IOSUD - University „Dunărea de Jos" Galați

Doctoral school for industrial and mechanical engineering

PhD. THESIS

RESUME

RESEARCH REGARDING MECHANICAL CHARACTERISTICS OF COMPOSITE MATERIALS WITH REINFORCED FABRIC AND POLIMERIC MATRIX WITH ELASTICITY GRADIENT

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Content

Introduction

The diversity of composite materials with polymer matrix, developed and marketed in recent decades, have led to unprecedented economic growth, which has also led to a higher consumption of fossil fuels and, implicitly, this has led to an increase in environmental pollution. From the need to develop new materials that would lead to a reduction in the mass of the finished product to putting them into operation, it was just a simple, normal step. The share of composite materials in the field of aeronautics and in the automotive industry has increased with each year. Also, in the case of the shipbuilding industry, the volume of composite materials with polymer matrix used showed a significant increase. These are used for manufacturing small boats (recreational, sporty, military, ROV), but also for medium-sized vessels (military corvettes), for appendages (propellers, hydrodynamic profiles) or marine structures (underwater piping). Also, epoxy resin is prevalent in this area, due to its high-performance properties.

With the introduction of the energy efficiency design index (EEDI) in the shipbuilding industry, shipowners began to increasingly introduce into naval structures and ship hull, polymeric composite materials with properties similar to metal matrix composites in order to comply with international building rules. First introduced in 2013, EEDI has entered the second phase, where the level of pollutants must be reduced by at least 20%, compared to the reference year 2013. Polymer matrix composites can provide a very good alternative for this defined purpose. In fact, recent studies aim to reduce mass without affecting structural stability. Therefore, composite materials with polymer matrix are of particular interest in this field. They can be used to replace watertight doors, floating panels, walls, stairs, railings, drilling piping.

The use of composite materials with polymer matrix reinforced with fabrics, is supervised by independent entities, also called classification societies. They are designated to ensure a high quality of the final product. Thus, any new developed material, must be subjected to a whole series of tests and subsequently approved by such entities. From testing the corrosivity in saline medium, fire resistance, material fatigue in saline medium, to mechanical testing of the material consisting of determining the elastic modulus, resistance to axial stresses, shear resistance and bending resistance, these being key elements for determining the properties of a material.

Forming techniques are diverse in this field of composite materials with thermosetting polymer matrix. Each has advantages and disadvantages. Autoclave forming of composite materials, can lead to a high-performance material, but requires additional costs for equipment as well as highly qualified personnel for operation. Bag formation under the combined action of vacuum-pressure is one of the recently developed techniques for the mass production of materials with complex shapes, but of a smaller size, since for a larger product the forces required for closing the mould are considerably higher. The technique of manual layering by impregnation or wet layup, which can provide high-quality laminates with a minimum of high-performance equipment and quick training of the operator, should not be neglected.

The present work can be considered a sequel to a previous study started at the Research and Development Center for Thermosetting Matrix Composites (CCDCOMT), within the Dunărea de Jos University of Galati. The idea from which the development of this study started, was the identification of a composite material with controllable resistance for car bodies. So, the purpose of the study was to develop a composite material with diluted epoxy matrix and reinforced with fiber fabric and showing a resistance gradient. The present study is an attempt to continue the researches started in the mentioned study, by including in the development an innovative material. Moreover, this study also includes an investigation of the effect of heat treatment (baking/annealing) on the properties of the formed materials. The aforementioned study started from the hypothesis of the possibility of modifying the bending behavior of a material with polymeric matrix reinforced with fabrics by intervening on the properties of the matrix. The results obtained confirmed that for a homogeneous reinforcement (the same type of fabric used for the reinforcement of the composite) the dilution of the matrix (epoxy resin) with 1-methyl 2-pyrrolidinone at different levels leads to different material responses depending on the direction of engagement (in the direction perpendicular to the direction of reinforcement) in the case of three-point bending tests.

Given the complexity of designing the properties of composite materials, the study mentioned, was a rather limited one because one of the easiest ways of designing the

properties of fabric-reinforced composites is that of using fabrics made of different fibers, as it has resulted from other studies carried out at CCDCOMT. Epoxy resins – the most stable and resistant polymeric materials, are the most valuable option for reinforcement, all the more so as their handling is simple and almost completely free of dangers to the health of the body. Their great disadvantage is that they are rigid and the fracture of the matrix (in the case of a composite) leads, most of the time, to the cutting of the reinforcement fibers. Studies conducted at CCDCOMT have indicated that the strength of a lamine also depends on the thickness of the polymer layer (epoxy resin) in which the reinforcement is immersed – it should be noted that the mentioned studies had as subject the laminae reinforced with fabrics (i.e. a reinforced material with a single layer of fabric).

The solvent used – 1-methyl 2-pyrrolidinone (or N-methyl 2-pyrrolidinone – NMP) – is quite well known in the polymer industry and is often used as a plasticizing agent for various thermosetting or thermoplastic polymers. Without exclusivity, at CCDCOMT, the effect of diluting epoxy matrices with various organic solvents was studied, one of which was the NMP itself, on the mechanical properties of epoxy resins. This effect consists in reducing rigidity and, as a consequence, leads to a reduction in the risk of fracture of the fibers in the event of a break in the matrix.

No studies have been conducted on the adhesion of diluted epoxy resin to reinforcements (various types of fabrics) but the reasonable assumption that the presence of NMP (in small quantities) does not change this remarkable property of epoxy resins may be a starting point for a study of the mechanical behavior of fabric-reinforced composite materials and epoxy matrix diluted with NMP. Moreover, here too I assume a concept often used at CCDCOMT, the pseudo-laminates formed should present the same qualities of matrix-fabric interphases. However, as it is known, the quality of the interphases also depends on the treatments applied post-forming and, in this case, it is necessary to know the mechanical behaviors of both the consolidated materials and the materials unconsolidated by special treatments.

Starting from these and motivated by the diversity of applications for composite materials with polymeric matrix in the field of shipbuilding, I initiated the study whose results are presented below without realizing – from the very beginning – its complexity. I suspect, and I think I'm not the only PhD student who has this belief – that, at first, no PhD student can imagine a broad enough picture of what he has to do. The study of bibliographic sources is overwhelming, each new source of information bringing new perspectives on the researched field and leading one to an informational confusion that, apparently, cannot be stopped. The moment of decision is a critical one, it is the moment when the information is extensive enough to allow you to make a selection and decide the direction or directions in which one will continue the bibliographic study. I believe – and I am convinced that I am not the only one – that this is one of the most important moments in the life of a PhD student. That's when I started to trust that I can. That was the moment when education and self-education allowed me to work independently.

The next critical moment was that of the forming of materials, of working directly with expensive materials. Although, in appearance, the simple process proves complicated because it raises problems that sometimes need to be solved in real time. I would not have thought that placing fabrics layer by layer in a mold can be so complex and time-consuming. Once again, the experience and achievements of CCDCOMT proved to be essential.

In a pattern now common at CCDCOMT, I started the study so as to achieve the complete characterization of the materials formed and this means – electromagnetic properties, thermal properties, mechanical properties. I even conducted all the electrical tests, on all the materials formed and we understood that the electromagnetic properties as well as the thermal ones depend more on the reinforcement than on the matrix, and, as the reinforcements are identical the measured differences are very small and, unfortunately, can be attributed to measurement errors. Moreover, performing mechanical tests revealed to me another complexity (of behaviors) that had to be explained, so, the following is an analysis of the mechanical responses of the materials formed (tensile tests and three-point bending tests) along with the attempts to explain these behaviors starting from the properties of the epoxy resins used and from the properties of the fabrics used as reinforcing elements.

1. Current directions and orientations in the design of composite materials reinforced with fabrics.

1.1 General considerations regarding composite materials

Technological development has always depended on progress in the field of component materials of any kind. Even the most advanced design of an installation, machine or craft, cannot be very efficient if the appropriate materials are not available to withstand the conditions and efforts of use. Whatever the field of interest, the final limitation of technology advancement depends on the materials. Composite materials are nothing more than a huge step in the ever-constant effort to optimize materials [1].

1.1.1 Applications of composite materials in the field of shipbuilding

The applicability of composite materials in the naval field is diverse, from pleasure boats (boats, water bikes, sailboats, yachts and super-yachts), boats for water sports (motorized or sailing competitions), commercial vessels (passenger, bulk carriers, oil tankers, chemical tankers), technical ships (rescue, PSV), military ships (patrol, corvettes, ROV), marine structures (drilling rigs or subassemblies), up to signal beacons or carcasses of military torpedoes.

The need to develop new reinforced composite materials is also demonstrated by the RAMSSES project (Realization and Demonstration of Advanced Material Solutions for Sustainable and Efficient Ships), financed with European funds and which aims to introduce fiber-reinforced polymers in shipbuilding of passenger, cruising or LNG ships, in order to achieve a total weight reduction of up to 40%, which implies lower operating costs of ships, low fuel consumption and most importantly, reduced CO_2 emissions. The applicability of these lightweight materials is found in the walls of the ship's superstructure, which, in the case of modern cruise ships, can reach up to several thousand m^2. Indeed, the current legislation does not allow the widespread use of reinforced composite materials in the naval field, but this is also an objective of the RAMSSES project, and a first step has already been taken by the DAMEN shipyard in Gorinchem, Netherlands, by construction of a custom hull for offshore vessels, under the supervision of Bureau Veritas classification society. The entire structure of the hull of the ship consists of thick laminates, up to 6 m high consisting of reinforced composite materials and using an innovative resin, developed specifically for this purpose. It should be noted that the project is still in the implementation phase [5]–[7].

The Americas Cup international competition has propelled the research and development of composite materials in this field since 1988, by imposing construction restrictions [8], such as the maximum thickness and weight of the hull shell and the maximum temperature or pressure relative to gel time. Thus, in the same year, Bruce Farr designed a monobody, using carbon fiber and Kevlar / Nomex in a sandwich structure [12]. An indisputable proof of the progress in the field of competition boats, we find it even in this regatta, which, after almost 30 years, still offers current technological solutions, both in the field of materials engineering, or hydrodynamics, and in related fields. Thus, for the 36th edition of the Americas Cup competition, which took place in 2021 [13], an AC75 slider was designed, with a maximum length of 22.86 m, a maximum width of 4.87 m and a reduced weight. The last parameter is crucial, taking into account that this boat can reach speeds of over 90 km/h in sliding mode. The movement of the ship is ensured by the active propulsion in the form of a double sail which thus forms a wing. The body of the slider as well as the appendages were made of carbon fiber and epoxy resins.

In [14], a semi-glider catamaran is presented, intended for passenger transport, with a capacity of up to 52 passengers, including crew. The hull was made of fiberglass reinforced polyester (PAFS). This boat was built by Spatyard srl, from Galati.

Reinforced composite materials are also used for ship propulsion. Ship or submarine propellers are usually made of nickel-aluminum-bronze alloy (NAB), due to their high corrosion resistance performance as well as high yield strength. The use of reinforced composite materials [15], [17]–[21], in the manufacture of propellers would lead to a number of

improvements. For example, processing NAB alloy, even through CNC machines, is a very complex process, but especially expensive. NAB propellers are prone to cracks due to material fatigue and have a low degree of acoustic damping, which can induce noise problems due to vibrations on board the ship. The advantages of reinforced composite propellers are related to: low manufacturing costs for series propellers, reduced weight of up to 5 times [22], compared to NAB, also the shaft diameter will be smaller due to a low moment of inertia of propeller, good vibration damping properties and implicitly, high comfort on board the ship for seafarers, corrosion resistance, cavitation initiation at higher speeds, by using thicker and more flexible blades. Propellers made of composite materials reinforced with carbon fibers can be both fixed pitch and adjustable pitch.

Reinforced composite materials are used on all types of ships, whether we are talking about pleasure craft, motorized or sailing competitions, passenger transport, fishing, commercial use, technical vessels, lifeboats, research vessels or ROVs, even offshore marine structures, including military vessels.

1.1.2 Applications of composite materials in the aeronautical industry

Composite structural elements [23], [24] are now used in a variety of components for aerospace or aeronautical structures. Military aircraft designers were among the first to realize the extraordinary potential of composites with high specific strength and high specific rigidity, because the performance and maneuverability of these vehicles depend so much on mass. The construction of composite materials also leads to smooth surfaces (without rivets or sharp transitions of surfaces as in metal construction), which reduce the resistance to advance. Since boron and graphite fibers were first developed in the early 1960s, applications of advanced composites in military aircraft have accelerated rapidly.

Carbon fiber composite structural elements [23], such as horizontal and vertical stabilizers, dampers, wing cover and various control surfaces have been used in fighter jets for many years. More recently, carbon fiber composite fuselage and wing structures have been incorporated into fighter jets, such as the F-35 Lightning II Joint Strike Fighter, and the entire B2 bomber structure.

Applications of composite materials in commercial aircraft have steadily increased as material costs have decreased, as design and manufacturing technology evolves and as experience with composite materials in aircraft continues to grow [25]. A 1994 NASA report indicated excellent operational performance of composite components in commercial aircraft over a 15-year evaluation period, and such results encouraged increased use of composites in aircraft structures, including small business aircraft as well as large commercial and transport aircraft. Initial use in these aircraft has been limited to smaller, lightly loaded secondary structures, but lately, composites are being used more and more in large, heavily loaded primary structures, such as wings and fuselage. For example, the Cirrus SR-22 has a single engine, is an aircraft that can carry four passengers and has the fuselage as well as the wings made of composite materials. Being an excellent example of innovative design made possible by composites, their use for the construction of this aircraft led to a significant reduction in weight so it was accepted to add an additional weight of an aircraft parachute system for safe lowering of the entire aircraft in case loss of engine power [23].

The level of sophistication achieved in the construction of aircraft through composite materials is strikingly illustrated by the rotor blade of a helicopter in Gibson's book [23]. Some elements made of composite materials are: outer shell of carbon, front reinforcements of unidirectional fiberglass fibers, lateral reinforcements of hybrid laminates with unidirectional fiberglass / carbon fibers, coating of fiberglass fabrics, honeycomb Kevlar paper.

Due to the extraordinary cost per unit weight for placing an object in space, the value of the weight saved is even higher for aerospace ships. Thus, composites are extremely attractive for applications in this industry. NASA's space shuttle has a number of composite parts, including cargo warehouse doors made of graphite / epoxy resin as well as experimental engine housings also made of graphite / epoxy resin. For large aerospace structures, such as the International Space Station, or others, the key properties of structural materials are high stiffness / density ratio, low coefficient of thermal expansion, and good vibration damping

characteristics. In all three areas, composites offer significant advantages over conventional metallic materials [23].

SpaceShipOne from Scaled Composite, the first privately manned aerospace spacecraft to make the suborbital flight, is built primarily of composite materials and promises to lead the way to commercially manned space travel. In other components of the spacecraft, such as precision reflectors, special composite structures, such as carbon fiber reinforced isogriles, are used for their superior dimensional stability characteristics. As is well known, some advanced fibers, such as carbon, have extremely low (and, in some cases, negative) thermal expansion coefficients, which makes it possible to design composite structures with excellent dimensional stability [23].

