimens necessary for mechanical testing, the electromagnetic properties of the materials have also been assessed.

According to the size of the plates (especially thickness, which varies for each material because of the differences in fabric thickness), we then designed the specimens necessary in static and dynamic testing. As in the case of laminae, specimens have been extracted both on warp and weft direction, and also specimens oriented at 30° and respectively 45° on warp direction. Because the initial sizes of the laminated plates did not allow their being introduced in the drying cabinet, the thermal treatment of these materials has been applied directly to specimens, after their extraction by high-pressure water jet cutting.

There have been produced 16 laminated materials with 9 layers each (each one in the form of two plates), in which all reinforcement layers (laminae) display the same beam orientation and, obviously, thicknesses of these materials are smaller than nine times the thickness of the lamina obtained using the same fabric and the same epoxy matrix.

Chapter 4. Thermal properties of laminae

4.1. Specific heat

Differential scanning calorimetry (DSC) is a thermo-analytical technique widely used to determine the thermo-active processes that occur in materials during a well-defined programme of exchanging energy in the form of heat. [72]. The differential scanning calorimetry technique is used to detect and measure the thermal event in a material. These thermal events include melting, crystallization, chemical reactions and volatilization [73].

One of the most important parameters that can be evaluated using this technique is specific heat [74]. In addition, this technique, due to highly precise control of the variation of temperature, allows the determination of some critical values for materials – boiling point, melting point, glass transition temperature (in the case of polymers) or, in some circumstances, heating value (when combustion of sample is possible) and ignition value. DSC presents the advantage of directly and precisely measuring the exothermic and endothermic heat at constant scanning rate [75].

In order to obtain exact measurements, the tested samples have been kept in similar conditions. The STARe software application allows the direct assessment of the specific heat on heating and, respectively, cooling curves of the material under evaluation. In order to ensure accuracy of determinations, specimens have been weighted before and after the determination of the specific heat, they weighing from 1.3 to 5.8 mg.

The curves presented in Fig. 8 represent the heat flow for a carbon-fibre fabric reinforced and epoxy resin HT2 sample (lamina) in heating and cooling conditions. They are the basis for selecting the temperature intervals for the study of specific heat for the formed materials, without considering high temperatures, as long as the resin producer recommends usage at temperatures under 200°C. A cyclical thermal programme (heating-cooling) has been applied twice for each material, noting a peak at the first heating, which may correspond to composite consolidation. Certainly, the peak notion is not the most appropriate in this case, nevertheless, in the graph below, on the ordinate axis energy loss from the flow generated by the machine is indicated, which, as far as the sample is concerned, represents energy absorption.

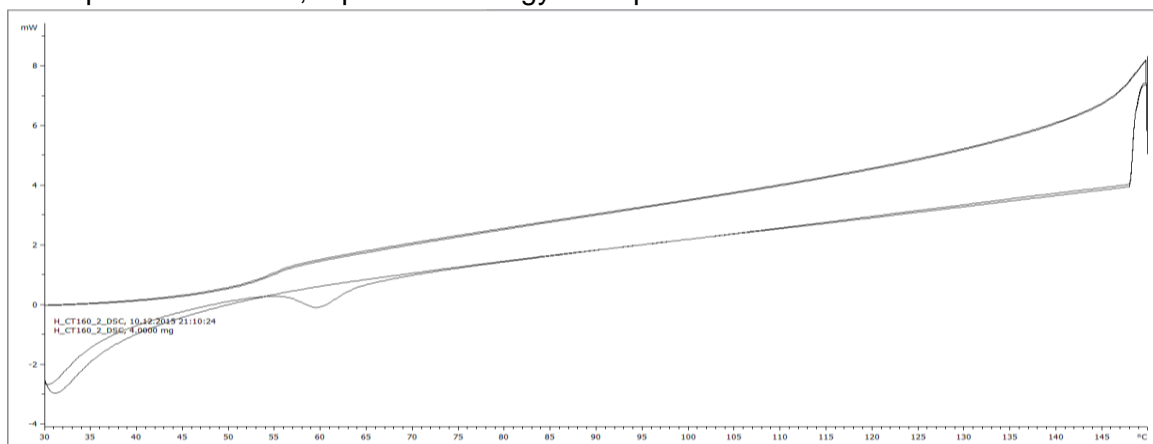


Figure 8. DSC curve specific to a carbon-fibre fabric-reinforced and epoxy resin HT2 lamina.

As the volume of the sample is provided as entry parameter, the software application may display as exit parameter the value of specific heat of the material (the amount of energy that the sample must exchange with the exterior so that the value of the temperature of the sample to

alternate by a unit). In fact, the application generates a curve (specific heat value according to temperature value), being known that, for any material, specific heat has low dependency on temperature. The method of data analysis is that of assessing the mean specific heat at various temperature intervals, i.e. the ones on which the curve of specific heat keeps the same gradient..

4.2 Coefficient of thermal expansion

Size stability of composites is one of the most important qualities required, given the very wide area of application for these materials. Generally, polymers are substances whose size stability is fairly low (owing to their chemical structure based on covalent binds) at temperature limits that do not affect their structure (oxidation for thermosetting polymers and melting, eventually followed by oxidation, for thermoplastic polymers). As in the case of mechanical properties, polymer reinforcement may have either positive or negative effects on size stability. It is obvious that the composite expansion is determined both by the polymer (matrix) expansion and by that of the fibres (reinforcement), their mediated effect (expressed by the coefficient of linear thermal expansion) depending to a great extent on the nature and quality of the matrix-fibres interphase. However, in the case of laminae, the thermo-mechanical effects of material heating depend less on the temperature gradient corroborated with intra-laminar tensions, given the fact that lamina thickness is extremely reduced when related to the laminate thickness. These effects may occur (but not because of the temperature gradient) at areas where warp fibres go under or above weft fibres.

The measurement of the coefficient of thermal expansion has been pursued, for each lamina type, with the TMA-SDTA 840 (Mettler-Toledo) thermomechanical analyser and the STARE software application (Mettler-Toledo). Basically, the determination of the coefficient of linear thermal expansion is acquired by permanently measuring the thickness of the material under analysis at various temperature values (the temperature regime is controlled by the software application). In this context, a significant source of errors is generated by placing the press ram on the sample, as it can be placed, for example, in the area where warp fibres go above or under the weft ones. In such a case, the results will be greatly influenced by an oblique position of the press ram on the ideal measurement surface (which should be perfectly plane). As the press ram presses on the sample (0.02N loading) and because a spherical head is used during measurement, it could slide uncontrollably during measurements.

4.3. Conclusions

The thermal analysis of fabric-reinforced laminae reveals the great dependence of the analysed parameters (specific heat and the coefficient of linear thermal expansion) on fabric type and matrix used. At the same time, for both parameters, one notes low dependence on temperature (increase in values at the same time with the increase in temperature value).

Apparently, E-type resin needs longer thermal treatment for obtaining its best characteristics and, at the same time, it is noticeable that it is the least rigid of the three epoxy systems used. Even more important is the fact that, as far as size stability is concerned, materials reinforced with fabrics made up of similar fibres display an approximately identical expansion behaviour. The differences could be accounted for based on the differences between fibres volume ratios.

In the case of fabrics considered as reference for the study of specific heat, one notes that the variation profiles of this parameter, at the analysed temperature intervals for laminae with C- or HT-types matrices are almost identical, which may signal very low variations of the formulae of the resins used.

Ana BOBOC (CĂPĂȚÎNĂ)

In all cases under analysis (fabric-resin pairs), one could notice that the values of specific heat at the latter heating and, respectively, at the latter cooling, are lower than the ones recorded during the former analysis cycle, which signals the material consolidation (as a fundamental stage in forming a composite, according to various authors), although all materials have been subjected to thermal treatment as recommended by the resins producers.

Chapter 5. Mechanical properties of laminae

5.1. Laminae

The laminate model, as argued by numerous authors in the field of composites, allows the prediction of the values of mechanical parameters of a laminated material when the values of these parameters are known for its constituents – layers or laminates. Within the framework of the same model, but as part of the demonstration of the properties of laminae, one finds the model of determining the values of elastic parameters of the lamina in a reverse reference system from that for which experimental determinations are made (regarded as fundamental and having one axis along the reinforcement fibres). The assumed aim of this study was that of checking the possibility of using the laminate model for fabric-reinforced laminae.

A clear distinction should be made between materials with thermoplastic polymeric matrix and with thermoset polymeric matrix. In the case of the former, orthotropic laminae can be easily obtained, i.e. uniformly distributed, parallel fibres introduced in polymeric material (polyethylene, polypropylene). Technology is not complicated, consisting in unwinding the fibres on the shafts on which thermoplastic sheets are usually formed. These reinforced sheets can be further piled-up (at various fibres orientation) and the packs thus obtained can be consolidated in presses at controlled temperatures. Under these circumstances, the polymer melts and reorganises, leading to the formation of a composite material that can be unidirectional, symmetric, asymmetric, balanced or unbalanced (and other categories which are always present in the case of an exhaustive tackling of composite laminates).

Things are different when it comes to thermoset polymers, as these polymers present special conditions of triggering polymerization reactions (UV exposure, substances mix, spraying with polymerization-triggering agent, using catalysers, exposure to light and, sometimes, even heating), so that the forming conditions of orthotropic laminae are more difficult. Once obtained, orthotropic laminae cannot be packed as laminates by simple pressing and heating but they need to be glued using adhesives. However, the use of adhesives generates a series of problems which do not occur in the case of thermoplastic matrix laminates – adhesive strength, adding to that of the polymer and of the reinforcement elements), strength of the matrix and adhesive bond (a polymeric joint), the influence of the presence of the adhesive on the general properties of the laminate (not only mechanical but also thermal). If in the case of laminates one speaks of thermo-induced inter- and intra-laminar tensions, in that of the thermoset matrix laminates, one should also consider the thermo-induced inter-laminar tensions between polymer and adhesive. It is obvious that a number of these issues can be solved by using, wherever possible, an adhesive similar to the matrix (which is not possible, according to our tests, for epoxy resins).

Using fabrics would simplify, to a certain extent, the undertaking of obtaining thermosetting matrix laminae but it would not solve the adhesive issue at the same time. Fortunately, the wet lay-up method allows obtaining materials that can be classified as laminates, being much more advantageous than the methods employed for thermoplastic polymers because, in the case of thermosetting polymers, they could be modified by dispersing agents with well-known effects (carbon nanotubes, starch, nanoferrites, other organic or inorganic agents – the list can be continued by mentioning the research of my colleagues at the Research and Development Centre for Thermoset Matrix Composites) – which eventually contributes to changes in the laminates properties (and why not, in the control of their properties)

Although simple and not requiring using sophisticated equipment, the wet lay-up method cannot be used for manufacturing laminates with orthotropic laminae (because of the impossibility

of maintaining the reinforcement elements distribution during the process), which is why, in this case, fabrics are easier to use. At the same time, the method ensures (after the completion of polymerization) the presence of the same matrix type in the entire material. Provided that the matrix-reinforcement agent interphase is qualitative, the material obtained is also qualitative.

This is the moment when trouble occurs. It is possible to describe the mechanical properties of a fabric-reinforced laminated material in point of mechanical properties of its laminae. Certainly, as shown in the chapter on material forming, both laminae and laminates were formed using the same technique (wet lay-up) but there is no guarantee that all elements which may influence the properties were controlled (especially the fabric layers sliding when placed in the matrix or the almost unavoidable sliding – at microscopic level – of the beams of warp and weft fibres during the impregnation with pre-polymeric mix).

There are in the literature a few attempts at describing a fabric-reinforced lamina, the current trend being that of considering the waviness of the fibre beams making up the fabric or (a previous model), the description of such a lamina as superimposition of two orthotropic laminae (a model in which the problem of the fascicle deformation occurs, without introducing the waviness notion). Intuitive is the fact that a fabric-reinforced lamina will respond differently from an orthotropic lamina when applying a load test, for instance. Why is it intuitive? Easy! Fabric means placing weft fibre beams over and under warp fibres fascicles. When applying tension on the warp direction, the beams readjust without the possibility of avoiding the more accentuated deformation of the weft beams. If the fabric is made rigid by inserting it into a matrix (of any type), when the warp beams stretch, the weft ones, more rigid, will fracture instead of becoming more deformed. This is just an intuitive presentation. The analysis can be furthered, as the polymer's presence among the beam of warp or weft fibres, not to mention the polymer that ensure the bind of the fibres from both beam types, produces micro-mechanical effects that are difficult to describe and that are not taken into consideration when analysing orthotropic laminae. When these aspects are considered, it is almost natural that the properties of fabric-reinforced laminae are very difficult to describe in terms of fibres and matrix properties. The way in which a fabric-reinforced lamina may behave could be described (in terms of numerical analysis) as a welded network in which the wires would go above and under other wires before welding. Even in this case, we should assume (for the sake of simplification) that the wires and the welding points are identical and perfect. Valuable results could probably be obtained empirically, although identical testing conditions cannot be ensured (regardless of the efforts made) for fabric-reinforced lamina, the fabric itself and the matrix (in the form of a layer whose thickness equals that fabric thickness, and which is usually submillimetric).

In this context, the present study set out from the idea of forming laminae from a number as large as possible of fabrics and using as many polymers as matrix as possible. By testing these laminae, data obtained could be used to validate the two assumptions mentioned in the beginning of this chapter: on the one hand, checking the model for the assessment of the laminae loaded at various angles, and on the other hand, comparing the results obtained for laminae to the results obtained for laminates formed using the same fabric type.

5.2. Tensile testing – axial loadings

Tensile testing is one of the most important tests that focus on determining the mechanical properties of materials. They actually become the most important when the materials are used for manufacturing benchmarks [76]. At the same time, however, these tests are, probably (alongside the compression ones) best approached theoretically. Of course, this presentation refers to

homogenous and isotropic materials, but it should not be disregarded either when it comes to reinforced composites (which do not meet any homogeneity or isotropy criteria, this being the major difficulty when the numerical analysis of these material is in view). Any attempt at numerical description of a fabric-reinforced material will encounter difficulties, even when the material is subjected to a very simple axial loading.

Pursuing tests on laminae is necessary for determining their mechanical properties with a view to using these parameters when making the right decisions in the design of laminated composites. Properties considered in laminae testing are [77, 78, 79]:

- longitudinal and transversal elasticity modulus;
 - Poisson ratios;
 - Strength in longitudinal and transversal strain.

In order to obtain correct results, a series of factors must be taken into account, factors that directly influence strength, i.e. the specimen shape and size, loading speed and working temperature (without disregarding humidity, in the case of composites with polymeric matrix). The technique of testing composites is [80]: the specimen (Fig. 9) is elongated along its main axis at constant speed until breaking or until tension (load) or deformation (elongation) reach the pre-set value. During testing, the loading (force) and elongation are measured [81].

What needs to be mentioned is the fact that the standard specimen presented below cannot be used for testing fabric-reinforced laminae or fabric-reinforced laminates. In the case of laminae, the most important problem refers to material thickness. In addition, the engagement area will not have any safety, nor will it ensure strain concentration along fibres. Cutting such specimens would directly affect the integrity of the material analysed, the process determining micro fissures of the matrix near the cut, due to combined rigidities of the material and, respectively, of the cutting tool.

These are the main reasons why all tensile tests in this study (on both laminae and laminates) have been carried out using rectangular specimens. These specimens are, in our opinion, more suitable in the case of composites reinforced with fabrics orderly distributed in the matrix.

For each tested specimen, there have been obtained a loading/ deformation curve, alongside with a set of values of elastic parameters, of more or less interest for this study. Of course, there have been carefully analysed the values of elasticity modulus and strength to breaking. Following this stage, the statistical analysis was carried out for data obtained for 10-specimen series (for loadings applied along the warp - 0° or weft 90°), and for 5-specimen series (for loadings applied at angles of 30° and respectively 45° on the warp direction).

Why both on warp and weft direction? Because fabric making entails warp pre-tensioning in order to ensure the weft stability during the process. In addition, following the fabric making, in order to ensure fibre and fibre beams stability, fabrics are covered in a very thin polymeric layer (of whose nature no manufacturer provides any information) and which actually represents a pre-matrix which, unfortunately, ensures and preserves internal strains – all these strains can determine small differences between the elastic parameters measured along warp and those measured along weft. It is true that most manufacturers of reinforcement fabrics warrant that any polymer may be used as matrix of a fabric reinforced composite, while also warranting the adherence of the respective polymer to fabric (in fact, to another polymer). As mentioned in the introduction, before forming laminae, we have conducted adherence tests and tests of imbuing the fabrics with each of the three polymers used as matrix in the study, and we have not encountered difficulties for any of them..

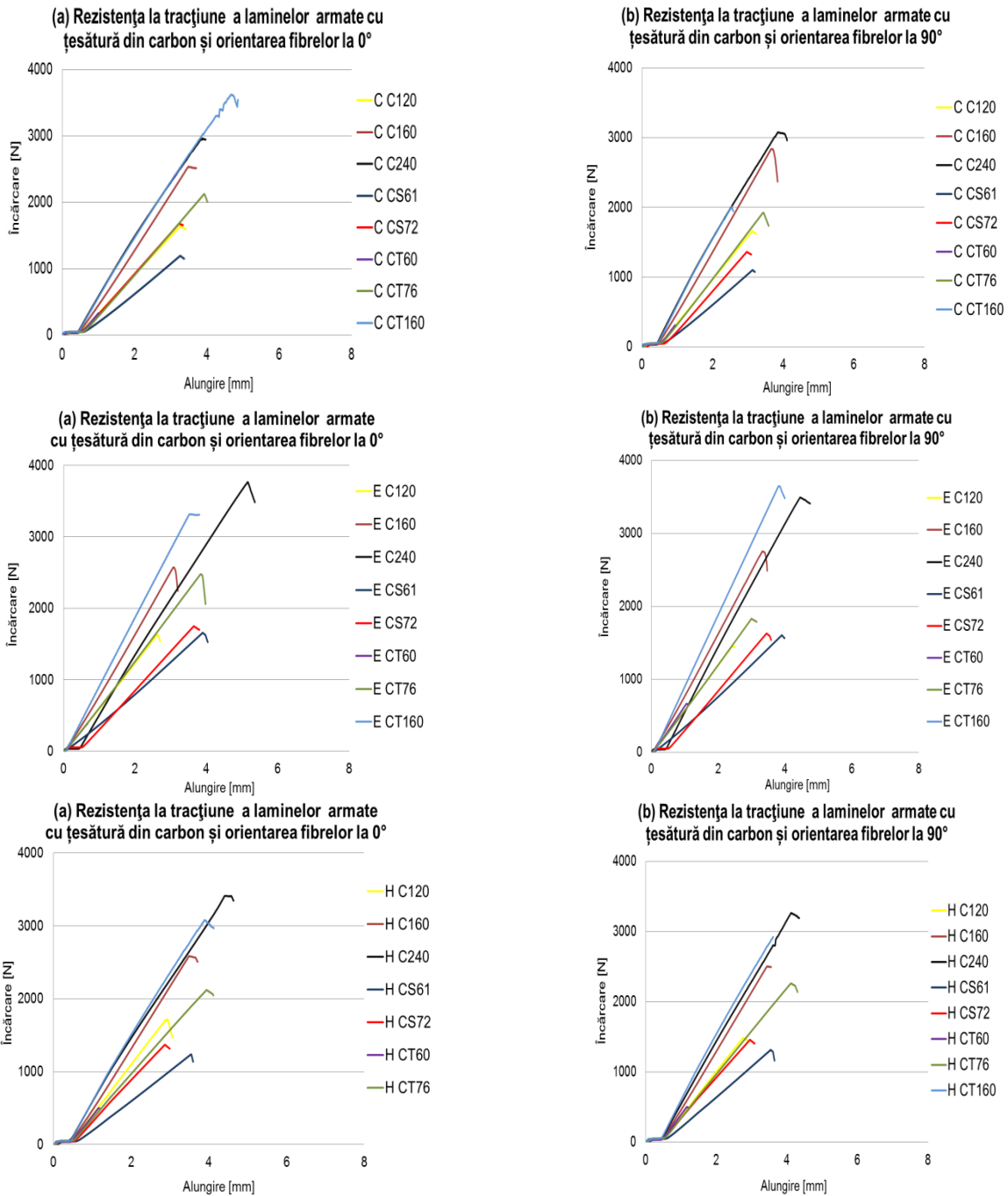


Figure 24. Load/ elongation curves (averaged for 10 specimens) for carbon-fibre fabric reinforced laminae with different matrices. Left – along the warp; right – along the weft.

Analysing the curves displayed in Fig. 24, one notes that the maximum loadings for the laminae on the weft direction are slightly higher than those on the warp direction (which justifies, to a certain extent, the assumption of the existence of some strains, presented in the opening lines of this chapter. In what the gradients of the curves are concerned, they do not seem to be significantly modified, but there are certain differences generated by the resin type used. In all curves one distinguishes the curves corresponding to C240 fabric, which seems to have the greatest strength, the only explanation possible being the big number of fibres on length unit. This also explains the results displayed in Figures 25-27 for laminae with C and respectively HT matrix, in which a very

dispersed and random breaking behaviour has been noticed. The explanation is that, in the beginning, fibres in direct contact with the matrix are fractured and, throughout the test, the fibres not bound to the matrix also break (the ones inside the beams, probably not reached to by the pre-polymeric mix used for impregnation because of high viscosity of the liquid and beam thickness). This behaviour is not encountered in the case of C160 fabric (thin beams), nor in that of E-type resin (more fluid and having longer gel time than the other two resins).