1.1.3   Applications of composite materials in the field of civil engineering

Composite materials have been, are and will be widely used in the field of motorized vehicles. The structural mass is very important in vehicles, and the use of composite components for automobiles continues to increase. Fiberglass-reinforced polymers continue to dominate the automotive composites market, but advanced composites with carbon fiber reinforcement are receiving increased attention as the cost of carbon fibers continues to decline [23], [27].

In the case of freight trucks, the reduced weight of the composite components translates into increased payloads, which can have a significant economic impact. For example, the composite concrete mixer drum (for vehicles used to transport fresh concrete), made of advanced composite materials reinforced with carbon fiber [28], weighs 900 kg less than the conventional steel drum it has replaced. According to the manufacturer, this means that an additional $1/3$ m^3 of concrete can be transported per run, which translates into a significant productivity gain [23].

Weight savings on certain components, such as composite springs, can exceed 70% compared to steel springs (composite springs have shown to be more resistant to fatigue). In some research projects, an engine block was made of graphite-reinforced thermoplastic composites, but the ultimate goal is a ceramic composite engine that would not require water cooling. Composites are also used in the fuel supply system, interior dashboard finishes or upholstery, as well as in the braking system, where brake discs made of carbon fiber-reinforced composite materials have proven to be extremely effective in due to the coefficient of friction which increases with temperature. To date, the applications of composites in motor vehicles have been mainly in secondary structural elements and body parts, and the full potential of primary composite structures remains to be explored. With the growing interest in electric vehicles comes the need for composite structures to reduce the structural weight of the vehicle to compensate for the heavy batteries that are needed to move it [23].

Wind turbines are receiving more attention as alternative energy sources [31], representing an attractive method from the point of view of environmental protection, and their blades are usually made of composites due to the high resistance-density ratio, high rigidity-density ratio, damping excellent vibration and fatigue resistance.

Chassis of large mass vehicles, such as passenger transport at the airport, are often made of composite materials. Reinforced composite materials are also used in high-speed trains.

1.2   Classifications of composite materials

In the literature, researchers classify [2], [4], [23], [32], [33] composite materials according to various criteria. The more we explore the specialized bibliography, the more we find various definitions or classifications of composite materials. In fact, any classification system involves the choice of a criterion: technical (which can be physical, chemical, thermal properties), economic (price, final purpose), depending on use or even alphabetical. Thus, it is very difficult to classify composite materials according to their properties. The range of these properties is so wide that it cannot be cataloged in a homogeneous way. Also, no alphabetical cataloging is very practical. Thus, the most operative way of classification is according to price, quality or molecular structure, as presented by the author in [34].

Thermosetting polymers usually contain [35] two or more components: hardener and catalyst. The polymerization or solidification begins with the mixing of the components at room temperature or at a higher temperature. The resulting chemical reaction produces a rigid crosslinked network. Some advantages and disadvantages of using a thermosetting epoxy resin in the formation of a material are presented in [32], [35].

Reinforced composite materials form discontinuous phases. They can be formed with various sizes and shapes of reinforcement, resulting in a composite with a different structural shape.

Laminated composites are made of several thin sheets, stacked and bonded together. Thus, different laminated composites can be formed (plastic reinforced with fabrics, bimetals, laminated / laminated wood). In laminated composites reinforced with fabrics, each sheet / layer is a reinforced plastic, usually with unidirectional fibers [38]. The reinforcement can be unidirectional, bidirectional or multidirectional. In some cases, short fiber-reinforced sheets are used, in which case the fibers are oriented disorderly [33], [39].

1.2.1 Classification of fiber-reinforced composites

This group of composite materials consists of a complementary phase in the form of hard, rigid and fragile fibers evenly distributed in a more ductile matrix. The role of the matrix is to transmit the strength of the fibers, ensuring the ductility and toughness of the composite, while the fibers support a large part of the applied force. The fiber is a fine filament of material with a diameter of about 10 microns and a length / diameter ratio between 100 and infinity (continuous fibers). Materials brought to micron diameters have a very high strength, much higher than the strength of the same raw material. The phenomenon is due to the fact that in the raw material the sliding planes are wide, while in the fibers of the same material, these planes are reduced to the minimum surface corresponding to the cross section of the fiber. Also, many manufacturing processes involve twisting and stretching the fibers, during this time some important microstructural orientations occur. The immediate effect involves the improvement of mechanical properties, such as strength and modulus of elasticity of the fibrous reinforcement. Other characteristics of fibers are: stiffness, density (low), hardness (high), flexibility and coefficient (form factor) [33], [40]–[46].

In the design of a composite material [40], the aim is to obtain a high strength and rigidity relative to the unit of mass, parameters expressed by two characteristics: specific strength and specific modulus. Fiber length influences the mechanical properties of fiber composites.

Simple, non-matrix reinforcement is not usable as a structural part [33]. The mechanical properties of the matrix are inferior to the fibers, however, it influences some mechanical properties of the composite, such as: transverse strength, shear strength, compression strength, fatigue characteristics [47], interlaminar shear strength and coefficient of thermal expansion . The matrix of the composite material has some critical functionalities. Additive powders lead not only to the improvement of electrical but also thermal properties, including thermal and mechanical resistance [2], [32].

1.3 Types of fibers

A fiber-reinforced composite material is the most important technological group, in which the dispersed phase is in the form of a fiber. In these systems, the fibers take over the main load and the function of the matrix is mainly limited to the load distribution and its transfer to the fibers, as well as their maintenance in the originally designed distribution. In general, design objectives for a fiber-reinforced composite material include stiffness and / or high strength, both relative to the mass of the material and which are named, specific modulus (ratio of modulus of elasticity to density) and specific strength (ratio of strength to traction and density) [35], [48]–[52].

The matrix in a composite is the continuous phase that ensures a uniform distribution of the load to the curing constituents. The matrix, in addition to protecting the hardening constituents, protects the composite surface against abrasion, mechanical damage and environmental corrosion. The uniform distribution of the load is due to the existence of an adequate adhesion between the different constituents, which can be minimum or maximum (as

in most cases), depending on the application. The matrix itself should provide sufficient breaking strength or ductility and optimum hot / wet performance. Because the final thermomechanical characteristics of the composite are governed mainly by those of the matrix, the thermal resistance and thermal properties of the latter are also very important. In addition, a high-performance resin matrix is expected to have a modulus of at least 3GPa for strength and a shear modulus high enough to prevent buckling of fiber-reinforced components, especially when under compression, with although, it is known, the matrix plays a minor role in the mechanical response of a composite. The matrix has a major influence on interlaminar shear (especially important for structures under bending loads) and on planar shear properties (important for structures under torsional loads) [35], [55].

1.4   Epoxy resins

One of the most important classes of resins used are epoxy resins, developed over 60 years ago. They are more expensive than polyester resins, but have superior mechanical properties and good resistance to alkaline conditions. Epoxy resins are by far the most widely used polymer matrix for carbon fibers and currently make up over 90% of the matrix resin material used in advanced composites [48], [101], [102].

For a long time, epoxy resins have been of major interest from a technical and scientific point of view. They are a family of thermosetting materials that are widely used as adhesives, coatings and polymer matrix composites due to their low viscosity, good insulating properties of the finished material even at high temperatures, and good thermal and chemical resistance [103]–[105].

Due to their high adhesion forces and low costs, epoxy resins have been the primary matrix of adhesives and various curing agents such as nitrogen (amides and polyamides), oxygen (anhydrides) and sulfur (mercaptans) [104], [106].

Curing agents, such as aromatic and aliphatic amines, which are most commonly used for epoxy curing, are known to be toxic, and amines are sometimes used to reduce toxicity. Recently, the feasibility of using amino acids as an ecological means of crosslinking epoxy resins has been demonstrated. Therefore, the development of environmentally friendly epoxy systems is of major importance for the design of biocompatible materials in various applications [104], [107].

Fundamental understanding of synergy in tribological performance [2], among various functionalities, is essential for successful application in this field. The tribological behavior of epoxy matrices can be significantly improved by the addition of materials such as lysine and isoleucine. In addition to the type of material, its shape and size influence the tribological properties [104].

Within the Research-Development Center for Thermoset Matrix Composites (CCDCOMT), numerous studies have been developed on the formation of composite materials as well as their addition with various agents for modifying the structure of the matrix.

In one such study from CCDCOMT, the addition of lysine and isoleucine was used in the formation of epoxy composites. Because lysine is not miscible with epoxy resin, in this study, it was dispersed in methanol. One conclusion of the study is that lysine in a concentration of 1% or 3% and used in epoxy resin type C, did not influence the coefficient of friction, it remained constant at an applied force of 10N. The same observation applies to type C epoxy resin with 5% isoleucine for applied forces of 10N, 15N or 20N. Another material tested was epoxy resin type H (Ht) with 3% lysine, a material that had an unsatisfactory coefficient of friction, at an applied force of 20N. Finally, the authors concluded that it is mandatory to perform mechanical tests for a better understanding of the effect of these amino acids in the epoxy matrix [104].

Epoxy resins have unique properties, adhesion to most surfaces, good mechanical [108] and chemical resistance. Epoxy resins are used for surface coating, in the electrical and electronics industry, in the aerospace industry as well as as a matrix for fiber-reinforced composites, additive composites or hybrid composites. Due to their chemical characteristics, thermosetting epoxy resins are used for engineering applications. In addition to the advantages, they present, low moisture absorption, mechanical and thermal resistance, resins have an undesirable property. Due to the structure of the crystal lattice, they are fragile, thus having a low resistance to fracture. One of the problems addressed by researchers in this class of

materials was the increase in toughness. One solution to this problem is to incorporate (add) the epoxy matrix with different additives or even by combining two different classes of polymers, thermosetting with thermoplastics. With the use of a category of additives, characteristics corresponding to the additive are induced in the material, mechanical strength decreases in the case of soft particle additives, or malleability in the case of rigid particle additives. Finding an optimal recipe for obtaining a material with desired characteristics is a challenge for engineering. The answer given by the formed material is directly related to the phase interface [78], [105], [109].

Epoxy resins have excellent mechanical properties, low shrinkage and form adequate bonds with fibers. They have, for a short period, a low viscosity during polymerization, which allows the use of manual casting techniques. Epoxy systems, which are heat treated at temperatures of 120 ° C and 180 ° C, have higher working temperatures of 100 ° C and 130-150 ° C, respectively [51]. Also, epoxy systems C, Epiphen RE4020 / DE4020, H and L are used to form materials by impregnation and layering, they polymerize at room temperature, have very good resistance to moisture and ultraviolet radiation, excellent adhesion, good thermal resistance after polymerization and a transparent and clear appearance[110]–[116].

1.5   Fabric-reinforced composite materials

In order to understand the tensile behavior of the individual fabric layers for composite reinforcement, there must be comparative information for the same terms.

Maier from the BMW group's for research and innovation center [73], together with the co-authors, studied some composite materials reinforced with fiberglass and carbon fiber fabric, also called hybrid materials. The aim of the research was to identify hybrid effects of the bending mechanical properties of hybrid profiles, compared to profiles from a single material. The result was that the formed materials showed a tensile deformation which depends largely on the type of fiber on the outside of the laminate, and which generally increases with the increase of the glass fiber fraction.

In [117], the authors conducted a study on the construction of a hull made of reinforced composite materials as an alternative to the classic construction of recreational craft reinforced with fiberglass. Thus, 6 sandwich structures and 3 hybrid structures were studied. Concluding the results of the study, it can be said that fiberglass-reinforced composite structures used in recreational craft can be improved by alternative arrangements of sandwich structures, and these improvements can be assessed in early stages of design in a systematic manner using adequate numerical tools [118]–[121].

In [122], the authors conducted a study to determine how the type of material architecture influenced the tensile strength of carbon fiber. Thus, using epoxy resin RE 4020 / DE 4020 as well as simple, bidirectional and twill carbon fabric, laminates were formed. The hand lay-up method was used, and the polymerization took place in a vacuum bag at a constant pressure of 0.9 bar and subsequently treated in an oven at 80°C for 8 hours. Using the water jet cutting machine, 5 specimens were extracted from each laminate. The maximum tensile strength obtained was 1050MPa for the unidirectional fabric laminate, 570MPa for the single fabric laminate and 525MPa for the twill fabric laminate. Microscopic verification of the microstructure of the material indicated a very good impregnation of the carbon fiber and a good compatibility between the fibers and the matrix.

In [123], the authors conducted a study on the mechanical properties at compression and tensile, of hybrid reinforced laminates formed with fiberglass and carbon fiber fabric and using epoxy resin, by the wet lay-up method. The final conclusion is that compared to fiberglass-reinforced composites, hybrid composites have proven excellent mechanical properties. Therefore, this hybrid fiber reinforced composite can be used as a replacement for glass fiber reinforced material only where good mechanical properties are needed. Composites reinforced with a mixture of fiberglass and carbon fiber have increased the strength and stability of the material.

In [124], the authors conducted a study to determine the tensile and bending behavior of epoxy composites, which were reinforced with oxidized polyacrylonitrile fibers and high-performance fibers (carbon, glass and Kevlar). In hybrid composites, the hybridization parameters show positive or negative hybrid effects on its mechanical properties. Thus, epoxy

resin and hand lay-up method were used to make the materials, the fiberglass fabric being simple, the fabric with oxidized polyacrylonitrile fibers was unidirectional, and the carbon fiber and aramid fabric was of the twill type. In this way, the laminates formed were both in hybrid and simple control form. Thus, four simple laminates were formed, with four layers of carbon fiber, with four layers of aramid fiber, with four layers of fiberglass and finally with four layers of oxidized polyacrylonitrile fiber. The first hybrid laminates had the following configuration, namely one layer of oxidized polyacrylonitrile fiber, three layers of carbon fiber. The same configuration is valid for the hybrid laminate formed with fiberglass and aramid fiber. The next laminates had two layers of oxidized polyacrylonitrile fiber, two layers of carbon fiber. Similarly, the other hybrid laminates had the same configuration. The last hybrid laminates formed, consisted of three layers of oxidized polyacrylonitrile fiber, one layer of carbon fiber, the same configuration being valid for the hybrid laminate formed with glass fiber and aramid fiber. One of the conclusions of the study highlights the fact that carbon, glass and aramid fibers have fragile breaking surfaces in hybrid composites, while oxidized polyacrylonitrile fibers have ductile breaking surfaces. The conclusions of the study emphasize the importance of hybridizing the reinforcement to obtain improved materials and confirm the hypothesis of using various types of reinforcement from the design stage.