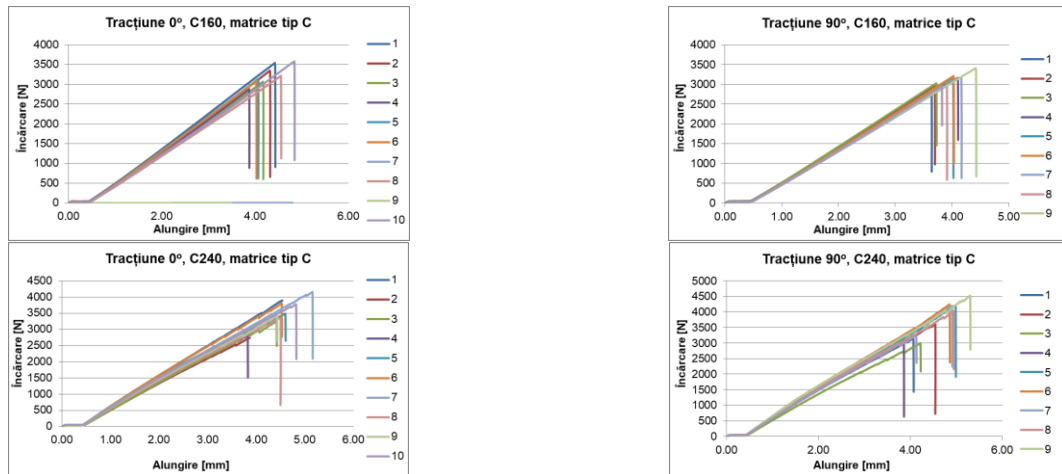


Figure 25. force/ elongation curves for fabrics made of identical carbon fibres and matrix made up of C-type resin.

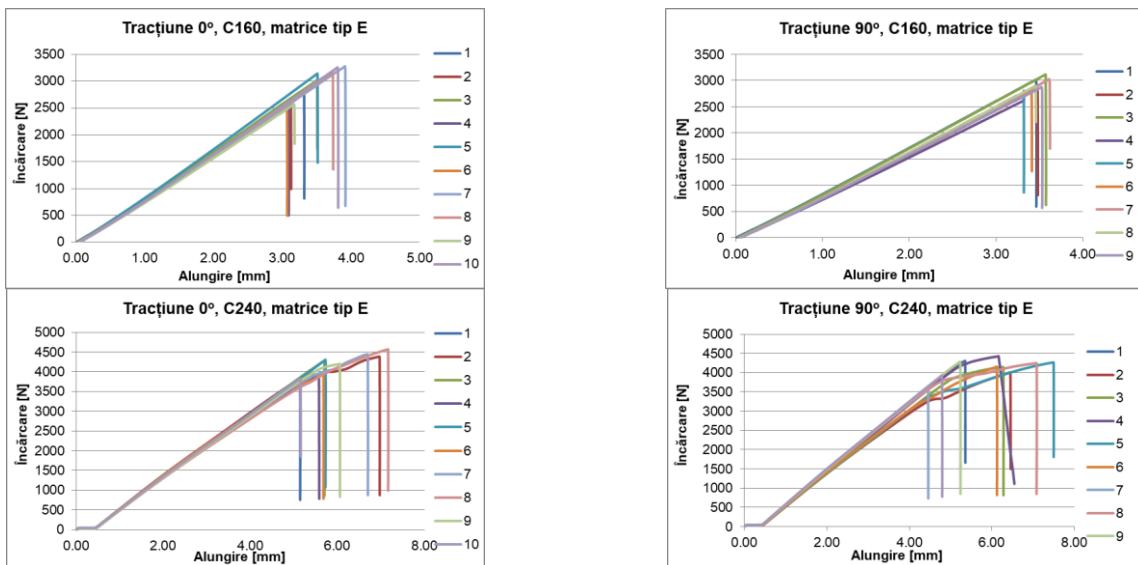
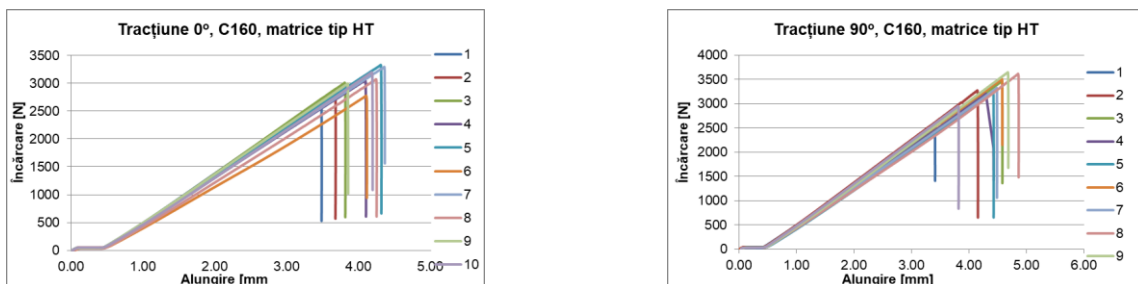


Figure 26. force/ elongation curves for fabrics made of identical carbon fibres and matrix made up of E-type resin.



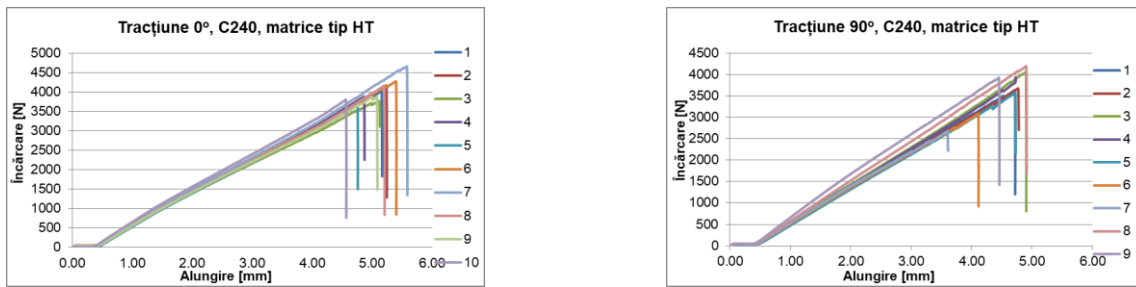
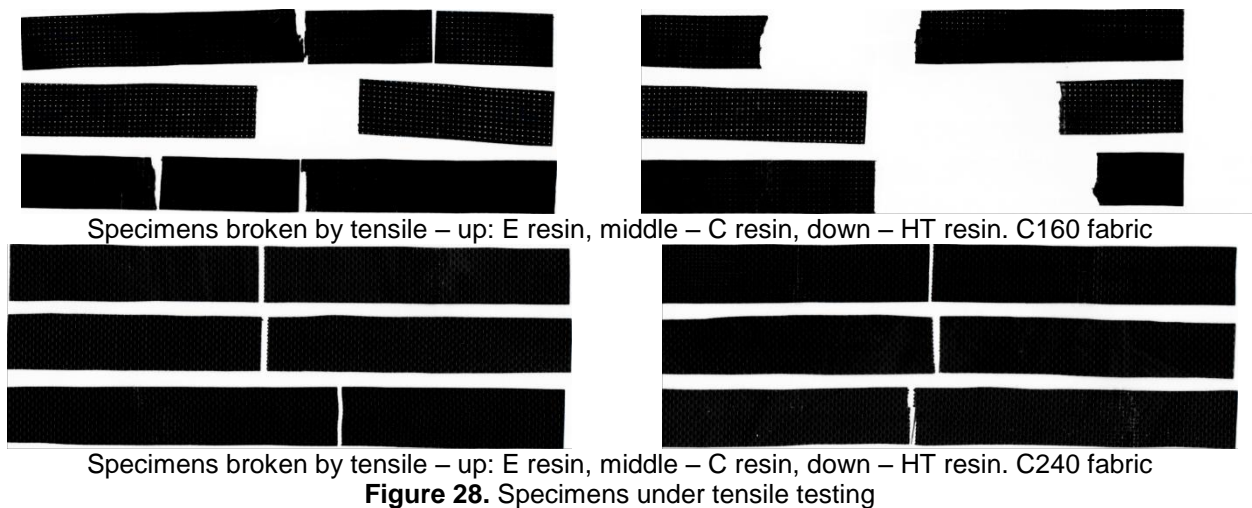


Figure 27. force/ elongation curves for fabrics made of identical carbon fibres and matrix made up of HT-type resin.

The visual analysis of the tested laminae is also a source of interesting information, as obvious from **Fig. 28.**



The differences between tensile effects on C160 fabric (up) and, respectively C240 fabric reinforced laminae (down) are easily noticeable. The images represent specimens engaged along the warp (the left column) and, respectively, specimens engaged along the weft (the right column). What is more, in the case of this fabric type, specimens are multiply fractured and, generally, fractures are perpendicular on the engagement direction.

In the case of C240 fabric reinforcement laminae, fractures on the threading dice are no longer visible; the materials are fractured at the engagement area and, sometimes, fibres extracted from the material as a result of fracturing can be observed. It is certain that the fracturing mechanism of the material is generated by matrix fracturing (in a sensitive area – along a fibres beam).

Another material with remarkable behaviour is that reinforced with CT160 fabric (a fabric manufactured using a special technology, Textreem, as indicated on the manufacturer's website, which consists of expanding a 3K beam of carbon filaments followed by the production of the fabric). In fact, **Fig. 24** does not exclusively display high curves for traditional carbon-fibre-made fabrics – the CT-coded fabrics (produced using the aforementioned technique) are made up of 1K, 2K or 3K expanded beams, the CS fabrics (Samurai – commercial brand) are also made up of expanded beams (but we could not identify a description of the technique employed).

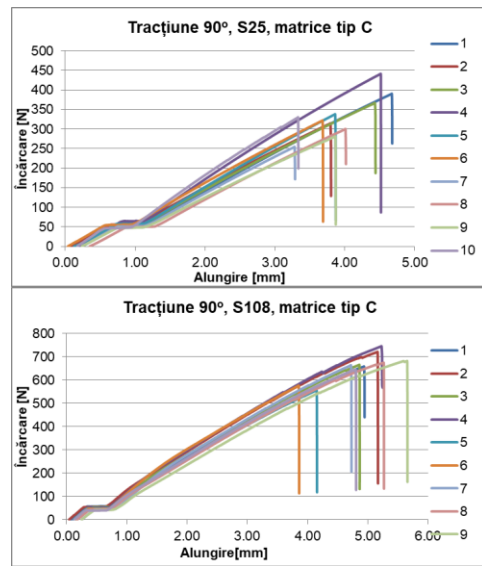
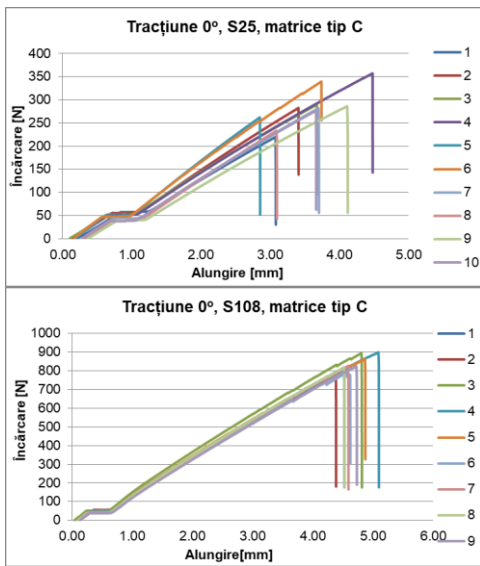


Figure 30. force/ elongation curves for fabrics made of identical glass fibres and matrix made up of C-type resin.