In laminated composites, the order of arrangement of the layers can affect the properties of the material. In Chawla's work we find such an example in which a composite tested at tensile stress at ambient temperature, shows creep for 2 types of laminated materials. At a certain stress, the laminate having a carbon fiber oriented at ±45°, shows a higher creep deformation than the laminate formed with layers 0°/90°/±45°. The reason for this was that, in the ±45° stratification, the epoxy matrix contributed to the creep deformation from axial stresses, shear at angles of ±45°, as well as the fabric stratification in a way that sudden shear was favored. The layers oriented at 0° and 90°, do not contribute to the rotation induced by shear due to the absence of tension-shear coupling in this special orthotropic laminate. Thus, the addition of layers oriented at 0° and 90° reduces the shear deformation of the matrix. Likewise, to increase the creep resistance, it is preferable to layer 0°/90°/±45° instead of layering at angles of ±45°. Also, a different stratification (0°/±30°/±60°/90°, 0°/60°/-60°, 0°/45°/90°/-45°) can provide quasi-isotropic properties in plane [1]. It should be noted that a quasi-isotropic laminate, in the xy plane, is isotropic. In such a laminate, the stiffness of the tensile matrix behaves like that of an isotropic material [A]. The other two stiffness matrices [B] and [D] may not behave like isotropic materials [33]. However, it must be taken into account, that, although manual layering is a sufficiently qualitative process, it is by its nature long-lasting and involves a substantial amount of labor [56].

Laminated composites formed with fibers and having different properties are also called pseudo-laminates or hybrids [84], [124], [125] and may have an additional degree of flexibility [126]. They can also be used to reduce the final cost of the material by carefully designing it [127], [128] as well as choosing the right fibers to achieve greater strength in areas with concentrated stress [129]–[132]. Thus, in hybrid composites, the final cost can be optimized by reducing the carbon fiber content [76], while maintaining the best performance obtained by optimizing the layering and orientation of the fibers [1].

In the literature, various studies on the behavior of composite materials reinforced with carbon fiber fabric are presented. Zhou and co-authors [133] performed a series of tensile tests for 3 types of composites formed with carbon fiber and epoxy resin to investigate mechanical behavior and structure-property relationships. The same Zhou also studied the bending of a material formed with carbon fiber and epoxy resin, thus observing delamination of the fabric layers [134].

Within the Research-Development Center for Thermoset Matrix Composites (CCDCOMT), numerous studies have been developed on the formation of composite materials and their reinforcement with various types of fabrics or agent modification of the matrix structure. Next, the latest research from CCDCOMT on the development of new composite materials reinforced with fabrics will be presented.

Thus, in [135] the authors conducted a research by comparative analysis of 8 types of fabrics for the formation of sheets and laminates. The fabric used in the above work was 100% carbon fiber fabric, the difference being only density, and the epoxy system used was epiphen RE4020-DE4020. The 8 fabrics used are CF120, CF160, CF240, CS61, ST72, TF60, TF76,

T160, where the numbers represent the specific density of the material in g/m2, these being purchased from various manufacturers. At the same time, it should be noted that the above work is an empirical one and was developed to better understand the tensile behavior of fabric-reinforced sheets and possibly to identify how mechanical behavior influences the matrix for cases when the sheets are layered in a polymer matrix reinforced material. One of the conclusions of the study shows that laminates have a lower modulus of elasticity than laminates. This may be due to the effect of agglomeration of different layers (nesting) due to the technique of forming the material, or the laminates have air bubbles inside the matrix, and this leads to the hypothesis that the pre-polymer mixture does not reach inside the fibers. Also, in the case of laminates, the specimens loaded in the axial direction yielded perpendicular to it. At the same time, for laminates there are significant differences in the appearance of the tear zones for ordinary fabrics and materials reinforced with special fabrics.

In [136], the author shows that the matrix, the reinforcement and the additive are, in most cases, materials of different natures, so that there will always be a spatial area of passage from one material to another (from a set of properties to another) called interface. The macroscopic response of the composite material depends essentially on how the interface allows the transfer of stresses (mechanical, thermal, electromagnetic) between the two components. It is well known that the mechanical properties of polymeric composites depend not only on the mechanical properties of the components, but to a large extent, on the nature of the additive surfaces and the load transfer mechanism from the matrix to the additive [137]. The quality of the interface determines the structural integrity, the response to the action of the environment and the physical and mechanical properties of the composite material.

In [138], the authors formed a series of materials reinforced with carbon fiber, mixed fiber and aramid fiber, using epoxy resins C, epiphen RE 4020, H, and L to test tensile and bending. Also, a solvent with mechanical response modifying properties was added to the pre-polymer mixture to the above-mentioned tests. The use of the solvent N-methyl-2-pyrrolidinone (NMP), can introduce in the matrix some specific properties, such as shape memory [139] or modification of the mechanical response to traction and bending, in the sense that it has different values of modulus of elasticity to traction/bending, compared to epoxy systems in general. One of the conclusions of the study was that the presence of NMP allowed control over the strength of the composite. Also, using the wet lay-up formation method, a concentration gradient of NMP was obtained and, as a consequence, a gradual strength of the material, determined by the strength gradient of the matrix. Similarly, the modulus of elasticity in bending does not depend on the direction of force application, however, the bending behavior of the material depends to a large extent, on the direction of force application. As a rule, the tensile modulus depends on the nature of the matrix, however, in the study, the modulus of elasticity seems to be independent of the nature of the matrix and, rather, depends very much on the type of reinforcement.

In [140], the authors studied the low-velocity impact response of epoxy composites reinforced with simple or hybrid fabrics (carbon fibers, aramid and glass as well as copper wires) and pre-polymer blends in the epoxy matrix (powder of aramid, potato starch, barium ferrite and carbon black). The aim of this study was to characterize and evaluate the effects of fiber orientation at different angles and pre-polymer mixtures in the epoxy matrix on the impact response. All tests were performed at constant impact energy, i.e. 90,629J. The results indicated that the orientation of the fibers at different angles has a positive effect on the impact response, mainly in the case of composites reinforced with aramid fabrics. Regarding the influence of the modification of the pre-polymer mixture with different powdering agents resulted in an improved response to impact of composites reinforced with hybrid reinforcement compared to composites reinforced with the same type of fabric.

1.6 Breaking of laminated composites

When studying how a laminate breaks [136], the deformation and tensile stress of the laminate are actually analyzed by increasing the stress until the rupture occurs. The purpose of this analysis is to determine the strength of the laminate, represented by the maximum load at the time of breaking the material. It should be noted that the breaking of the laminate depends on the breaking of the constituent sheets, the breaking of all the sheets does not happen

simultaneously, and the breaking of a single layer does not necessarily lead to the breaking of all layers [141], [142]. As it is known a laminate consists of sheets, so the structural strength of the laminate depends on the individual breaking of the constituent sheets. Sometimes the laminae detach from each other due to interlaminar shear even if the individual laminae remain intact. The sheets are laminated at different angular orientations, and the constituent sheets can be made of different materials, as a result the strength and rigidity characteristics of different sheets are different [143]. Each sheet responds to the applied loads according to its own strength/rigidity characteristics so that each sheet can be expected to have its own breaking loads, and the breaking of all the sheets in the laminate will not take place simultaneously [144]. When a lamina breaks, the stress applied to it will have to be taken over by the other intact laminae and thus will cause increases in stresses and strains. If the stresses and strains in the remaining sheets are still within safe limits, the laminate, as a whole, continues to take on higher loads until the final breakage of the laminate due to the breakage of all the sheets. Thus, as mentioned above, it is clear that the breaking process of a laminate is not predictable, but rather a gradual process, and the breaking, it can be said, takes place at the point when the breaking began, at the point when it is over, or between the two moments. Thus, the breaking load of a laminate depends on the philosophy adopted regarding the breaking of the laminate. Broadly speaking, there are two ways to define laminate breaking, first layer breaking and last layer breaking. Breaking the first layer is defined as breaking a laminate when the process of breaking the sheet begins with breaking the first layer [145]. This method is a fairly conservative approach to the design of a composite product and is generally used in primary structures. Breaking the last layer is defined as breaking a laminate when all layers yield. This method exposes the maximum load capacity of a laminate [33].

The methods initially developed for the production of materials with polymer matrix and reinforced with fiberglass, were also adopted for the design of other materials reinforced with different fibers. Fiberglass-reinforced polymer composites represent the largest class of polymer matrix composites. Polymeric materials with thermosetting matrix harden during polymerization. Polymerization or crosslinking takes place in thermosets by adding chemical agents, or by applying a heat treatment or a pressure gradient. These processes can cause some problems such as residual stresses as well as long periods of polymerization. Residual stresses can cause serious problems in asymmetric or thick laminated composites with polymer matrix, where deformations in the direction of warp, corrugation of fibers, micro-cracking of fibers and delamination can occur [1], [146], [147]. We also encounter other possible structural defects in the process of forming polymeric matrix composites, such as regions with excess resin (implicitly a deficit of resin near the fibers), cavities (at the intersection of the fibers in the warp direction with the fibers in the the direction of the weft, as well as between the blades positioned at different angles). As you can imagine, this is a problem that must be avoided if you want to obtain a material with small cavities and improved shear strength performance. Micro-cracking of the laminate or matrix must be avoided, this phenomenon may occur due to stresses during polymerization or moisture absorption during the formation of the laminate. Another possible defect could be the separation or delamination of some regions from the laminate, as well as variations in the alignment of the fibers [1].

1.7   Thermography of composite materials

Before considering conventional thermal properties, such as conductivity, it is necessary to briefly consider the effect of temperature on the mechanical properties of plastics. It was mentioned earlier that the properties of composite materials depend significantly on temperature. This is due to their molecular structure. Considering an amorphous composite material in which the molecular chains have a random configuration, inside the material, even if it is not possible to visualize them, we know that the molecules are in a state of continuous motion. As the material is heated, the molecules receive more energy and increase their relative motion. This makes the material more flexible. Conversely, if the material is cooled, then molecular mobility decreases and the material becomes more rigid [3], [148].

Thermography or thermal imaging is based on the principle that the thermal conductivity of a material is altered locally due to the presence of defects, such as delamination or inclusions. If the material containing such defects is subjected to a uniform heat source on a

surface, the passage of heat through this thickness will be uneven due to the local variation of the thermal conductivity. This will lead to an uneven surface temperature distribution, which is detected and related to the presence of defects inside the material. For example, the heat flow obstructed by a delaminated area increases the surface temperature above the delaminated area compared to that of the surrounding area. The most common technique in thermography is the application of a uniform heat source on one surface of the material and the recording of transient temperature contours on the other surface, either through liquid crystals or with an infrared heat detection chamber. The heat source can be as simple as hot water bags, hot air dryer or a light bulb stand. The flat location and flat size of the defect can be easily detected from the thermogram [56], [105], [149]–[173].

In [174], the authors developed a study on the characterization of defects and the thermomechanical behavior of polymeric materials reinforced with fiberglass, which were subjected to static loads at tensile stresses, using a passive infrared spectrography technique. Infrared thermography is a contactless technique and can be easily used under inspection, verification or maintenance. This allows a quick and real-time inspection (benefiting from the traceability of the inspected area) and can examine a relatively large area of a structure. Thus, the analysis of images from the infrared spectrum, allow the identification of defects on the surface of the material, but only in loading conditions >250MPa. However, this method has a limitation regarding the characterization of the material at low loads, due to the small temperature variation. In the mentioned study, the tested material was formed with unidirectional fiberglass, the layers being oriented at 0° and 90°. The conventional analysis of the images from the infrared spectrum, allowed to identify some defects of the material (interphase and interlaminar delamination) only at high levels of effort. For low stress levels (σ <250 MPa), no defect was identified in the infrared spectrum images, even if it really exists. To complete the information provided by the images in the infrared spectrum on the characterization of defects during the maximum loading process, the research was focused on the study of the thermomechanical behavior of the material. It has been shown that the thermoelastic coefficient of the material changes each time the applied stress exceeds the value of the material damage initiation threshold (σ_D = 50MPa). This variation of the thermoelastic coefficient was used to quantitatively assess the overall state of deterioration of the material even if it is early. Therefore, this variable describes well the state of deterioration of the material, not its state of stress and can be useful in monitoring the damage of mechanical components subjected to static loads of different amplitudes. However, it is not able to identify and locate damage to material. A combination of infrared spectrum imaging and thermoelastic coefficient variation analysis is recommended for a more reliable IR thermographic diagnosis.

A modified form of thermography is called vibrothermography [175]–[177], in which low-amplitude mechanical vibrations are used to induce localized heating in the material. The authors noted that localized heating occurs preferentially around internal defects, such as delamination and large cracks in the matrix.

1.8 Reinforced composites - mathematical models for properties

Laminated and fiber-reinforced composites are considered macroscopically, as inhomogeneous and anisotropic, i.e. they have distinct properties in different directions, starting from the same point. Under load, composite materials can be considered as linear-elastic bodies, so the relationships between specific stresses and strains are those corresponding to Hooke's law [32], [178]–[189].

A brief overview is described in [141] on relevant information on fracture mechanics. Of course, a number of specialty books are available in this area.

In a previous study, conducted at CCDCOMT [50], [136], a series of theoretical models on the behavior of reinforced materials from a mechanical point of view were presented.

An alternative to determining the mechanical properties of reinforced composite materials may be the use of FEM methods [190]. Thus, there is the possibility of numerical modeling of layered reinforced composites, starting from new concepts based on other approaches and other initial hypotheses. However, this method has some limitations due to the discretization interpolation system. For example, when there are deformations in the modeled system, distortions of the discretization of large areas occur and refined discretization are

required to solve structures with complex geometries and discontinuities. Thus, compared to the finite element method, some methods have been developed that do not require the discretization of surfaces. Interpolation in non-discretizing methods is based on a series of scattered nodes, this unique feature allows the partial elimination of the disadvantages of FEM methods. One of the first methods without discretization was fine particle hydrodynamics (HPF). Other methods have been developed based on HPF, which have applications in various engineering problems, such as simulating impact at high speeds, simulating surface stresses or forming metals. Nayroles, presented a non-discretization method, called the diffuse element method (MED), which uses approximations of the smallest square displacement. Belytschko also presented a method that includes derivations and interpolations omitted by MED, thus developing a more precise interpolation method, called the Galerkin without elements method (GFE), which has applications in the analysis of thin plates, thin coatings and dynamic fracturing of them. The methods mentioned above are widely presented in the literature [185], [191].

Taking into account the presented bibliographic studies, it is possible to outline a research paper on composite materials reinforced with fabrics, which could highlight the mechanical properties as well as a comparative analysis between the types of epoxy resins, as well as a comparative analysis between modified materials with solvent and unmodified materials. Thus, starting from the existing studies in the field of composite materials reinforced with fabrics, improvements can be made and I can optimize these materials through experimental methods, which led to the choice of the theme of this paper called: ***Research regarding mechanical characteristics of composite materials with reinforced fabric and polimeric matrix with elasticity gradient***

2. Research objectives

The main objective of this thesis is to establish a methodology for obtaining a new composite material reinforced with fabrics and flexible thermosetting matrix, by introducing into the epoxy matrix an agent of modification and flexibility. I also considered to identify the differences between materials with the same reinforcement structure, but with different types of epoxy resins as matrix.