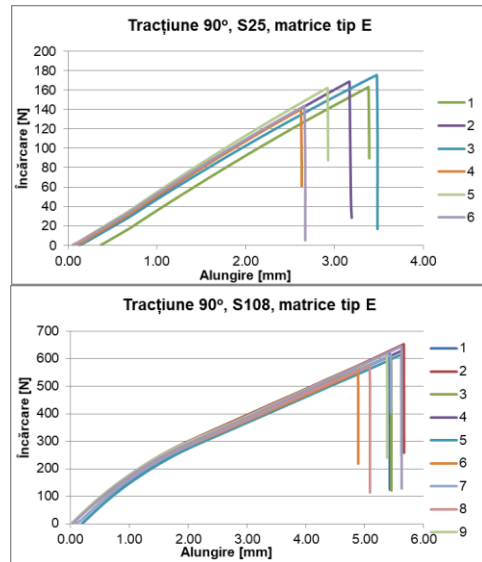
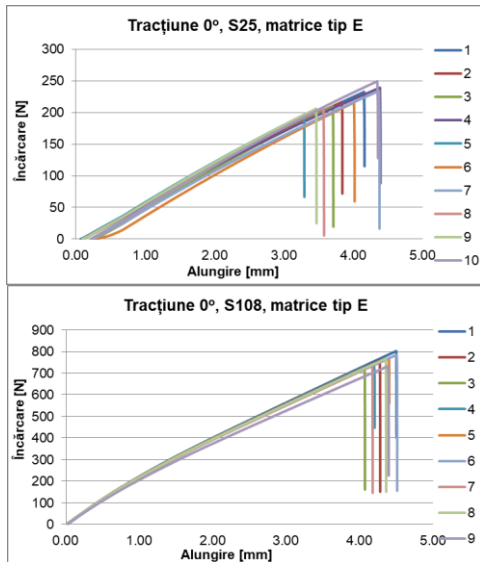
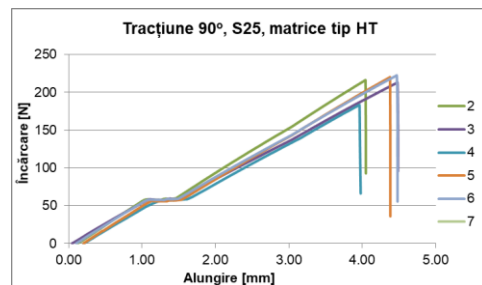
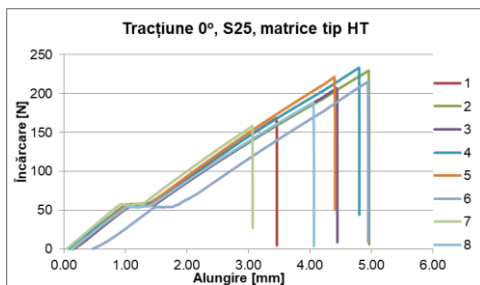


Figure 31. force/ elongation curves for fabrics made of identical glass fibres and matrix made up of E-type resin



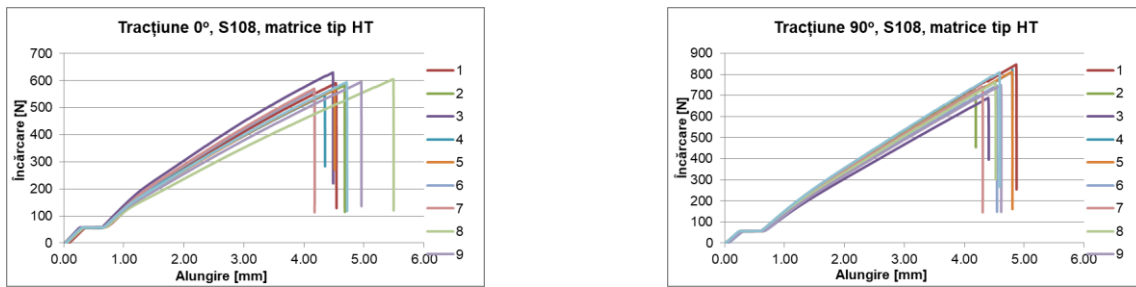


Figure 32. force/ elongation curves for fabrics made of identical glass fibres and matrix made up of HT-type resin

As in the case of thermo-dimensional analysis of laminae (presented in the previous chapter), the more detailed analysis of the results in tensile testing is pursued for laminae reinforced with fabrics made up of similar fibres in order to facilitate the understanding of the contribution of each constituent (matrix or fabric) to the breaking mechanism. **Figure 33** presents said laminae broken during tensile testing.

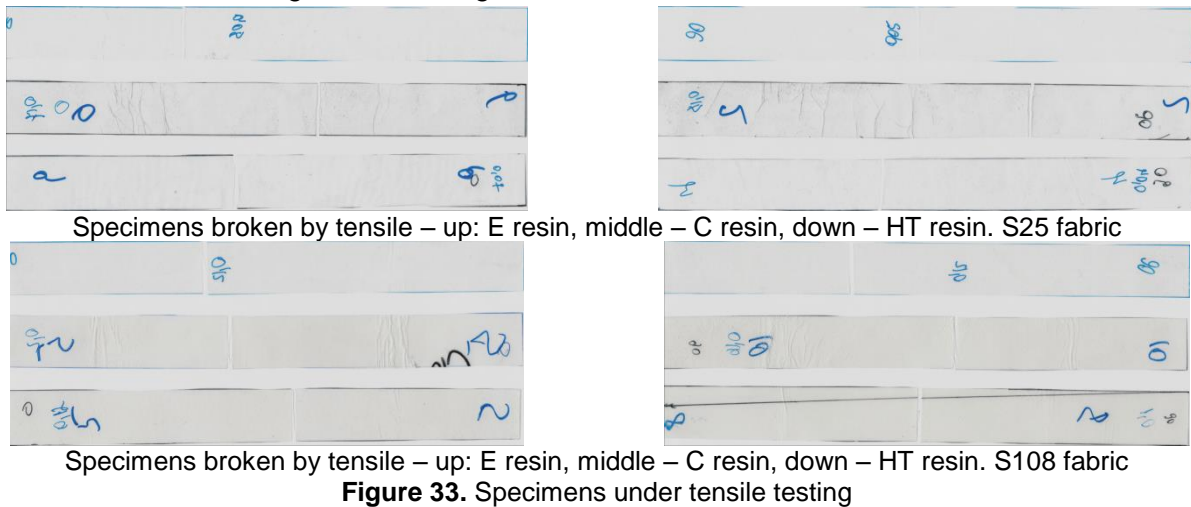


Figure 33. Specimens under tensile testing

As in the case of the two carbon fibres fabric types, it can be observed that the laminae fracture perpendicularly on the engagement direction. It can also be observed that, in the case of C and HT matrices, on the force/ elongation curves, right after the loading debut, there appears an horizontal bearing (unlike the E matrix), which corresponds to fibre tensioning. As the two matrices are rigid, they fracture at low loading values due to the matrix-fibre interphase (a very qualitative one thanks to the adherence of epoxy resins to glass). A wide dispersion of tensile behaviours for S25 fabric reinforced laminae is also easily noticeable for the two resins – given the extremely low density of the fabric, there are not sufficient fibres to mediate the response to loading and, practically, specimens break wherever a defect exists, be that difficult or impossible to detect – an indistinguishable variation in lamina thickness or the absence of continuity of a very small number of filaments in a beam. This behaviour dispersion (normal for any statistical series) is not as spread for S108 fabric reinforced laminae (although it also exists in their case). One also notes from the curves displayed in Fig. 30-32, that in the case of laminae with E-type matrix there occurs a fairly obvious concavity of the loading curves which corresponds to the behaviour to tensile of matrix type to behaviour of the fibre type (often described as failure mechanisms in the literature). This change in behaviour (sudden for the other two matrix types) is due to the fact that the matrix is less rigid. From this point of view, E matrix laminae come nearer what is basically expected from a composite

(the force/ elongation curve is practically linear along the entire loading domain). In the other two cases, although before and after the horizontal bearing behaviours are linear, the materials are composites only in the former region – after the bearing one can only speak of fibre behaviour, as the matrix has already yielded. Under these circumstances, it is as if the material was made only of fibres (but they cannot be maintained in a material without the matrix support). In other words, a material made up by layering a number of fabrics incorporated in a C or HT matrix would be good for testing for a certain application after the matrix fracturing, but the geometric integrity of the structure can no longer be guaranteed. It is not possible, however, to indicate the effect of alternating (in the same material) of the three resin types as it expected that the C and HT matrices better respond to transversal stress on the reinforcement plane.

In the case of reinforcement with the two fabrics made up of the same glass fibres types, the difference between the tensile tests carried out to the warp and, respectively, weft direction is obvious (the glass fibres have higher density and, consequently, maintaining the beams in a certain position entails more strain). What is actually even more interesting is the fact that, in the case of E-type resin, the loading along the weft leads soon to lamina destruction, whereas for the other two resins, the loading along the warp leads sooner to material destruction (which is explainable by the fact that fibres are not tensioned when applying loading in this direction and, after resin fracturing, fibres will be tensioned up to the tensioning level imposed on waft for fabric making).

Aside carbon fibres, aramid fibres are low-density fibres with remarkable properties which can be successfully used for polymer reinforcement. Unlike carbon fibres, aramid fibres have high shear modulus, which deems them highly valuable for the design and formation of materials able to resist at transversal loadings on the reinforcement direction (especially shock tests – and it is no longer necessary to stress the main application for these fibres – personal reinforcement). Aramid fibres, however, are not the best candidate for longitudinal properties, and consequently, fabrics of beams made up of these two fibre types have been formed. Although there are two fabrics made up of the same carbon and respectively aramid fibres, a comparison between the two (as the one above) should be made. One of the fabrics is still a simple fabric (each beam in the weft goes alternatively under a warp beam, then above the next warp beam and again under the next warp beam, etc.). This fabric is made up with a warp which has as a repetitive unit two carbon fibres beams followed by a beam of aramid fibres, while in the warp, the repetitive unit is made up of two aramid fibres beams followed by a carbon fibre beam.

All measurements performed allowed the determination of some elastic constants which may be used in the description of a laminate made up of the analysed laminae. In this case, a very sensitive issue was related to the determination of the Poisson ratios for these laminae, provided that the mechanical extensometer cannot be fixed on the lamina thickness (too thick) or width. Under the experimental conditions, it would have been almost impossible to determine the Poisson ratios by image analysis because the preparation of the specimens would have lasted, probably, more than a year. However, starting from the definition relations of Poisson ratios and taking into consideration the fact that the software application which allows the determination of elastic constants following mechanical testing does not operate any correction related to the transversal contractions in loading, we have evaluated the values of the Poisson ratios based on data obtained for elasticity modulus to the warp direction and, respectively, for elasticity modulus to the weft direction.

As it is well-known, in the case of plane loading of a lamina (disregarding its thickness), the deformation-load relations (on the two significant direction of analysis) can be expressed as follows:

:

$$\varepsilon_{xx} = \frac{1}{E_x} \sigma_{xx} - \frac{\nu_{yx}}{E_y} \sigma_{yy} \text{ și } \varepsilon_{yy} = \frac{1}{E_y} \sigma_{yy} - \frac{\nu_{xy}}{E_x} \sigma_{xx}.$$

The software application that allows the evaluation of the elastic constants of lamina following the tensile test assesses the elasticity modulus and, at the same time, allows the reading of deformation and load values. In this case, the value assessed for elasticity modulus (to the loading direction – the warp direction or 00) is not corrected with the contribution from the transversal direction, which is also valid for the perpendicular direction (when the test is carried out to the weft direction or at 90o), i.e. evaluations are carried out according to the following relations:

$$\varepsilon_{xx} = \frac{1}{E_x} \sigma_{xx} \text{ și } \varepsilon_{yy} = \frac{1}{E_y} \sigma_{yy}$$

Taking into consideration the fact that testing has been carried out on perpendicular directions (although it is known that differences may occur, as previously stated, from warp pre-tensioning), the data sets obtained may be used for the determination of the Poisson ratios (whose values – theoretically – should not be equal for a material). The last two relations can be used to determine (from the load-deformation curves) the elasticity modulus of materials to warp (E_x or E_xx) or weft (E_y or E_yy) direction as part of the graphs. Afterwards, these values are used for a point in the middle of the curve for determining the Poisson ratios. It is worth mentioning that all assessments have been made on the averaged load-deformation curves of the ten tested specimens.

Above, we have discussed the situation of the two types of brittle matrices (C and HT) in whose cases one can distinguish distinct planes of the load-deformation curve. From this perspective, one can only speak about composite material on the former part of the test (the horizontal plane representing, as already mentioned, the stretching area of the fibres released from the matrix, while in the latter part the behaviour is that of the fibres. For the laminae engaged to the warp and, respectively, weft direction, the load-deformation curves are presented below (Fig. 44-52) (previously, the loading-elongation curves have been presented) for thoroughly analysed fabrics, i.e. for fabrics formed of similar fibres. Based on these curves, the values of the elasticity modulus have been determined for the two planes (in the case of the C and HT matrices). Also, for these materials (with the obvious exception of the laminae made with E matrix), there have been assessed the values of the Poisson ratios. In addition, it is important to note that there are differences between the values of the elasticity modulus automatically measured by the software application which controls the testing machine and allows the determination of the elastic parameters and the values of the elasticity modulus determined from the load-deformation curves. For comparison purposes, these values are outlined in tables 28-30

5.3. Tensile testing – oblique loading

The whole analysis undertaking related to the determination of the values of elasticity modulus and the values of Poisson ratios is in close connection with the objective of determining the values of elastic constants of the laminae oriented at various angles. The laminate model comprises a means of determining these values based on knowledge of the values of elasticity modulus and Poisson ratios in two known directions. At this moment, whether measured or calculated based on previously presented arguments, the values of these parameters are available and the values of elasticity modulus could be assessed for a lamina loaded at 30o or 45o from the warp direction. Unfortunately, it is not this easy because another important parameter is missing, i.e. the shear modulus G_12. In the relation below, the values E_x=E_([30] ^o), E_1=E_xx, E_2=E_yy,

$u_{12}=u_{xy}$, $u_{21}=u_{yx}$ can be used, with the corresponding values from tables 28-30 in order to determine the value of the G_{12} parameter, against which the relation for E_{θ} ($[45]^\circ$) could be verified.

$$\frac{1}{E_x} = \frac{m^2}{E_1} (m^2 - n^2 \nu_{12}) + \frac{n^2}{E_2} (n^2 - m^2 \nu_{21}) + \frac{m^2 n^2}{G_{12}}$$

As previously mentioned, tests with laminae loading at 30° and respectively 45° have also been carried out, extracting laminae oblique to the warp direction. The most relevant results refer to the observation – in most cases – of material flow, a flow that cannot be explained in reference to the models used for the description of the flow of metallic specimens when subjected to tensile testing.

As in the case of laminae engaged on warp and, respectively, weft direction, all results acquired will be presented for all fabric types (carbon, glass, aramid and mixed fibres), with consideration to the presentation algorithm for materials formed of similar types of fibres (C160 and C240, S25 and S108, CA68 and CA188, the last one, in two variants, 1C2A and 2C1A). As before, this last analysis may lead to significant conclusions on the nature and quality of the matrix-fibre interphase, while also allowing the analysis of the way in which fibre density influences the mechanical properties of the composite (aside from mixtures model), especially the flow of these materials..

5.4 Conclusions

Qualitative differences have been identified in what fracturing of the fabric-reinforced laminae is concerned, in relation to the matrix used; laminae formed with more rigid epoxy matrix (C and HT, which also have low gel time) seem to be elastic in a very narrow deformation domain; this observation refers to engagements to the direction of warp and respectively weft of the fabric used as reinforcement.

- Elasticity modulus determined on very short areas from the beginning of the engagement (in the case of C and HT matrices), before the horizontal planes, have different values for each fabric, which can only be accounted for by considering that there are variations in quality of the fabric-matrix interphase.
- The same argument can also be employed for the existence of different values of elasticity modules for laminae reinforced with the same fabric type but with C or HT matrix after the eventual break of the matrix (the end of the first linear area); if after the initial fracturing of the matrix follows the horizontal plane area (fabric tensioning), the response after this area should be identical (irrespective of the matrix type), which is not the case of the tests carried out.
- Different values of elasticity modules after the horizontal planes signify the fact that fibre tension is different according to the matrix used and, in this case, the statement that HT type resin ensures a more qualitative interface with fabrics is almost generally valid.
- In the case of fabric-reinforced laminae with E matrix, the planes are not visible (from loading-elongation or loading deformation curves), which means that the transfer of loadings between the two phases (matrix and reinforcement) is much more effective than in the case of the other two matrices, the linear behaviour being visible from engagement to breaking.
- Generally speaking, laminae fracture along one fibre or along a fibre beam perpendicular to the engagement direction (from the weft in engagement on warp direction and, respectively, from the warp in case of engagement on weft direction); the fracturing mechanism can be explained by the fact that, at an initial stage, matrix fractures and then, at a subsequent stage, the shearing of the two parts (produced by fracturing) determines the fibres cutting; a study with a high-speed camera may shed more light on this aspect;

- Tensile tests have been carried out both on warp and weft direction in order to underline possible difference in behaviour generated by warp pre-tensioning (absolutely necessary for fabric making), which may have a remnant component after the application of the polymeric layer (of whose nature nothing is known) by the fabric manufacturer with a view to keeping its integrity and favouring the adherence of epoxy resins;
- Differences have been noted (but not significant ones) between elasticity modules of laminae with same reinforcement and matrix engaged to the direction of warp and respectively weft, although all fabrics (except for CA188) are balanced fabrics – same density of warp and weft beams; the analysis of these results does not allow drawing definitive conclusions with regard to the assumption made;
- Comparative analysis has been carried out for the results obtained for fabrics made up of similar fibres (according to technical information provided by manufacturers), assuming that information related to the dependency of values of elastic constants on the fabric structure could be extracted; however, the results obtained do not allow this (also due to the fact that there are errors of specimen processing, with direct effects on the results);
- In the case of oblique engagements, at 30° and 45° from the warp direction, one could note the material flow, rendered, to some extent, with the help of photographs; basically, the flow is more accentuated for 30° engagements.
- The aspects of the loading-deformation (or loading-elongation) curves on flow areas display random variations that can be assigned to the fibres' severance from the matrix or to the matrix flow (its fracturing being hindered by the presence of fibres). One could assert, in this hypothesis, that the presence of fibres lowers the rigidity of the matrix.
- The mathematic model of the values of elastic constants of lamina has been tested in a random reference system, but the results of the application of this model (for determining the values of the elasticity modules of laminae engaged at 45°) does not provide significantly different results from those of a proposed empirical model.
- The results of assessments are relatively far from the values measured for the elasticity modulus of the laminae with C or HT matrix; it is very important to state that, in the case of oblique engagements, there are more sources of error than in the engagement along warp or weft (one of them being generated by the specimens' cutting, others, by their pinning to the vats of the testing machine);
- In the case of CA188 fabric-reinforced laminae, presented as 1C2A or 2C1A, different results are observed for engagement to the warp and respectively weft direction for C and HT matrices, probably signalling that the adherence of these resins to carbon fibres differs from that to aramid fibres; these results are also visible in the case of oblique engagements of laminae; in the case of E matrix, the obtained values are more similar;
- Both C and HT matrix are epoxy resins with relatively short gel time, an aspects that can be used to explain, to a certain extent, the lower quality of interphases, as the pre-polymeric mix does not have enough time to permeate and occupy all interstices between fibres;
- This study cannot be considered more than a starting point in the analysis of the mechanical properties of fabric reinforced laminae; any subsequent study should take into consideration the method of obtaining specimens, so that their cutting is made as precisely as possible, this avoiding lack of uniformity at the edges and especially their deformation;
- Based on these results, a decision was made to form composite plates exclusively with E matrix, given the fact that, for this matrix type, the response to loading is strictly linear (obviously, until breaking), irrespective of the reinforcement type.

Chapter 6. Mechanical Properties of Laminates

6.1. Tensile Tests

The materials formed at this stage are laminated materials (actually, pseudo-laminated) with a reinforcement made of nine fabric layers, all oriented in the same way (all the beams of the warp being parallel). In this case, the laminate model offers the possibility of determining the elastic constants of the formed material, starting from the lamina elastic constants. The issue is even simpler than in the cases studied in literature, where general theory is simplified for different types of laminates (symmetrical, with laminae oriented at different angles, etc.). One might say that the application of the laminate model should offer a value of the elasticity module (for example), one very close to that measured during the tests.

However, several comments must be made here. The first is related to the fact that the material is not exactly laminated and this is found in the way it responds to loads. Thus, through the forming method, the material is practically made up of nine fabric layers, fixed in a single polymeric block (in the case of laminates, the laminae are stuck together by using some adhesives).