To achieve this main objective, I have chosen the following specific objectives:

a) the study of the specialized literature regarding the formation of composite materials reinforced with carbon fiber, glass fiber, aramid fiber and mixed fiber fabrics as well as the influence of different types of fabric stratifications and orientations;

b) the study of the specialized literature regarding the influence of the epoxy matrix in the formation of composite materials reinforced with fabrics;

c) the study of the specialized literature regarding the influence of modifying the epoxy matrix with N-methyl-2-pyrrolidinone in the formation of composite materials reinforced with fabrics;

d) formation of composite materials reinforced with fabrics and epoxy matrix;

e) performing the experimental analysis for the characterization of the formed materials;

f) interpretation and study of the obtained results.

For each specific objectives proposed, theoretical studies are performed and the following experimental analyzes will be performed:

- following the analysis of the literature, I chose the architecture of pseudo-laminates in order to obtain a new material formed with flexible matrix;

- following the analysis of the specialized literature, I chose the technique for inserting and fixing the fabrics in the mold;

- following the analysis of the specialized literature, I chose the epoxy resins for the test matrix;

- formation of composite materials with epoxy control matrix. Thus, from each type of resin used, I formed a pseudo-laminate that was not subjected to heat treatment nor was it modified with N-methyl-2-pyrrolidinone;

- formation of composite materials with epoxy matrix and heat treated. Thus, from each type of resin used, I formed a pseudo-laminate that was subjected to heat treatment but was not modified with N-methyl-2-pyrrolidinone;

- formation of composites with epoxy matrix and modified with N-methyl-2-pyrrolidinone. Thus, from each type of resin used, I formed a pseudo-laminate that was not subjected to heat treatment but was modified with N-methyl-2-pyrrolidinone;

- formation of epoxy matrix composites, heat treated and modified with N-methyl-2-pyrrolidinone. Thus, from each type of resin used, I formed a pseudo-laminate which was subjected to heat treatment and was modified with N-methyl-2-pyrrolidinone;

- establishing the method of mechanical testing of the newly formed pseudo-laminate;

- cutting pseudo-laminates, in order to obtain test specimens for mechanical tests;

- performing tensile tests for fabric-reinforced pseudo-laminates;

- determine the maximum breaking temperature during the tensile test, using a thermal imaging camera;

- performing three-point bending tests for fabric-reinforced pseudo-laminates;

- determine the maximum bending temperature during the 3-point bending test, using a thermal imaging camera;

- comparison of test results to determine mechanical properties;

- identifying the role of the matrix on the tensile and bending behavior of pseudo-laminates (as long as they all have the same reinforcement structure);

- identifying the influence of heat treatment on the tensile and bending behavior of the material (because for each type of matrix one pseudo-laminate formed was heat treated and the other was not);

- identifying the influence of the NMP solvent on the tensile and bending behavior of the material (because for each type of matrix one pseudo-laminate formed was modified with NMP, and the other not);

-   monitoring the interphase effects and observing the effect of the solvent on the matrix, knowing that at the time of matrix failure, the fibers are subjected to the guillotine effect, this study also aims at the effects of NMP on the matrix, so that it stretches with the fiber, so that the strength increases.

3. Formation of composite materials reinforced with fabrics

3.1. Current hypothesis for the formation of composite materials and fabric preparation

In various studies conducted at CCDCOMT over the last decade, polymeric composites reinforced with epoxy resins and matrices have been characterized, which have shown remarkable mechanical results [98], [136], [192]–[206], thus, this study proposes to characterize the behavior of composite materials reinforced with carbon fiber fabrics, mixed fabrics, aramid fibers, glass fibers and homogeneous and heterogeneous epoxy matrix. Considering the advantages and disadvantages of the selected types of fabrics, I formed pseudo-laminates. These pseudo-laminates were created in order to obtain materials with a low degree of flexibility. For the formation of materials, it was established: the type of polymer matrix, the types of fabrics and the solvent (modifying agent), which was used in order to improve the degree of flexibility (elasticity gradient), mechanical properties, coefficient of thermal expansion and electric conductivity. The main advantage of pseudo-laminates is that, based on the forming technique, they allow a free distribution of the fiber arrangement [207].

Thus, in order to obtain structural stability of the pseudo-laminate [34], [35], [208], [209], bidirectional fabrics with simple weaving were selected, which also ensure the same mechanical strength both in the direction of weft and in the direction of the warp [62], [210]–[213], as well as resistance to delamination [51]. Using different types of fabric fibers for matrix reinforcement, a composite resulted, which shows the sum of the advantages of each constituent fiber as well as the attenuation of the disadvantages, to a certain degree.

However, it must be kept in mind that the finished product will have a maximum strength equivalent to its weakest constituent.

3.2. Epoxy resins used

The matrix role is to transfer a large part of the effort applied to the pseudo-laminate, to the fibers in the lamina or constituent layers [1], [193], [210], [214]. Thus, the matrix role is vital for the performance of the pseudo-laminate. For this reason, I chose the epoxy polymer matrix for the formation of composite materials. The advantages and disadvantages of this resin were presented in Chapter 1. From the category of epoxy resins was used the bi-component EPIPHEN system: the basic component EPIPHEN RE 4020 (bisphenol A) and hardener EPIPHEN DE 4020 (modified aliphatic amine), produced by Resoltech (hereinafter referred to as type E resin), due to the properties of the impregnation resin before/after polymerization, which are presented in [110]. The other bi-component epoxy resins used in this study, produced by R&G Gmbh Waldenbuch are called type C resin with C hardener, HT2 type resin with HT2 hardener, as well as L type resin with GL1 hardener (hereinafter referred to as type C, H and L resin). The properties of the resins are presented in [110]–[115].

3.3. N-methyl-2-Pyrrolidinone solvent

The ability of a fragile resin to absorb energy without permanent breakage can be increased either by flexibility or by hardening. Flexibility is achieved by mixing the resin with long chain compatible plasticizers, building long chain segments in the resin structure to increase the distance between the cross-linking sites or the use of flexible amine curing agents. Flexibility leads to the sacrifice of resin strength, rigidity and hardness, especially at high temperatures. Hardening, as in the case of flexibility, improves the ability of the resin system to withstand mechanical and thermal stresses. However, unlike flexibility, the raw properties of the matrix are almost unaffected. Thus, the compromise is reduced in terms of strength, stiffness, hardness or temperature resistance to increase toughness. Curing is achieved by dispersing a small amount of elastomer as a discrete phase of microscopic particles embedded in the continuous resin matrix [48].

N-methyl-2-pyrrolidinone (NMP) is an organic compound consisting of a five-membered lactam. It is a colorless liquid, although the impure samples may appear yellow. It is miscible

with water and the most common organic solvents. It can be used to disperse graphene in NMP, which gives a gradient of material flexibility [215]–[220] especially for those with shape memory [221]–[224]. It also belongs to the class of dipolar aprotic solvents, such as dimethylformamide and dimethyl sulfoxide.

The solvent used to form the materials was NMP. It is an important chemical raw material [227], [229], a polar solvent with good selectivity and stability properties, low toxicity, high boiling point, strong solvency, non-flammable, has many advantages, such as biodegradation, reuse, safety in use and suitable for many types of medical applications.

3.4. Material formation

In order to design and form the materials for this study, the manual layering method, known as hand lay-up or wet lay-up [193], was chosen because it is the most suitable technique when using a resin with a long gel time, thus ensuring a good impregnation of the pre-polymer between the fabric threads. Epoxy resins are generally polymers, which in the solid phase are obtained by chemical reactions developed in a mixture of two liquids, the pre-polymer mixture. Thus, the fragility of the polymer is higher when the gel time is shorter, which can lead to a lower quality of the polymer-reinforcement interface [207].

Hand lay-up, has an important advantage, namely, being a very simple process, requires very little special equipment and molds can be made of plaster, wood, sheet metal or even reinforced composite materials. The first step is to cover the mold with a release agent to prevent the material from adhering to the mold. It is followed by a thin layer of resin that has a number of functions. For example, it covers the irregular shape of the fibers and improves the physical appearance of the pseudo-laminate after it is removed from the mold. It also protects the reinforcement from the effects of moisture that would tend to destroy the fiber/resin interface. Continue with the application of the reinforcement and its impregnation with resin layer by layer. A roller or squeegee can be used to remove air bubbles between the layers. The advantage of this method is that the strength and rigidity of the composite can be controlled by increasing the thickness with other layers of material and resin, depending on the application. The polymerization takes place at room temperature, but sometimes a heat treatment is applied to accelerate this [3].

This forming method has beneficial advantages for this study, such as flexibility in the design process, the ease with which the reinforcements can be inserted in the mold, semi-professional tools, minimal investment in high-performance equipment, the relatively large dimensions at which pseudo-laminates can be produced, operator training is minimal and last but not least, the versatility through which the initial design of the pseudo-laminate can be modified. However, I must not neglect the disadvantages of this method, for example the large volume of hours required for pre-processing and composite formation, the quality of the finished product which depends largely on the training and experience of the operator, longer processing and polymerization times are needed, and material loss can be significant [208]. It should also be noted that this training technique has been perfected within CCDCOMT.

Thus, after consulting the literature and taking into account the latest advances in the field of reinforced composite materials, a certain arrangement of reinforcement layers (simple fabrics of carbon fiber, mixed fabrics, aramid fibers and glass fibers) was established. The next step was to cut sheets of fabric measuring 210×294mm. The sheets were cut, so that the orientation of the fibers was 90°, 30°, 45°, -30° and 0° to the warp direction.

To create a pseudo-laminate with low roughness surfaces, it was decided to use glass plates, one for the bottom, to ensure a uniform surface of the final product, and one to close the mold. The size of the glass plates was 250x340mm, and to ensure the tightness of the mold, a synthetic rubber gasket Iveolar type EPDM (modified ethylene-propylene-diene) was used. The dimensions of the gasket were 8x10mm with profile type D. Extracting the pseudo-laminate from the mold is itself a laborious process and for this reason, to facilitate a faster process, I placed a polyethylene foil (this being of superior quality , used for printing, so that the thickness of the foil is almost constant being previously laminated) between the glass plate and the first layer of reinforcement or resin because the epoxy resin does not adhere to the polyethylene surface and facilitates the extraction of material from the mold after natural polymerization of the matrix (14 days). It should be noted that a thin layer of wax release agent has been applied to the

surface of the glass plate so that the plate can be easily removed and thus reused. In addition, it is easier to peel off the formed polyethylene film than to peel off the glass plate.

Table 2 shows the fabrics used in the formation of pseudo-laminates, the orientation of the fabrics at different angles, as well as the concentration of solvent added to each package of fabrics. All fabrics are simple - that is, they are made by passing the weft threads alternately under a thread and over a warp thread.

Table 2 Pseudo-laminate architecture

	Fabric		Applied layers (fabric cutting angle)					NMP solvent concentration
1	Carbon fiber fabric	C_{240}	90⁰	30⁰	45⁰	-30⁰	0⁰	0%
2	Mixed fabric	C/A_{68}	90⁰	30⁰	45⁰	-30⁰	0⁰	3%
3	Aramid fiber fabric	A_{61}	90⁰	30⁰	45⁰	-30⁰	0⁰	6%
4	Fiberglass fabric	S_{163}	90⁰	30⁰	45⁰	-30⁰	0⁰	9%
5	Mixed fabric	C/A_{68}	90⁰	30⁰	45⁰	-30⁰	0⁰	12%
6	Aramid fiber fabric	A_{61}	90⁰	30⁰	45⁰	-30⁰	0⁰	15%
7	Carbon fiber fabric	C_{240}	90⁰	30⁰	45⁰	-30⁰	0⁰	18%

In the above table, the values next to the acronym that defines the type of fabric, identify the specific density of the material in g/m^2.

In order to form the pseudo-laminate, epoxy resin was also prepared. Thus, as I specified in the previous chapters, the resins used were bi-component polymeric systems, requiring the resin itself as well as a hardener. These components have been prepared in single-use containers. Also, for the modified materials with NMP, at this stage the solvent was prepared in a single-use container and being in percentage quantity, depending on the amount of resin used for the respective package. The amount of resin used, was determined in accordance with the manufacturer's recommendations. The bi-components of the pre-polymer mixture (three for NMP modified materials) were mixed with a mixer for 5 minutes until homogeneous.
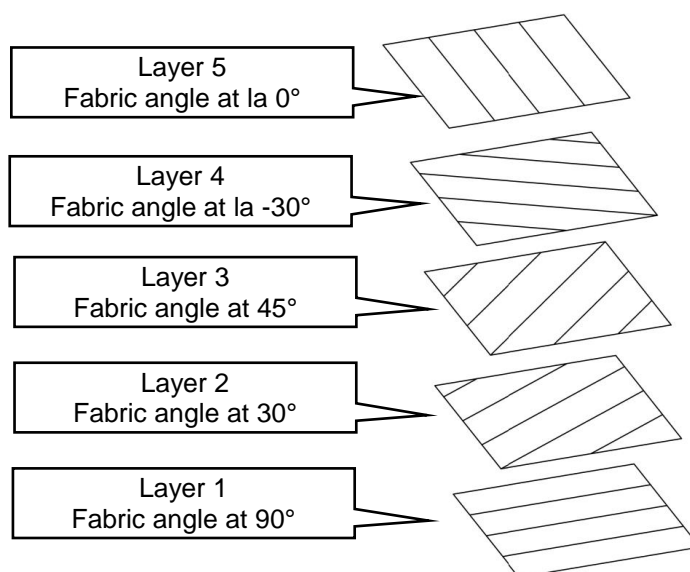

Fig. 3 Fiber orientation in each bundle of 5 fabrics

The stratification in the mold started by positioning the polyethylene foil, followed by the first carbon fiber fabric C_{240} oriented at 90⁰ (the orientation is relative to the direction of the warp of the fabric in relation to the longer side of the mold). This was followed by the second C_{240} fabric oriented at 30⁰. The third C_{240} fabric at 45⁰ was also positioned. Subsequently, I also positioned the fourth C_{240} fabric oriented at -30⁰. The last fabric positioned in the mold in this package was C_{240} oriented at 0⁰. This placement sequence is kept for all fabric types used for reinforcement, according to the information in **Table 2**. It should be specified that for NMP modified materials, in each package of the same reinforcement type, I added solvent in concentration according to the information from **Table 2**, and it was dispersed in the pre-polymer mixture. The sheets were also impregnated with resin. After inserting all the sheets into

the mold, it was closed, as specified above, with a second glass plate. The mold was fastened in metal clamps and placed in an upright position to facilitate the elimination of gases. The excess resin was removed by means of two slots of ~ 4 mm positioned at two opposite ends on the diagonal.