In the laminate model, reference is made to the thickness of the lamina – further complicating the proposed approach – because the laying of the soaked fabrics in the matrix brings (naturally) a sliding so that, surely, the beams from the fabrics and picking cannot ever be perfectly superposed. Thus, the thickness of each material is less than the sum of the thicknesses of the laminae from which it is made. Obviously, this fact will generate a deviation from the mathematical laminate model, which might be corrected, probably, by introducing a material compaction coefficient. Table 33 shows fabric thicknesses, laminae thicknesses, and laminated material thicknesses (all pseudo-laminated are made of nine fabric layers each, all with fibre beams from the warp and, respectively, weft, all parallel and with the Epiphen RE4020 – DE4020 epoxy system as matrix) made with 14 of the previously studied fabrics.

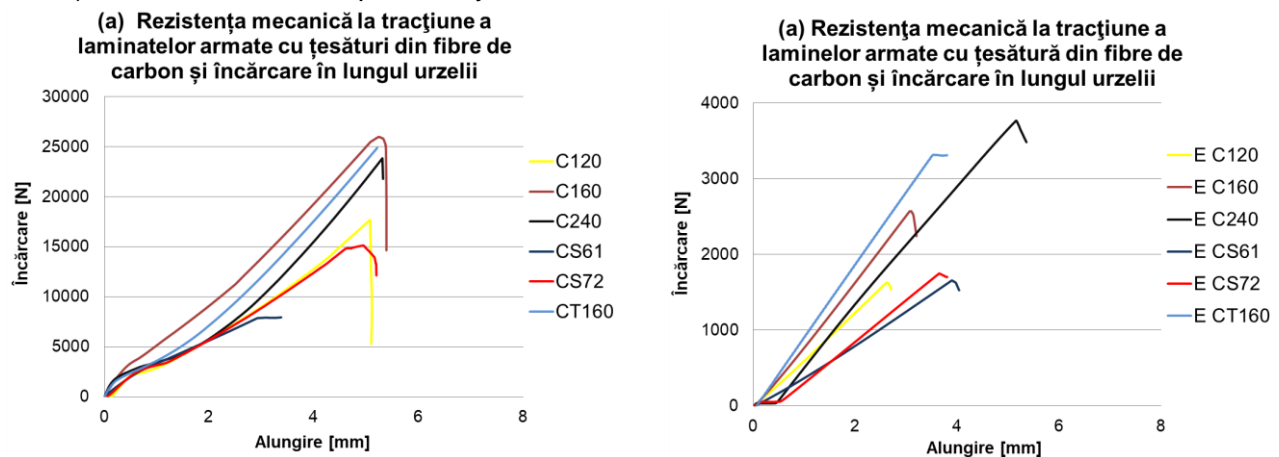


Figure 80. Strength with warp longitudinal engagement for carbon fibre fabrics reinforced laminates and laminae (E matrix)

The CT160 fabric reinforced laminate did not break during testing. It can be noticed that, in general, the maximum loading of the laminates is about nine times higher than that of the corresponding laminae, exceptions being the CT160 and the C240 reinforced materials. In the case of the latter, an explanation could be that, the carbon fibre density being the highest, during the

forming of the laminate, not all fibres were imbedded in the matrix (since the fibre beams of which the fabric is made are thick, the resin did not reach them).

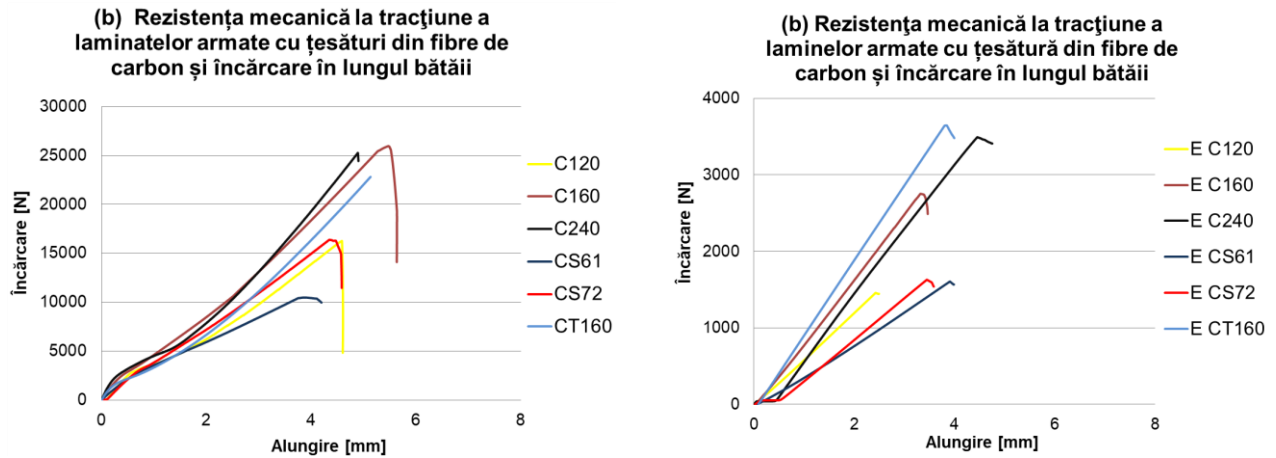


Figure 81. Strength with engagement along weft for carbon fibre fabrics reinforced laminates and laminae (E matrix)

As in the case of warp longitudinal engagement, it can be seen that, in both laminae and laminates, the weakest responses are given by CS61 and CS72 reinforced materials. These fabrics are made of carbon fibre strips obtained by expanding the beam carbon fibres, the low density of the fibres determining the low resistance, the response of these materials being dominated by the matrix behaviour.

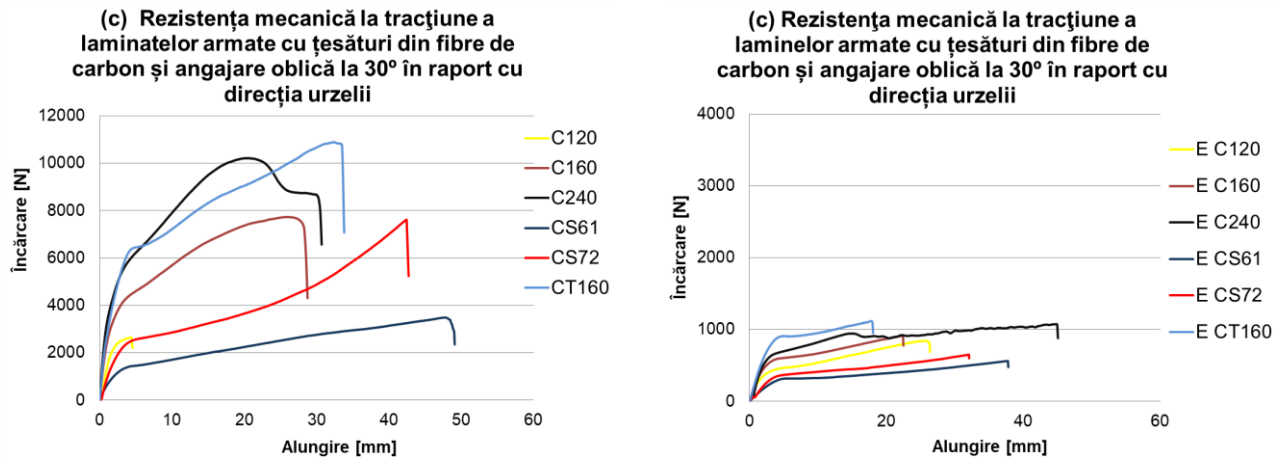


Figure 82. Strength in tensile testing with oblique engagement at 30° in relation to the warp direction for carbon fibre fabrics reinforced laminates and laminae (E matrix)

In the case of the oblique engagement presented above, it can be seen C240, CT160 and C160 reinforced materials give the best responses, their strength being ten times higher than those of the corresponding laminae. Also remarkable is the CS61 fabric responses linearity (regarding both lamina and the laminate).

Interestingly, the C160 and CT160 fabrics reinforced laminae and laminated (i.e., with the same density of the fabric) have different behaviours. This difference can be explained either by the fact that the employed carbon fibres are different, or by the fact that the fibre fabrication process is different – in the case of the C160 fibre, the beam fibres are compact (ellipsoidal in section), whereas, in the case of the CT160 fibre, the beam fibres are expanded, being almost flat, which

would be consistent with the assumption that, in the case of compact beams, the probability of the resin to reach the core of the beam fibres is smaller. As with the two straight engagements (along the warp and weft, it can be seen that, in general, the strength of carbon fibre fabrics reinforced laminates is approximately nine to ten times higher than that of the corresponding lamina, which would correspond – according to the principle of overlapping effects – to a test carried out on a nine-pack laminae, unrelated to each other. Compared to the results presented for the mechanical properties of the laminae, one can state that there is no corresponding laminate for the CS61 and CS72 fabrics, which had the weakest behaviour.

16 pseudo-laminated composite materials were analysed using the wet lay-up method, in glass matrix, from 9 fabric layers, all of them laid so that the beam fibres from the fabric warp are parallel. The EPIPHEN epoxy resin (RE4020-DE4020 system) was chosen as matrix following the analyses carried out on laminae, being less brittle and ensuring a better transfer of the loadings.

Axial tests (straight loadings along the warp and weft, respectively) and tests off-axes (oblique engagements at 30° and 45° in the warp direction) were carried out. The number of tested specimens (small, in terms of statistical analysis requirements) was five for straight engagements, and two for oblique engagements. The obtained results reveal the fact that there are two materials with quasi-isotropic behaviour (those reinforced with CAVS and CVS), while the best response (in terms of elasticity) is that of the materials reinforced with carbon fibre fabrics.

The attempts to evaluate the elastic parameters of the laminates according to the elastic parameters of the laminae did not lead to very clear conclusions. Usually, the maximum loading of a laminate is about nine times greater than that corresponding to a lamina. For the oblique engagements, a significant flow of the laminates is also noted (similar to the flow of the type E matrix laminae).

The laminate failure mechanisms are more complex than the ones corresponding to the laminae, primarily due to the occurrence of interlaminar interactions, generated by the way in which fabrics are naturally placed, after pressing. Another interesting aspect is related to the fact that, in most cases, the laminates thickness is less than the sum of the lamina thicknesses, which should normally lead to maximum tension values for laminates greater than nine times the maximum tension values corresponding to the laminae. Given the material forming way, a failure mechanism due to interlaminar shearing is possible.

6.2. Cycling loadings

Specimens for the analysis of the elastic properties evolution following cycling loadings were provided for all formed laminates. The resistance to fatigue is the highest amplitude and/or numerical level of dynamic cycling loadings that resist to a material trial, under known conventional conditions, without the occurrence of damages/destructions considered unacceptable for the quality of the material in question [82]. The damages that may occur are caused by the physical fatigue created by repeating the same type of strain [83].