As I mentioned in the Introduction, the formed materials are, in appearance, pseudo-laminated, being made up of several layers of fabrics impregnated with pre-polymer mixture. The essential difference between laminates is that the layers are not glued together but the polymeric (or modified polymeric) matrix is continuous through any layer to the adjacent layers so that it can be considered to be fully engaged at the time of loading. This is different from the theory of laminates where the external load is distributed on the sheets and on the adhesive that ensures the interlaminar integrity.

Fig. 4 Formation of pseudo-laminates by hand lay-up method (from left to right: layering; gas evacuation; mold polymerization)

16 anisotropic composites with identical reinforcement were formed, of which only eight materials were modified with N-methyl-2-pyrrolidinone. Of the eight modified with NMP, only four materials (one for each type of epoxy resin used as a matrix) were heat treated. Of the eight unmodified with NMP, only four were heat treated. This results in the 16 materials formed according to **Table 3**. After formation, the properties of pseudo-laminates will be analyzed to determine the influence of the types of fabrics used, the orientation of the lamina fibers at different angles and the change of epoxy matrix on pseudo-laminate performance. All pseudo-laminates were formed of 35 layers according to the structure in **Table 2**.

Table 3 Treatments applied to the material

No.	Resin used	Heat treated pseudo-laminate	N-methyl 2-pyrrolidinone modified pseudo-laminate	Pseudo-laminate coding
1.	C	-	-	C1
2.		x	-	C2
3.		-	x	C3
4.		x	x	C4
5.	E	-	-	E1
6.		x	-	E2
7.		-	x	E3
8.		x	x	E4
9.	H	-	-	H1
10.		x	-	H2
11.		-	x	H3
12.		x	x	H4
13.	L	-	-	L1
14.		x	-	L2
15.		-	x	L3
16.		x	x	L4

To better understand how these pseudo-laminates are coded, I chose the option of assigning a number, in addition to the letter that defines the resin. Thus, for the control pseudo-laminate, in the case of resin C, I used the code C1. For the heat-treated pseudo-laminate, I used C2. For NMP-modified pseudo-laminate, I used C3, and for heat-treated and NMP-

modified pseudo-laminate, I used C4. For the other pseudo-laminates formed with epoxy resin, I kept the same coding, in which I changed only the letter corresponding to the type of resin, as shown in **Table 3**.

As specified above, depending on the epoxy resin used (C, E, H, L), four pseudo-laminates were formed with each type of resin. Following the use of the epoxy system, in order to complete the polymerization of the epoxy matrix and improve the mechanical properties, it is recommended to heat treat the formed materials. Thus, two of the four materials formed with each resin were heat treated in the oven after 30 days from formation. Thus, according to the manufacturer's recommendation, the materials were placed in an oven even before being removed from the mold for a first stage of heat treatment. After removing the mold, the pseudo-laminates were heat treated in a new stage.

Because the pseudo-laminate is asymmetrical and taking into account its architecture shown in **Table 2**, the two sides of each plate must be identifiable (especially from the perspective of bending tests). Thus, the surface corresponding to the reinforcement packages C_{240}-C/A_{68}-A_{61} compared to the fiberglass package will be marked **I**, and the surface corresponding to the packages C_{240}-A_{61}-C/A_{68}, compared to the fiberglass package will be denoted **II**. This notation will be important in the analysis of mechanical properties for bending tests.

The working hypothesis, for the present study, was to identify a polymeric composite material with epoxy matrix and elasticity gradient. Thus, as previously specified, I formed the pseudo-laminates, after which I posed the problem of the methodology for testing the working hypothesis. Considering the proposed ones and taking into account the CCDCOMT endowments and the studies carried out previously, I set out to perform mechanical tensile and bending tests, in order to characterize, from these points of view, the formed materials. Given the fact that I needed specimens for the tensile and bending tests according to the dimensions in **Fig. 6** and **Fig. 7**, I designed composite plates with dimensions 210×294mm. After forming materials, as specified above, only eight pseudo-laminates were heat treated. For reasons beyond my control, I did not have the opportunity to waterjet cut the plates until after about 12 months and, as a result, they were practically tested after 14-16 months after forming. This also results in a sufficient time for the natural polymerization for the eight non-heat-treated plates.

Fig. 5 Steps for extracting the specimens, using the water jet cutting machine (from left to right: pseudo-laminate extracted from the mold; cutting the pseudo-laminate; specimens before extraction from the pseudo-laminate)

In order to extract the specimens, as can be seen from **Fig. 1**, to determine the mechanical properties of the formed materials, I used the MAXIEM 2040 water jet cutting machine. It should also be mentioned that the cutting was performed with both water jet and sandblasting grit (special abrasive powder, used in cutting for water jet machines). Exclusive cutting with water jet, which in some samples was possible (they were not introduced in this study), highlighted the fact that the cutting line cannot be continuous due to the presence, especially, of aramid fibers, which have great resistance at shear. They deflected the water jet so that the edge of the specimen was not continuous, but was corrugated, which led to obtaining test specimens that were not viable in terms of mechanical tests because the sinuous edges can be considered cracked. The introduction of the sandblasting grid meant nothing more than increasing the density on the cutting flow. The abrasive edges of the sandblasting grit

granules cut the aramid fibers and thus resulted in smooth edges of the specimens being viable for testing.

The starting of the equipment and implicitly, of the water jet, was performed outside the field of the plate so as to minimize the mechanical effect of the water jet (close to the behavior of a small projectile) on the material. Until the jet penetrates, the shear strength (in this case water-induced) of each layer prevents the jet from penetrating (into the whole material) and then the grit would have been scattered inside the laminate, which, with water pressure, can induce a first delamination [230]–[234].

In principle, there are no standards for thermosetting matrix composites such as epoxy resins, so I chose to use the standard ISO 527-1:2019, usually applied to composites with thermoplastic matrix, and from the point of view of this standard, in terms of tensile stress, both rectangular and standard specimens may be used.

The study developed by the author Boboc in the paper from CCDCOMT [194], regarding the choice of the type of specimen, showed us that if we have a material formed with fabrics, the direction of force, greatly influences the response of the material and depends on the orientation of the fabrics.

From each formed material, a number of eight test pieces for tensile stress and a number of twelve test pieces for bending stress were extracted, as can be seen in **Fig. 8**.

Testing of specimens on the mechanical testing machine, using the INSTRON 8802 equipment and having a maximum load capacity of 100kN, was performed in accordance with ISO 527-1:2019, for tensile stress, and in accordance with standard 178:2003, for bending stress.

Thus, all tensile tests for the materials included in this study, had the same parameters set in the test equipment, namely: the speed of movement of the upper arm of 5mm/min and the stopping condition being the decrease of force by 50% or a movement of the upper arm more than 50 mm, the sizing of the specimens being performed as in **Fig. 6**.

Also, for the bending stress of the test specimens, the parameters set in the test equipment were: the speed of movement of the upper arm of 5 mm/min and the stopping condition being the movement of the upper arm after 37mm, the sizing of the specimens being done as in **Fig. 7**.

The formed materials were tested approximately twelve months after their formation, so we can say that the polymerization process was completed at a slow pace. It should be recalled that eight of the sixteen materials were heat treated in the oven, as shown in **Table 4**.
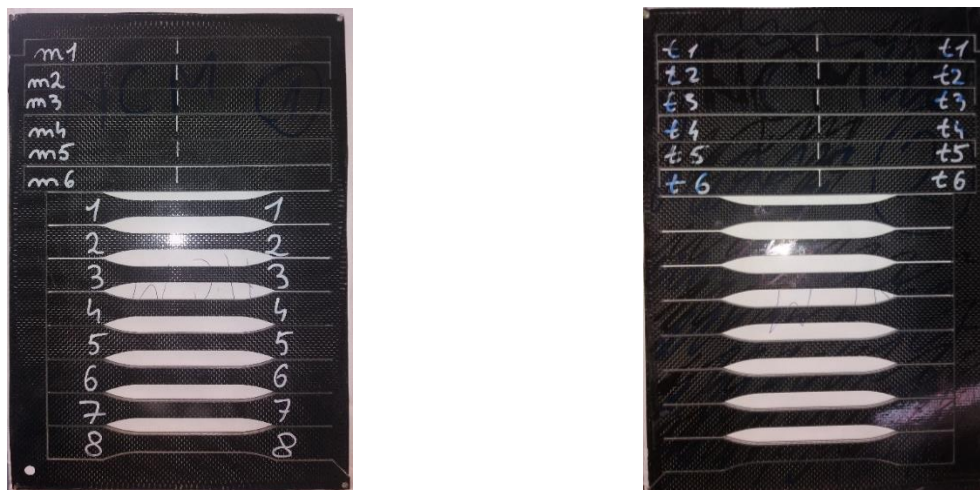

Fig. 8 Pseudo-laminate having the specimens noted, before being extracted

All tensile tests were recorded using two video recorders to accurately identify when the test specimen broke. The first video camera was thus positioned to record one of the faces (surfaces) of the specimen, and the second was positioned to record the side (thickness) of the specimen. This decision was made on the basis of the need to correlate some critical points on the loading curve with the breaking moments of some layers of the specimen. A single video camera was used for the bending stress, thus being positioned to record the thickness of the

specimen, in order to correlate significant moments (e.g. central delamination, relative slip of some layers, fracture of some layers).

For the bending stress, as the material is not symmetrical, I chose to test the specimens with the force applied from both directions: on surface I, respectively on surface II. Thus, the stress will be in the form of compression on the surface, for force application, and in the form of stretching on the opposite surface. This method is especially interesting in the case of pseudo-laminates modified with NMP solvent.

The bending moment, in the case of the 3-point bending test, increases from the end of the specimen to a maximum value in the middle of it. The maximum effort is reached along a line in the center of the specimen. The fibers may be arranged along or perpendicular to the specimen. When the fibers are arranged perpendicular to the length of the specimen, we obtain a value of the transverse resistance for the fiber-matrix interface. The shear strength remains constant and its maximum value will correspond to the maximum load [1].

With regard to the formed materials, it should be noted that the delamination phenomenon for pseudo-laminates is a delamination-like process. It does not consist in the classic delamination, as in the breaking of the adhesive between 2 sheets, in this case, the polymer yields between 2 sheets.

Among the objectives of the proposed study is to investigate the behavior of materials formed at bending and tensile stresses. The influence of the resin is also of interest, but also the type of fabric, as well as the alternation of the number of laminae or the change of angles. All this will determine the performance of the pseudo-laminate.

4. Analysis of mechanical properties of materials - tensile tests

The main advantage of using fabric-reinforced pseudo-laminates is that they offer properties that are more balanced in the 0° and 90° directions than unidirectional laminates. Although multilayer laminates can also be designed to produce balanced properties, the manufacturing time (layering) for fabric laminates is shorter than that of a multilayer laminate. However, the tensile strength and modulus of elasticity of a fabric laminate are generally lower than those of multilayer laminates. The main reason for the lower tensile properties is the presence of fiber ripple in the fabrics, as the fiber bundles in the warp direction traverse above and below the fiber bundles in the weft direction, to create an interlocked structure. Following tensile stresses, these corrugated fibers tend to straighten, which creates high interfaces, at reduced loads. As the load increases, micro cracks appear in the interface, which subsequently propagate into the matrix. This is also highlighted by the appearance of one or more peaks in the stress-strain diagrams of laminates formed with fabrics [56], [236], [237]. For testing the specimens, they are subjected to axial stresses to the point of rupture and the deformations occurring in the longitudinal and transverse directions are recorded in a continuous flow using the BlueHill software component for the control of the mechanical testing equipment. In order to determine the mechanical constants, this is done by analyzing the load-elongation (or effort-deformation) curves. The calculation of the mechanical tensile parameters is made only in the linear area, considered elastic area [136], [238]–[240].

4.1. Mechanical properties of composite materials formed with type C resin

4.1.5. Conclusion pseudo-laminate C

In the case of materials with unmodified epoxy matrix C from **Fig. 10** and **Fig. 16**, it can be easily observed that, in the case of naturally polymerized materials (C1), the results obtained (σ/ε curves) are more widespread than the results obtained for the heat treated material (C2), but the differences are not significant. The specimen with the most different result was specimen 8, for both C1 and C2, i.e. those located at the edge of the pseudo-laminate and, therefore, perhaps their structure was affected by the high-pressure water jet that penetrated the field of the plate, taking into account that the cutting began with these specimens. After the 8 specimens have been extracted, the rigidity of the pseudo-laminate is reduced and it is less likely to affect the following samples.

It should be noted that, in the case of specimens with approximately the same behavior, the specimen numbered 5 showed the highest increase in temperature (compared to ambient temperature), both for naturally polymerized materials (C1) and for heat-treated ones (C2). The difference between the highest (specimen 5) and lowest (specimen 6) temperatures (relative to ambient temperature) for the natural polymerized composite material (C1) is 18.63°C, while the other two specimens recorded temperature values around the extreme average. In the infrared spectrum of the material C1, it is observed that the heating of the test specimen 5 is extended between the two fracture points. In the graph of the mean curve for C2 (heat-treated) materials, the differences between the extreme temperature values are 5.74°C (48.49°C for specimen 5 and 42.75°C for specimen 6). In this case, a value of 48.47°C was recorded for specimen 2 (practically the same as for specimen 5), and for the last specimen the value recorded was 45.09°C. Analyzing only the thermal aspects, we could say that the heat treatment contributes to the consolidation of composite materials leading to a better quality of the interfaces. In general, for materials C1 and C2, in the case of medium specimens, the core package of the reinforcement (S_{163}) is not affected, being protected by the outer fabric packages C_{240}.

In **Fig. 26** and **Fig. 36**, are presented the curves of the materials with epoxy matrix C modified with NMP, resulting from the tensile stress. Test specimen 6, for both C3 and C4 material, shows the most different tensile response.

For pseudo-laminate C3, specimen 3 showed the highest increase in temperature relative to the environment (24.7°C), while specimen 7 had the smallest increase (16.2°C).

For pseudo-laminate C4, specimen 5 showed the highest increase in temperature relative to the environment (19.2°C), while specimen 2 had the smallest increase (12.9°C).

In the case of specimens included in the average behavior of composite C3, the temperature difference between the highest value (44.7°C) and the lowest (36.2°C) was 8.5°C.