Compared to static loadings, variable loadings repeated many times have an adverse effect on the resistance capacity of the material from which the resistance elements are made. This is how unexpected breakings occurred in many machine parts, such as: bent axles, toothed wheels, piston bolts, valve springs, etc., though from the material resistance point of view they have been correctly calculated. The breakings occurred at much lower loading values corresponding to the static load limit states. This premature breaking phenomenon, at strains below the limit ones, is known as material fatigue [84].

Following the tests carried out and analysed for both laminae and laminates, the testing stages and parameters of composite materials laminated at mechanical property were established. The testing stages of the laminates at dynamic tensile were carried out in three important steps, as shown in figure 100:

- a. step 1 is tested until the loading force reaches half the mean force value established at static tensile of the laminates with the same architecture, the corresponding load value for each laminate being set;
- b. step 2 consists of going through 1000 cycles and, again, with the parameters corresponding to the laminate, the speed set for each type of laminate and each engagement angle of laminate specimens; during a loading cycle, the loading varies between a maximum and a minimum value, these representing the extreme values of the loading cycle. The higher the maximum strain in the part, the less the fatigue breaking occurs in a smaller number of cycles. If the strain has low values, the fatigue breaking no longer occurs, no matter how many straining cycles exist in the part.
- c. step 3 – after the 1000 straining cycles, the specimen is statically loaded in order to determine the elastic constants and to highlight the cycling loading effect. .

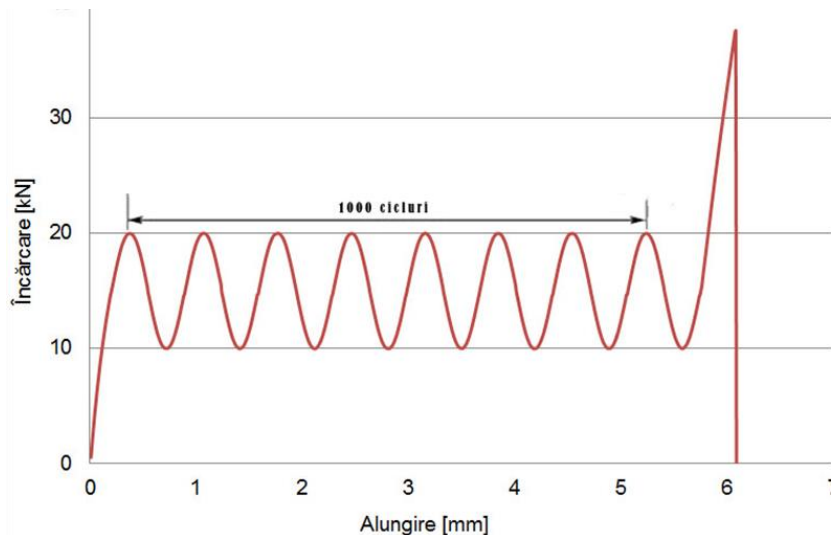
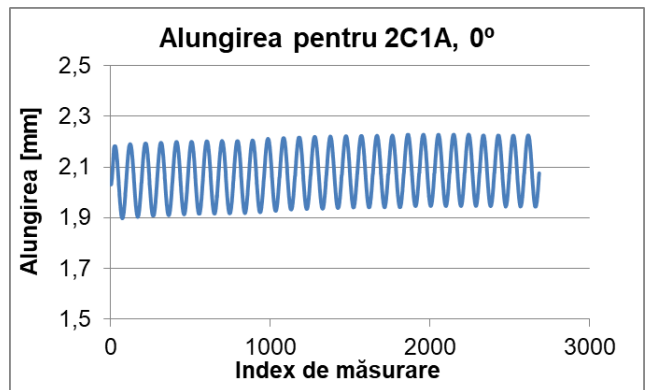
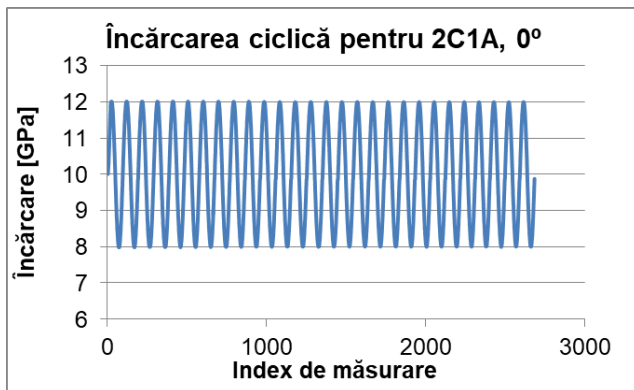


Figure 100. Representation of the three-stage testing process in cycling loadings



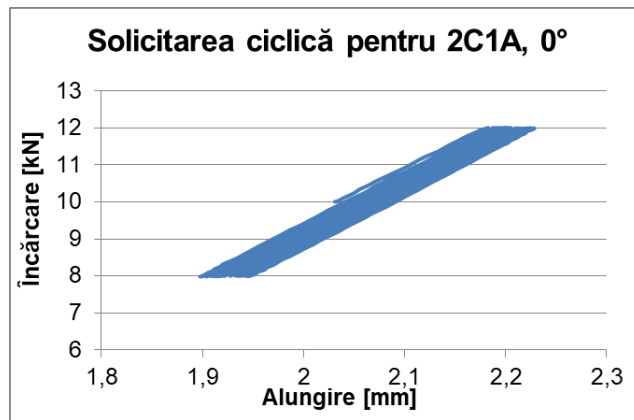
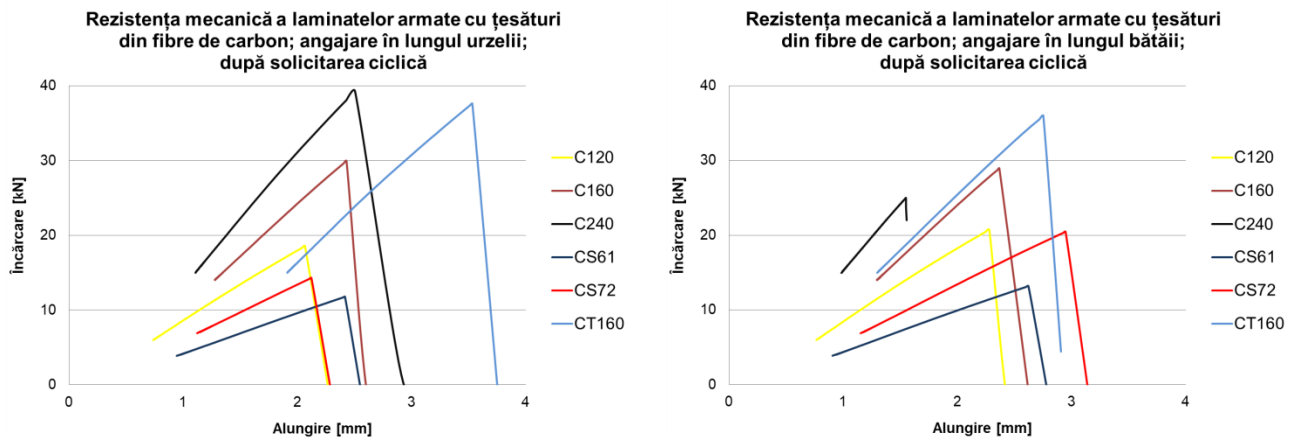


Figure 101. Loading and elongation in cycling loading (up). Loading/elongation curve in cycling loading

As it can be seen from the data presented in **table 34**, the setting data for the cycling loadings was established for each material and for each engagement orientation. Initially – based on the data provided automatically by the software application of the test machine – certain load limits were set and subsequent machine stop conditions, based on the distances between the rafts, were imposed. At the first tests carried out in this way, some of the specimens failed before reaching the proposed loading cycle number. In the end, the reached situation presented in **table 34** shows that, in principle, the maximum straining value per cycle is about 50-60% of the maximum loading recorded in the static tests (for one material and one engagement direction), and the minimum value is about 20-30% of the maximum loading value from the static tests.

The curves shown in **figure 101** can be considered representative (at other scales, of course) for any of the carried out cyclical straining tests. Each cycling loading was performed at the one cycle/ second rhythm, while the data acquisition rate was about 2,5Hz (2,5 times per second), and this time, the tests were carried out with a force cell of 100kN, which also allowed the determination of higher loading values, as it can be seen further. What was noticed was that, following the cycling loading, a remnant elongation of the materials results – observable on the curves below (the starting points of the final loadings). This remnant elongation corresponds to a plastic deformation of the specimens, that could be due to a mechanism analogous to the flowing one, found in the case of laminae testing.



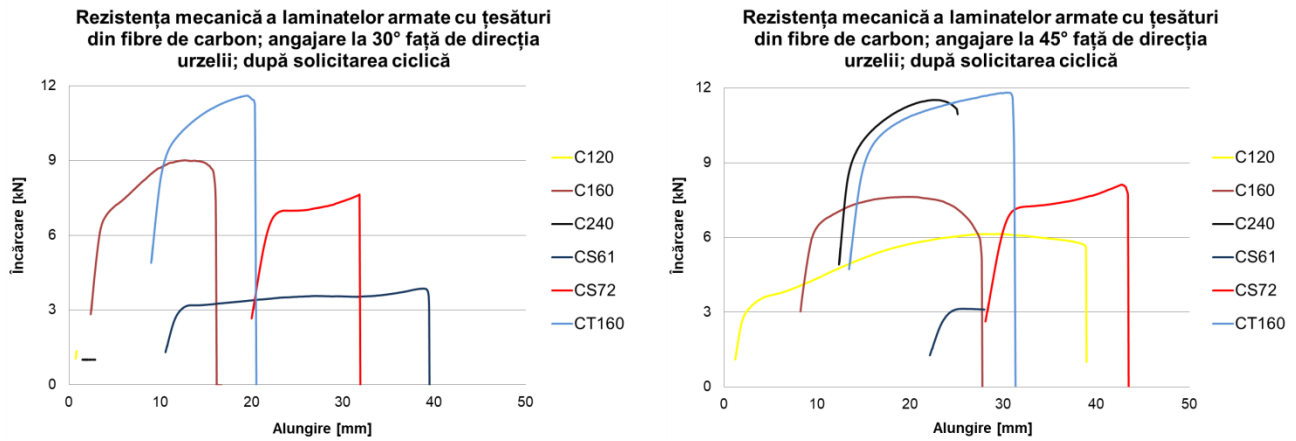


Figure 102. Loading/elongation curves for tested carbon fibres reinforced materials (step 3)

The analysis of the data in **figure 102** indicates the fact that the only material that responds practically the same after the axial and oblique loadings is the CT160 reinforced one (fabric-like material made of uniformly distributed carbon fibre strips – basically, an orthotropic strip fabric); in this case, the observed differences can be interpreted as test errors. In terms of material response, it can also be observed that the C160 reinforced material has the same type of response after the two axial cyclical loadings – including remnant elongation. From the design point of view, it is surprising that the responses of the C240 and C120 reinforced materials are very different. For the C240 case, the two axial engagements show a loading difference of almost 15kN (the remnant elongations being comparable). What is very interesting about these materials is that they practically do not resist cycling oblique loadings at 30° to the warp direction, but they well enough resist cycling loadings at 45° to the warp direction.