In the case of specimens included in the average behavior of the C4 material, the temperature difference between the highest value (43°C) and the lowest (36.7°C) was 6.3°C.
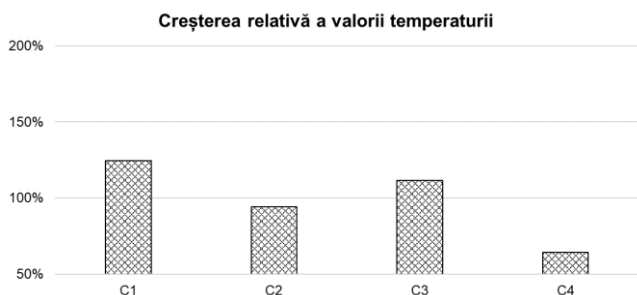

Fig. 46 Relative increase in temperature value, for materials formed with type C resin
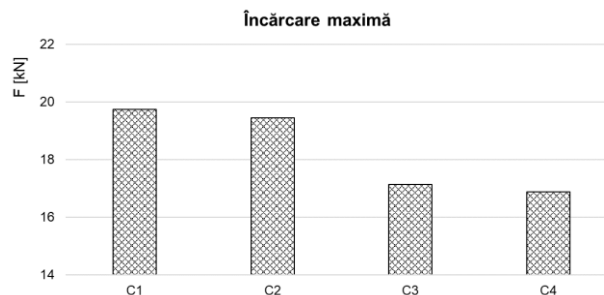

Fig. 47 Maximum load for type C resin materials

In **Fig. 46**, it can be seen that the material C1 showed the highest increases in temperature compared to the environment, because this pseudo-laminate was not heat treated or modified with NMP, resulting in a more rigid material compared to C4, which, it was both heat-treated and modified with NMP.

From a mechanical point of view, it is easy to see how the behavior of the specimens is more widespread in the case of heat-treated materials (C4) than in the case of naturally polymerized materials (C3). In the infrared spectrum of the C3 material, it is observed that the heating of the specimen 3 is extended between the two fracture points.

Analyzing the average results of the force at the maximum load in **Fig. 47** of the materials formed with type C resin, we can observe that from this point of view, the best pseudo-laminate, was the material that was not modified with NMP and was not heat treated, C1. However, perhaps this desideratum was known from the beginning. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that after the tensile tests performed, the best material was C3, i.e. the material formed with type C resin, modified with NMP and which was not subjected to a heat treatment, but was only naturally cured and tested one year after its formation. Material C3 has a force value at maximum load 1.6% higher than material C4.
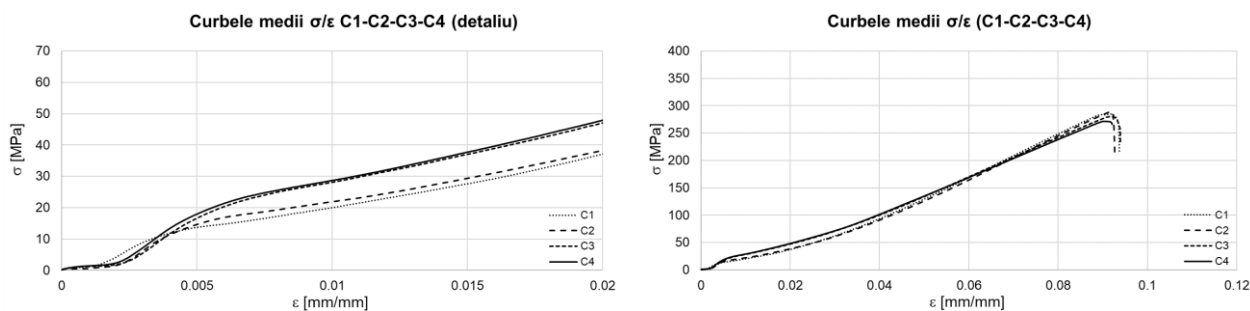

Fig. 48 Comparison between materials C1-C2-C3-C4

In the graph of the mean curves σ/ε C1, C2, C3 and C4, the results obtained after the tensile test of the selected specimens and extracted from the composite materials are compared. Following a quantitative analysis, we can conclude that the heat treatment applied influences the performance of the composite in a positive way, but only to a small extent, although the heat treatment brings a benefit to the polymerization of the composite, in the sense that it uniformizes the bonds between layers and matrices.

Analyzing the modulus of elasticity resulting from the mean curves σ/ε, from **Fig. 48** - **Fig. 49** of the materials formed with type C resin, we can observe that from this point of view, the best pseudo-laminate was the material modified with NMP but not heat treated, C3. It should be noted that in the calculation of the mean curve σ/ε, only specimens with similar behavior were included. Also, in **Fig. 49**, a graph is presented with the modulus of elasticity

obtained automatically by the software used by the traction machine, and the main observation here is that the best material in terms of modulus of elasticity would be C2, but having a higher value by 1.6% compared to C1, or if we refer to the elasticity gradient, C3, but it is also higher than C4 by only 4.5%. Thus, if we did not analyze the data obtained through an in-depth study, we could reach unrealistic, expensive solutions, or materials that can fracture under certain extreme conditions.
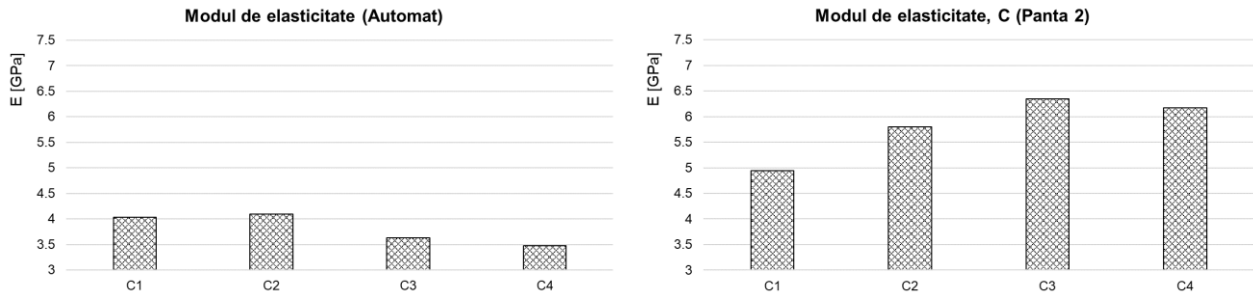

Fig. 49 The modulus of elasticity of pseudo-laminates formed with type C resin

In **Fig. 49**, the values of the modulus of elasticity calculated for each material formed with type C resin are presented. Heat-treated materials should have a higher value of the modulus of elasticity (calculated) than those not heat-treated, which in the case of materials not modified with NMP, C1 and C2, are verified, C2 being 17.3% higher than C1. In the case of materials modified with NMP, the situation is exactly the opposite, i.e. the modulus of elasticity of the C4 material is 2.8% lower than C3, the difference not being significant.

Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that after the tensile tests performed, the best material was C3, i.e. the material formed with type C resin, modified with NMP and which has not undergone any heat treatment but which was tested one year after its formation. C3 has a modulus of elasticity (calculated) higher than C4 by 2.9%, compared to C1 higher by 28.3%, compared to C2 higher by 9.4%.

4.2.   Mechanical properties of composite materials formed with type E resin

4.2.5. Conclusions pseudo-laminate E

In **Fig. 50** and **Fig. 56** are presented the σ/ε curves of the materials with epoxy matrix E, resulting from the tensile stress. Material E2, unlike E1, was heat treated. Test specimen 6 for material E1, respectively specimen 8 for material E2, shows the weakest tensile response. Regarding the poor response of test piece 6, its behavior is probably due to internal defects (lack of pre-polymer between two or more reinforcement layers), and the fracture of the test piece affected both packages of external reinforcement layers with their displacement opposite the central reinforcement package (S_{163}). The difference in heating temperature values for material E1 is 17.43°C (between specimen 1 and specimen 8). In the case of material E2 (specimen 3 and specimen 6 respectively), the temperature difference is 3.31°C, but the highest value is reached in the case of specimen 4 and is 51.68°C.

In **Fig. 66** and **Fig. 76**, are presented the σ/ε curves of the materials with epoxy matrix E modified with NMP resulting from the tensile stress. The E4 material, unlike E3, was heat treated. Test specimen 1 for material E3, respectively specimen 3 for material E4, shows the weakest tensile response, although both were included in the calculation of the average behavior of the materials. For pseudo-laminate E3, specimen 8 showed the highest increase in temperature relative to the environment (29.89°C), while specimen 1 had the smallest increase (15.29°C). For pseudo-laminate E4, specimen 4 showed the highest increase in temperature relative to the environment (29.26°C), while specimen 8 had the smallest increase (14.47°C). In the case of specimens included in the average behavior of composite E3, the temperature difference between the highest value (51.59°C) and the lowest (36.99°C) was 14.6°C. In the case of specimens included in the average behavior of composite E4, the temperature difference between the highest value (50.96°C) and the lowest (36.17°C) was 14.79°C. From a

mechanical point of view, it is easy to see how the behavior of the specimens is more widespread in the case of naturally polymerized materials (E1 and E3) than in the case of heat-treated materials (E2 and E4).

In **Fig. 86**, it can be seen that the material E4 showed the highest increase in temperature compared to the environment, while the material formed with type C resin, the highest relative increase in temperature was recorded in the untreated pseudo-laminate and unchanged, C1 (24.4°C). In the case of materials formed with type E resin, the highest increase in temperature was recorded as opposed to the first, i.e. the material heat treated and modified with NMP, E4 (23.8°C).
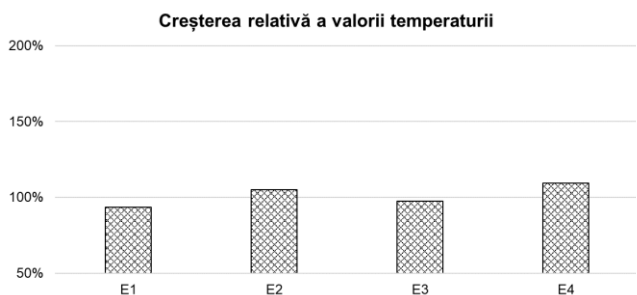

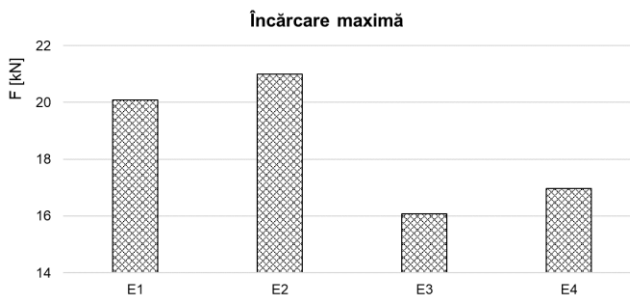**Fig. 86** Relative increase in temperature value, for materials formed with type E resin

Fig. 87 Maximum load for materials formed with type E resin

Analyzing the average results of the force at the maximum load in **Fig. 87** of the materials formed with type E resin, we can observe that from this point of view, the best pseudo-laminate was the material that was not modified with NMP, but was heat treated, E2. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that after the tensile tests performed, the best material was E4, i.e. the material formed with resin type E, modified with NMP and subjected to heat treatment and which was tested one year after its formation. The E4 material has a maximum loading force 5.5% higher than the E3 material, and compared to the pseudo-laminate with the best response, E2, 19.2% lower.
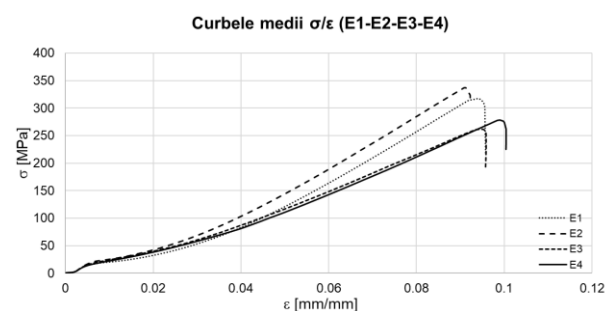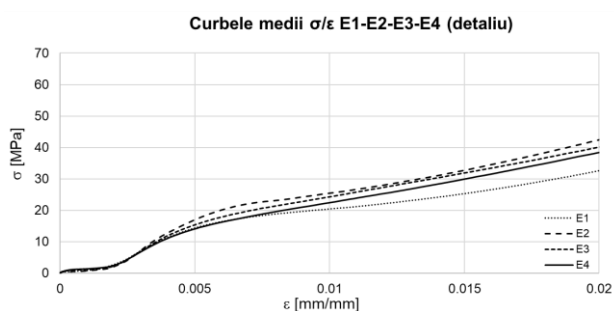

Fig. 88 Comparison between materials E1-E2-E3-E4

In the mean curves graph σ/ε E1, E2, E3 and E4, the results obtained after the tensile test of the selected specimens and extracted from the composite materials are compared. Following a quantitative analysis, we can conclude that the heat treatment applied influences the performance of the composite in a positive way, for materials not modified with NMP (E1, E2), while for materials modified with NMP (E3, E4), the heat treatment is not beneficial, although the heat treatment brings a benefit to the polymerization of the composite, in the sense that it uniformizes the bonds between the layers and the matrix.

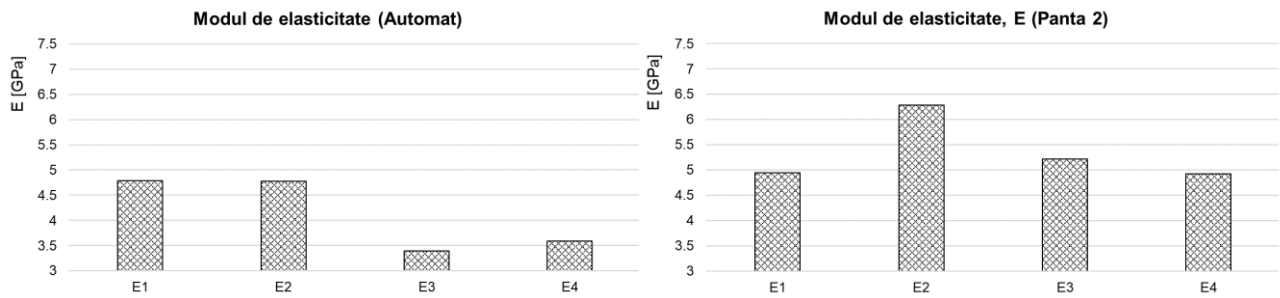**Fig. 89** The modulus of elasticity of pseudo-laminates formed with type E resin

Analyzing the modulus of elasticity resulting from the mean curves σ/ε, from **Fig. 89**, of the materials formed with type E resin, we can observe that from this point of view, the best pseudo-laminate was the material not modified with NMP but heat treated, E2. It should be noted that in the calculation of the mean curve σ/ε, only specimens with similar behavior were included. Also in **Fig. 89**, a graph is presented with the elastic modules obtained automatically by the software used by the traction machine, and the main observation here is that the best material from the point of view of the elastic modulus would also be E2, but having a value 1.1% higher than E1, or if we refer to the elasticity gradient, E4, but it is also higher than E3 only 3.5%. Thus, if we did not analyze the data obtained through an in-depth study, we could reach unrealistic, expensive solutions, or materials that can fracture under certain extreme conditions.

In **Fig. 89**, the values of the modulus of elasticity calculated for each material formed with type E resin are presented. The heat-treated materials should have a higher value of the modulus of elasticity (calculated) than those not heat-treated, which, in the case of NMP, E1 and E2, are verified, E2 being 27.3% higher than E1. In the case of materials modified with NMP, the situation is exactly the opposite, i.e. the modulus of elasticity of the material E4, is 5.5% lower than E3, the difference not being significant.

Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that after the tensile tests performed, the best material was E3, i.e. the material formed with resin type E, modified with NMP and which has not undergone heat treatment, but which was tested one year after its formation. E3 has a modulus of elasticity higher than E4 by 5.8%, compared to E1 higher by 5.5%, compared to E2 lower by 17.1%.

4.3. Mechanical properties of composite materials formed with type H resin

4.3.5. Conclusion pseudo-laminate H

Regarding the materials formed with type H resin, the results are presented in **Fig. 90** and **Fig. 96**. As with other materials presented, the behavior of the specimens after tensile testing is more widespread in the case of natural polymerized material (H1) and, as in the results already presented, for these materials the fracture affected both packages of layers of reinforcement (those made of C_{240} carbon fiber fabric), but in some cases (such as those with the lowest response - test specimen 4 for H1 and test specimen 8 for H2), the core package of the material (S_{163}) is also affected. The average curves for these materials are made on five specimens, in the case of material H1, and on three specimens, in the case of material H2.

The heating effect at fracture of the specimens is also present and has the highest value of 19.07°C (67.10°C for specimen 1 and 45.82°C for specimen 5 and specimen 6 of 45.93°C, respectively) for material H1 and H2, the highest value is 4.52°C (for specimen 5 and specimen 7).

In **Fig. 105** and **Fig. 114**, are presented the curves of the materials with epoxy matrix H modified with NMP resulting from the tensile stress. Test specimen 8, for material H3, and test specimen 7, for material H4, shows the weakest tensile response. For pseudo-laminate H3, specimen 2 showed the highest increase in temperature relative to the environment (21.15°C), while specimen 3 had the smallest increase (15.48°C). For pseudo-laminate H4, specimen 4 showed the highest increase in temperature relative to the environment (28.07°C), while specimen 5 had the smallest increase (13.63°C). In the case of the

specimens included in the average behavior of the H3 composite material, the temperature difference between the highest value (42.85°C) and the lowest (37.18°C) was 5.67°C. In the case of the specimens included in the average behavior of the H4 composite material, the temperature difference between the highest value (49.77°C) and the lowest (35.33°C) was 14.44°C.

From a mechanical point of view, it is easy to observe how the behavior of the specimens is more widespread in the case of naturally polymerized materials (H1) than in the case of heat-treated materials (H2), and for materials with H matrix and modified with NMP, the effect seems to be reversed.

In **Fig. 142**, it can be observed that the material H1 showed the highest increase in the temperature value compared to the environment, as in the case of the material formed with type C resin, where the highest relative increase in the temperature value was recorded at pseudo-untreated and unmodified laminate, C1 (24.4°C). For material H1, the temperature increase value was 25.5°C.
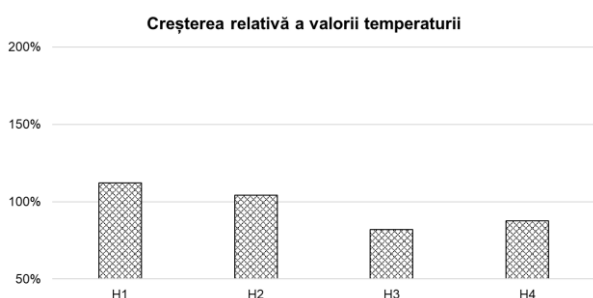

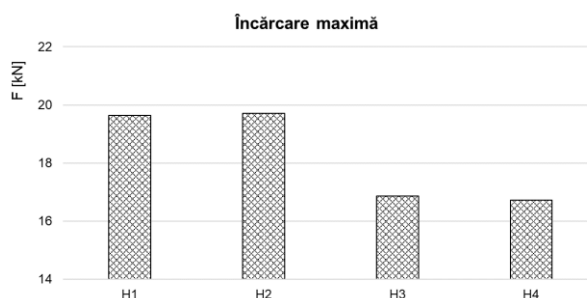**Fig. 124** Relative increase in temperature value, for materials formed with type H resin

Fig. 125 Maximum load for type C resin materials

Analyzing the average results of the force at the maximum load in **Fig. 125**, of the materials formed with type H resin, we can observe that from this point of view, the best pseudo-laminate was the material that was not modified with NMP, but was heat treated, H2. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that after the tensile tests performed, the best material was H3, i.e. the material formed with resin type H, modified with NMP and which was not subjected to a heat treatment, but was only naturally cured and tested one year after its formation. The H3 material has a maximum loading effort by 0.9% higher than the H4 material, and compared to the pseudo-laminate with the best response, H2, lower by 14.4%.
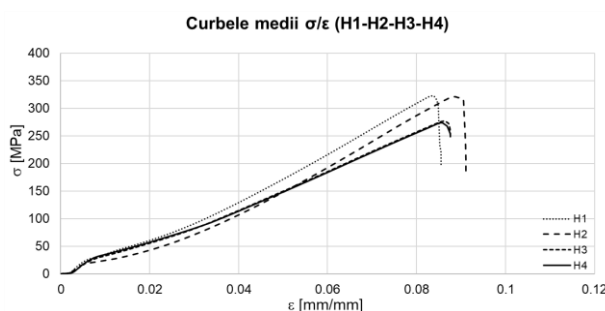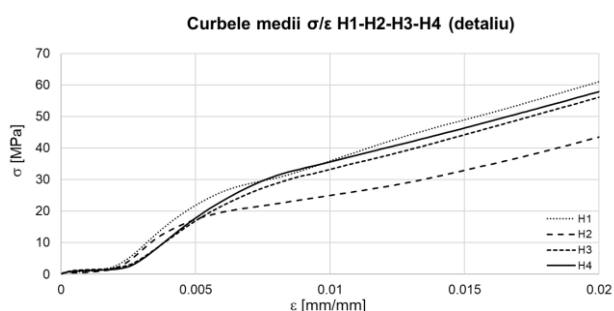
Fig. 126 Comparison between materials H1-H2 and H3-H4

In the mean curves graph σ/ε H1, H2, H3 and H4, the results obtained after the tensile test of the selected specimens and extracted from the composite materials are compared. Following a quantitative analysis, we can conclude that the heat treatment applied influences the performance of the composite in a positive way, for materials modified with NMP (H3, H4), while for materials not modified with NMP (H1, H2), the heat treatment is not beneficial, although the heat treatment brings a benefit to the polymerization of the composite, in the sense that it uniformizes the bonds between the layers and the matrix.
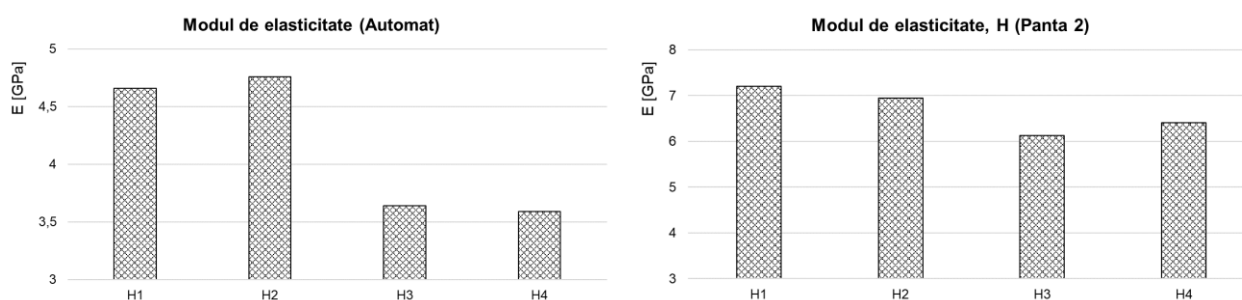
Fig. 127 The modulus of elasticity of pseudo-laminates formed with type H resin

Analyzing the modulus of elasticity resulting from the mean curves σ/ε, from **Fig. 126** - **Fig. 127**, of the materials formed with H type resin, we can observe that from this point of view, the best pseudo-laminate was the material not modified with NMP and not heat treated, H1. It should be noted that in the calculation of the mean curve σ/ε, only specimens with similar behavior were included. Also, in **Fig. 127** a graph is presented with the elastic modulus obtained automatically by the software used by the traction machine, and the main observation here is that the best material in terms of modulus of elasticity would be H2, but having a higher value by 3.8% compared to H1, or if we refer to the elasticity gradient, H3, but it is also higher than H4 by only 1.8%. Thus, if we did not analyze the data obtained, through an in-depth study, we could reach unrealistic, expensive solutions, or materials that can fracture under certain extreme conditions.

In **Fig. 126**, the values of the modulus of elasticity calculated for each material formed with type H resin are presented. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that tensile tests performed, the best material was H4, i.e. the material formed with type H resin, modified with NMP and which was subjected to a heat treatment, but which was tested one year later after its formation. H4 has a modulus of elasticity higher than H3 by 4.6%, compared to H1 lower by 11.0%, compared to H2 lower by 7.6%.

4.4. Mechanical properties of composite materials formed with type L resin

4.4.5. Conclusions pseudo-laminate L

The last four materials - just because I chose to present them alphabetically - are shown in **Fig. 128**, **Fig. 134**, **Fig. 144** și **Fig. 153**. For the first two, it is easy to see that the spread of naturally cured specimens did not allow an average of more than three specimens. In the case of the L1 matrix material, the lowest response is again that of specimen 8, while in the case of the L2 matrix material, the lowest response was considered to be that of specimen 7. For these composite materials, we observed that the core package (S_{163}) of the reinforcement is affected by tensile tests (in both cases of naturally polymerized matrix - L1 and heat-treated matrix - L2). Regarding the relative increase in temperature, there was a difference of 8.07°C for the L1 matrix material and 13.02°C for the L2 matrix material (which appears to be opposite to the other three materials). It should be noted that the highest temperature values reached during these material tests were 62.20°C for material L1, specimen 7 and, 65.09°C for specimen 2 of material E2.

In **Fig. 144** and **Fig. 153**, are presented the curves of the materials with epoxy matrix L modified with NMP resulting from the tensile stress. Specimen 2, for material L3, respectively specimen 4 for material L4, shows the weakest response to tensile, although both were included in the calculation of the average behavior of the materials. For pseudo-laminate L3, specimen 2 showed the highest increase in temperature relative to the environment (36.11°C), while specimen 8 had the smallest increase (15.57°C). For pseudo-laminate L4, specimen 3 showed the highest increase in temperature relative to the environment (24.43°C), while specimen 6 had the smallest increase (16.68°C). In the case of the specimens included in the average behavior of the L3 composite material, the temperature difference between the highest value (59.01°C) and the lowest (38.47°C) was 20.54°C. In the case of specimens included in

the average behavior of L4 composite material, the temperature difference between the highest value (47.33°C) and the lowest (39.58°C) was 7.75°C.

From a mechanical point of view, it is easy to see how the behavior of the specimens is more widespread in the case of naturally polymerized materials (L1) than in the case of heat-treated materials (L2), and for materials with L matrix and modified with NMP, the effect seems to be approx. similar.

In **Fig. 163**, it can be observed that the L1 material showed the highest increase in the temperature value compared to the environment. These increases in the temperature value were recorded, in the case of materials formed with epoxy resin type C and H, also for the naturally polymerized material. Thus, for the material formed with type C resin, the highest relative increase in the temperature value was registered for the unheated and unmodified pseudo-laminate, C1 (24.4°C). And for the material formed with type H resin, the highest relative increase in temperature was recorded in the unheated and unmodified pseudo-laminate, H1 (25.5°C).

Analyzing the average results of the force at the maximum load in **Fig. 164**, of the materials formed with L type resin, we can observe that from this point of view, the best pseudo-laminate was the natural polymerized material and which was not modified with NMP, L1. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, it can be said that following the tensile tests performed, the best material was L3, i.e. the material formed with type L resin, modified with NMP, naturally polymerized and tested one year after its formation. The L3 material has a maximum loading force 6.1% higher than the L4 material, and compared to the pseudo-laminate with the best response, L1, 7.1% lower.
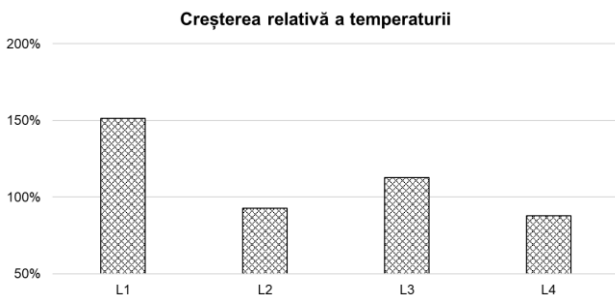
Fig. 163 Relative increase of the temperature value, for materials formed with L type resin
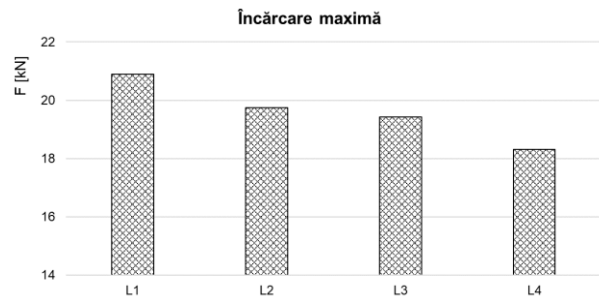
Fig. 164 Maximum load for materials formed with type L resin
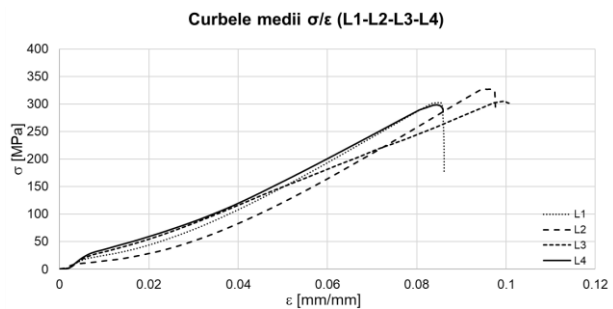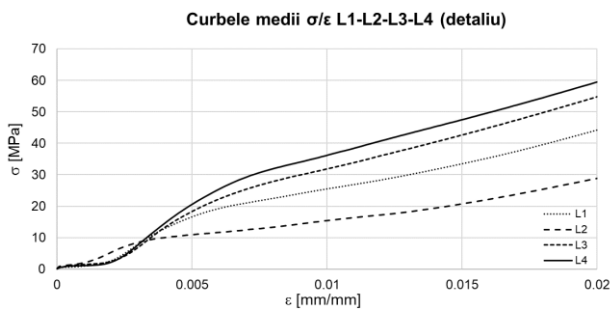
Fig. 165 Comparison between materials L1-L2-L3-L4