From the design point of view, data interpretation could lead to a sequencing formula for the types of fabrics used to reinforce materials, which could be optimized in order to obtain materials more resistant to cycling loadings. However, this approach is restricted by the fact that there is no analysis that targets the mechanical behaviour of laminates made of two laminae (reinforced with the same type of fabric or with different fibres, with the same fibre orientation or with different orientations). Such a study would be an extremely interesting topic for the continuation of the present study even though, from the approach complexity point of view, it is a long-term attempt.

Materials reinforced with balanced fabrics respond differently to cycling loadings, although, normally, there should be no differences between axial engagement responses (along the warp or the weft). In general, the response along the weft is below that along the warp, which means (according to an initial hypothesis) that there is a difference between the properties of the fabric along the warp and weft, respectively. This difference cannot result from the fibre or fibre beam properties, but only from the assumption that the warp is pre-strained (to ensure the weaving process) or from the hypothesis that an important influence on the properties is the way of applying the polymer (never specified by the fabric manufacturer) which, on the one hand, provides fabric stability and, on the other hand, the adhesion of the polymers used as matrix (generally, epoxy resins).

It is noted that the maximum loadings of the laminates have higher values after cycling loadings than before (the test conditions were identical – five specimens for straight engagements and two specimens for oblique engagements). This could be explained by the fact that, following cycling loadings, a massive micro-fracture of the matrix (equivalent to a generalized separation of the fibres in the matrix) takes place so that, during the final loadings, (step 3) the tensions are transmitted

only by the fibres. In other words, following cycling loadings, the fracture mechanisms of the composite that depend on the behaviour of the matrix are excluded, i.e. no longer being a continuum, the matrix fracture can no longer produce the fibre sectioning (through shearing).

The same hypothesis can also be used to explain the absence of interlaminar fracture mechanisms, the tension transfers along the polymer layers between the two reinforcement layers being blocked by micro-cracks, and the tensions transmitted along the fibres produce other micro-cracks.

An interesting response is given by the CAVS reinforced materials, in which straight engagements lead to smaller loadings of the cyclically-loaded material. This is explicable if, once again, it is considered that the reinforcement material is not, in fact, a fabric (as explained above). Under these conditions, felting fibre separations (after repeated loadings) take place, with direct consequences on how loadings are transmitted through the material. In the case of oblique loadings, practically, the response of the material does not change.

An also interesting situation is that of glass fibres fabrics reinforced materials, especially the material reinforced with S163 (also approached when analysing the laminate elastic parameters), whose response is constantly better (from the maximum loading perspective) following cycling loadings, however without recording very high value variations. It can be seen that the materials reinforced with very thin simple fabrics (A61 and S25) practically give the same response (within the limits of experimental errors). In the case of these materials, given the low fibre density, the fracture mechanisms may be the same (i.e. the matrix micro-cracks cause the fractures of some fibres).

Chapter 7. Conclusions and study development proposals

The main purpose of this study was connected to the attempt to describe the elastic parameters of a fabric-reinforced laminate when the lamina elastic parameters are known, following the well-known laminate model. This approach has not been successful because fabric reinforcement implies the inherent occurrence of fabric distribution defects/flaws within the material (deviations from lamina flatness) with immediate consequences on the application of the model which implies the absence of interlaminar shearing.

The present research, based on the analysis of a considerable number of studies carried out in this direction and on the study of some specialized works in the field, aimed at analysing the elastic properties of reinforced laminae with 19 different types of fabrics and matrices made of three different epoxy resin types.

Starting from the main purpose of the study, a documentation, training and testing of materials and interpretation of experimental data program was followed. This program has been agreed upon with the Ph.D. supervisor and guidance committee members, and presents the following originality elements:

- compiling of an up-to-date documentation on the mechanical behaviour of polymer composites;
- research design based on the studied documentation, in order to achieve the objectives set;
- developing an original forming composite material method (laminae and laminates);
- the actual forming of the composite materials (laminae and laminates);
- establishing the material testing program;
- obtaining and analysing a set of experimental data regarding the mechanical, thermal and electrical properties of the formed composites (laminae and laminates);
- carrying out of the mechanical and thermal tests;
- the interpretation of the results following the carried out tests;
- presenting and publishing the results in international conferences and specialized journals.

In order to obtain materials with improved mechanical and thermal properties, the physical and chemical characteristics of the epoxy systems and the properties of the fabrics used in the formation of the new materials were analysed.

Mechanical tensile tests for lamina and laminate specimens were performed to provide data that would allow both the comparative analysis of their elastic properties (when reinforced with the same type of fabric) and the comparative analysis of different materials. As for the laminae, the experimental analysis also targeted their thermal properties, considering two essential parameters in composite material designing: specific heat and linear expansion coefficient in a direction perpendicular to the reinforcement plane.

In the case of fabric reinforced composites, there are many approaches regarding the theoretical determination of the elastic constant values, most of them taking into account certain fabric geometric characteristics: the size and shape of the beam fibres, and the beam fibre waviness factor. In this context, tensile behaviour differences among materials (laminae or laminates) reinforced with fabrics made of the same type of fibre were observed.

Tensile tests for laminae have highlighted the following:

- by the graphical representation of the data obtained, it is observed that materials reinforced with carbon and epoxy resin have the best results;
- the use of fibres in a composite increases the maximum strength, but their distribution in the composite must be taken into consideration;
- the angle formed between the fabric strand direction and the engagement direction affects the mechanical characteristics of the composite. The values of these angles, 0° and 90° , display the best performance against material tearing resistance, the big differences between the experimental result value of the single layer composites (laminae) were obtained for the 0° and 90° angles versus the 30° and 45° ones. This is due to the fibre distribution in the composite;
- following the experimental results, a variation of the Poisson coefficient values based on the epoxy resin, the used fabric, and the angle at which the lamina is cut, is observed for calculating the Poisson coefficient on the specimens from the lamina cuttings; according to the SR EN ISO 527- 4 standard, it is sufficient to use machine exposed data, using a method that considers the machine limitations where there is no possibility of permanent monitoring of transverse dimensions in the loading direction (especially in the case of laminae);
- regarding the lamina elasticity modulus, analysing the behaviour of the longitudinal elasticity module values from the elasticity modulus related graphics, in terms of fabrics used in laminae, the carbon provides good mechanical behaviour, whereas the glass and aramid fibre fabrics reduce the elastic properties. When comparing the elastic modulus values for fabric reinforced materials in terms of the used epoxy systems (E, C, HT2), an approximately equal elastic modulus value can be observed in all three cases, irrespective of the type of used resin. Yet, a scale can be made according to the values of each system, following the lamina mechanical testing. The materials with the best values were those with E-type epoxy matrices, followed by the HT2 type epoxy matrix materials and type C epoxy matrix materials. The forming, cutting and testing conditions were the same for all systems, regardless of the used epoxy system. After analysing the graphics resulting from the lamina tensile tests, it appears that the employed epoxy systems can influence the mechanical properties of the material.

Following the analysis of the relevant data on the state of the tested material (laminae), the materials that show an improvement of the properties compared to the used epoxy systems are observed for the E type resin laminae; therefore, laminates reinforced with nine-layer fabrics and type E epoxy resin were designed and formed.

The mechanical properties of laminated composites reinforced with fabric can be estimated by applying the mechanical characteristics of each lamina used in the lamination, finding a correlation between the mechanical properties of the lamina and the obtained results of the laminates by direct measurement, so that the simulation results are close to the real ones. In this case, a new factor correlation (maximum loading, energy) can be observed between the values corresponding to the laminae and those corresponding to the laminates.

A proposed algorithm for determining the properties of the lamina subjected to tests in order to determine the mechanical properties of the laminate should also take into account the fabric particularities as well as the fact that, in the conditions of placing the fabrics layer over layer, the distances between the median planes of the fabrics are not constant.

An important aspect is the one related to the analysis of the mechanical properties of the laminates after they are subjected to a cycling loading. In this respect, 1000 loading cycles of each material were performed, the loading limit ranging between 40-60% for the maximum loading per cycle and 10-30% for the minimum loading per cycle, the percentages referring to the maximum loading of the laminate under static test conditions. The behaviour of the materials after cycling

loadings is superior (in terms of maximum loading) to that of the materials tested under static conditions, which leads to the conclusion of massive micro-cracking of the matrix following cycling loading.

These tests were not fatigue tests proper, so that no conclusions can be drawn regarding the material resistance to fatigue. In addition, fatigue tests involve cycling loading in which the maximum and minimum loading values are reported at the maximum load corresponding to the elastic behaviour of the material, and not to the maximum load value of the material which, due to the particularities of the fabric reinforcement, displays, after the pseudo-elastic zone, a higher value than that corresponding to the elastic response. Thus, the carried out cyclical tests can be considered as loadings that gradually destroy the matrix and reorganize fibre distribution.

Research on mechanical fatigue of the thermoset polymeric composites is of particular interest due to the material applicability fields. The epoxy resin, as found in the literature, is the most commonly used in forming thermoset matrix composites. As for the fabrics and their influence on the properties of composites, research is constantly developing, offering unlimited possibilities. For an effective fatigue analysis of fabric-reinforced composite materials, it is necessary to increase the thickness of these materials without greatly increasing the fabric specific density because the analysed behaviours of materials reinforced with high density fabrics reveal the fact that the matrix cannot reach the core of the beam fibres. An alternative would be the use of polymers with higher gel times and fluidity, but these endeavours could lead to obtaining some materials with gradient concentration fibre.

For future studies (from the perspective of completing the presented information) are interesting:

- testing the composite materials reinforced with fabrics and epoxy matrix (laminates) on thermal behaviour: specific heat (DSC) and determining the thermal expansion degree (TMA);
- carrying out mechanical tests of laminates in order to determine the flexure properties;
- forming new composite materials reinforced with the same fabrics and other types of epoxy matrix;
- analysing the elastic properties of some special composites made of only two laminae (with the same orientation of the beam fibres or with different orientations) in order to better understand the mechanisms proposed for explaining the different behaviour of the materials formed and tested within this study;
- from the perspective of design, a study on the mechanical behaviour of some special composites made of two layers of reinforcement (two laminae), reinforced with different fabrics, would also be interesting;
- for the dynamic straining tests, a careful analysis of the maximum and minimum loading values per cycle is required so that these fall under the elastic response range of the material (very narrow, as shown for both laminae and laminates);
- finding a software solution to accurately identify the load corresponding to the exit from the first elasticity level, to correctly define the fatigue testing conditions (at this time, the data from the machine following the static and dynamic tests differs due to the fact that two different software applications are used);
- finding a viable solution regarding the video monitoring of the mechanical tests in order to identify the critical moments;
- finding a technical solution for the quality inspection of materials before and after testing, in order to validate different hypotheses regarding failure mechanisms.

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