In the graph of the average curves σ/ε L1, L2, L3 and L4, the results obtained after the tensile test of the selected specimens and extracted from the composite materials are compared. Following a quantitative analysis, we can conclude that the heat treatment applied influences the performance of the composite in a positive way, for materials modified with NMP (L3, L4), while for materials not modified with NMP (L1, L2), the heat treatment is not beneficial, although the heat treatment brings a benefit to the polymerization of the composite, in the sense that it uniformizes the bonds between the layers and the matrix.
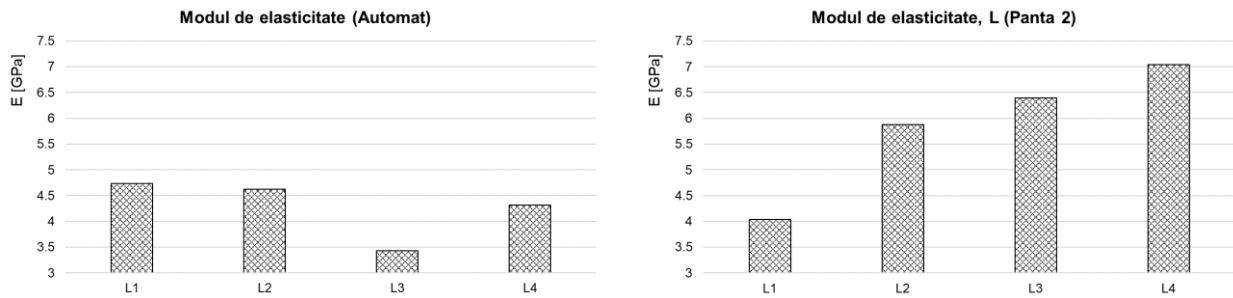
Fig. 166 The modulus of elasticity of pseudo-laminates formed with type L resin

Analyzing the modulus of elasticity resulting from the mean curves σ/ε, from **Fig. 165** - **Fig. 166**, of the materials formed with L type resin, we can observe that from this point of view, the best pseudo-laminate was the material unmodified with NMP, but heat treated, L2. It should be noted that in the calculation of the mean curve σ/ε, only specimens with similar behavior were included. Also, in **Fig. 166**, a graph is presented with the modulus of elasticity obtained automatically by the software used by the traction machine, and the main observation here is that the best material in terms of modulus of elasticity would be L1, but having a higher value by 4.0% compared to L2, or if we refer to the elasticity gradient, L4, but it is also higher than L3 by 24.4%. Thus, if we did not analyze the data obtained, through an in-depth study, we could reach unrealistic, expensive solutions, or materials that can fracture under certain extreme conditions.

In **Fig. 166**, the values of the modulus of elasticity calculated for each material formed with L-type resin are presented. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that the best material was L4, i.e. the material formed with L-type resin, modified with NMP and which was subjected to a heat treatment, but which was tested one year later after its formation. L4 has a modulus of elasticity higher than L3 by 10.1%, compared to L1 higher by 74.1%, compared to L2 higher by 19.8%.

4.5. Comparative analysis of pseudo-laminates

Analyzing the average curves of the materials, we can identify some slopes of the mechanical behavior of all specimens. The first slope is very short and almost horizontal and could correspond to the grip of the specimen between the pneumatic arms together with the beginning of the loading. The second slope corresponds to the elastic response of the material while slopes three, four and five could correspond to the defects of certain interfaces, such as slope four, which could be associated with the detachment of the carbon fiber layers from the others in the core package. The last slope for each material corresponds to the linear response of the carbon fiber fabrics outside the matrix. Even in this case, it is difficult to analyze all mechanical loads, because the appearance of fractures of the carbon fiber reinforcement packages at the outer layers looks mainly as bending-induced fractures and, for some specimens after fracture, they rotate relative to the central package (which is held in the loading position of the clamps), signaling that some shear forces occur. In **Table 21**, the values of all slopes are presented for each mean curve and, of course, only the second could be considered the modulus of elasticity.
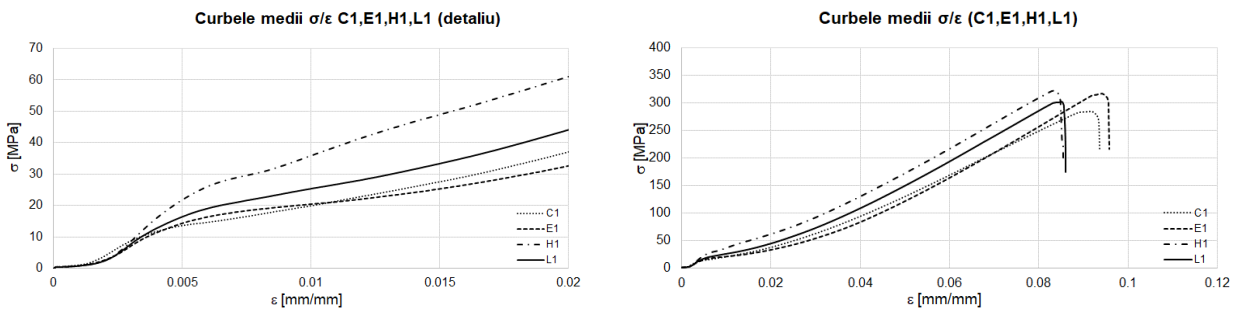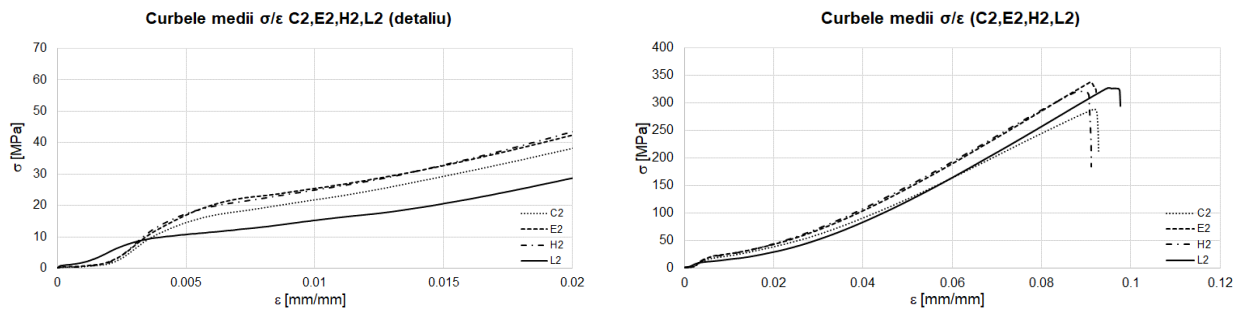
Fig. 167 Comparative analysis of the average curves of heat treated/untreated materials

In the case of pseudo-laminates naturally polymerized and unmodified with NMP, it can be observed that from the point of view of the mean curves σ/ε, the best answer is presented by the material formed with type H resin, while the material formed with type E resin has the weakest response, followed very closely by the material formed with type C resin.

For the pseudo-laminates unmodified with NMP, but heat treated, it can be seen that from the point of view of the average curves σ/ε, the best answer is presented by the material formed with type E resin, being followed very closely by the material formed with type H resin, while the material formed with type L resin shows the weakest response.

In the case of pseudo-laminates modified with NMP and not heat treated, it can be seen that from the point of view of the average curves σ/ε, the best answer is the material formed with resin type H, followed very closely by the material formed with resin type L, while the material formed with type E resin shows the weakest response.
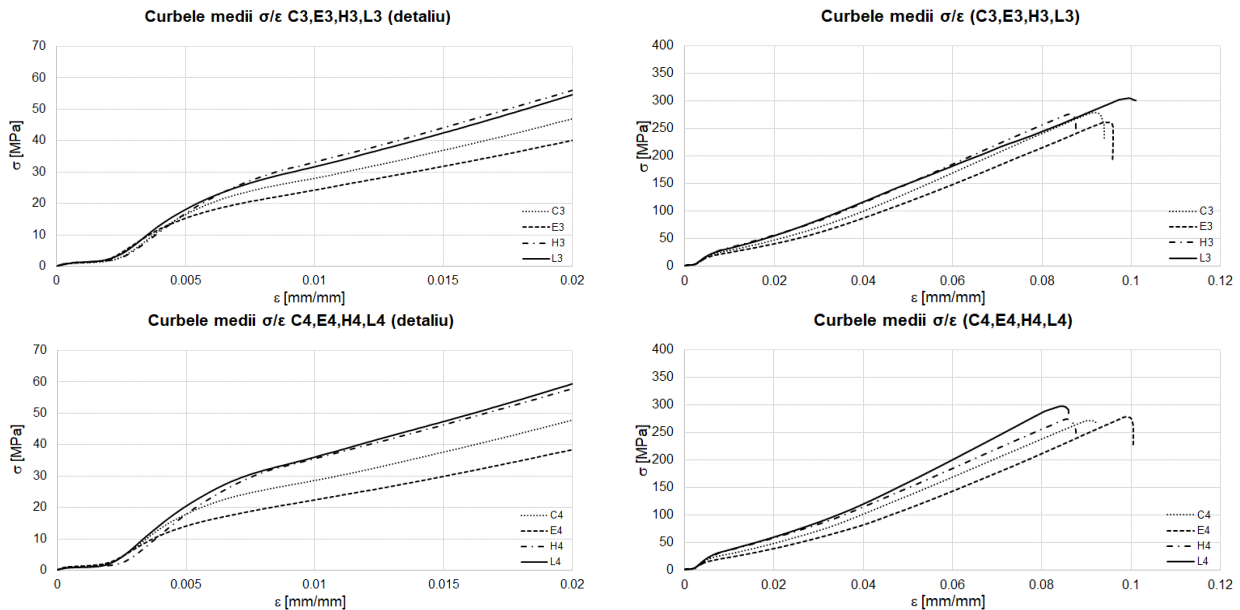
Fig. 168 Comparative analysis of average curves of NMP modified and heat treated / untreated materials

For pseudo-laminates modified with NMP, but heat treated, it can be seen that from the point of view of the average curves σ/ε, the best answer is presented by the material formed with resin type L, being followed very closely by the material formed with resin type H, while the material formed with type E resin shows the weakest response.

						Table 21 Calculated modulus of elasticity values [GPa]	
Material	E (slope 2)	Material	E (slop 2)	Material	E (slop 2)	Material	E (slop 2)
C1	**4.95**	E1	**4.94**	H1	**7.20**	L1	**4.04**
C2	**5.80**	E2	**6.29**	H2	**6.94**	L2	**5.88**
C3	**6.35**	E3	**5.21**	H3	**6.13**	L3	**6.39**
C4	**6.17**	E4	**4.92**	H4	**6.41**	L4	**7.04**

The modulus of elasticity seems to be higher for unmodified but heat-treated materials, except for the material of the H matrix. Thus, C2 is higher than C1 by 17.3%, E2 is higher than E1 by 27.3%, H2 is lower compared to H1 by 3.7%, L2 is higher than L1 by 45.3%.

Heat-treated materials should have a higher value of modulus of elasticity (calculated) than non-heat-treated ones, which, in the case of non-NMP modified and resin-formed materials type C, E and L, is validated, except only materials formed with H-type resin, where the effect appears to be opposite. Thus, for this, we need to analyze what happens differently from other types of resins. Taking into account the gel time of the H resin of only 60 minutes, which is quite short, the result can lead to a more rigid finished material. Applying a heat treatment can influence the expansion and contraction of the fibers due to the fact that the H resin is very rigid but also due to the fact that the heat treatment stiffens the polymer and thus the material can be weakened and the cracks propagate and section the fibers.

Also, we can formulate a similar conclusion for the materials modified with NMP, following the influence of the applied heat treatment, on the modulus of elasticity. Thus, from the test results we can conclude that the heat treatment for materials modified with NMP, led to higher values of modulus of elasticity only for materials formed with resin type H and L, while for materials formed with resin type C and E, the effect seems to be the opposite. Thus, C4 is lower than C3 by 2.8%, E4 is lower than E3 by 5.5%, H4 is higher than H3 by 4.6%, L4 is higher than L3 by 10.1%.

For the last three resins (matrix E, matrix H and matrix L) in **Table 5**-**Table 20**, the final intervals (fifth and sixth) are joined so that the same values of the corresponding slope are presented. The last interval shows almost the same value which confirms that the final response of the material is a fiber response. The higher value of the modulus of elasticity is reached for materials with H matrix, in the case of materials unmodified with NMP and non-heat-treated, but also for materials unmodified with NMP, but heat treated. Also, the higher value of the modulus of elasticity is reached for materials with L matrix, in the case of materials modified with NMP and not heat treated, but also for materials modified with NMP, but heat treated.
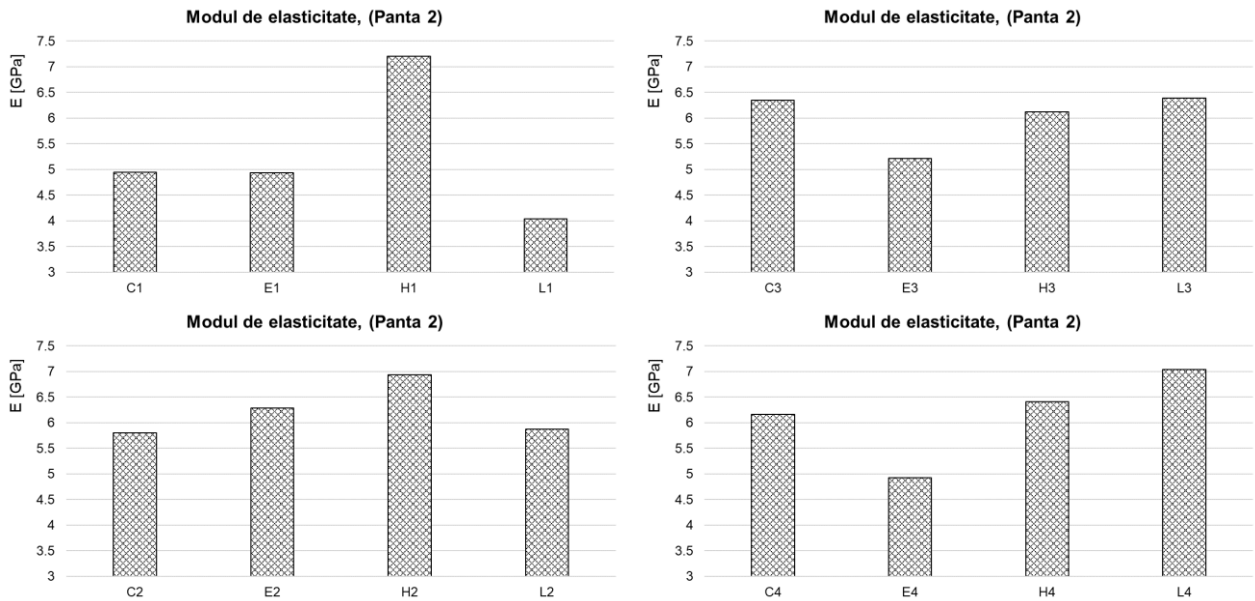
Fig. 169 The modulus of elasticity of pseudo-laminates

In the case of non-modified NMP pseudo-laminates and non-heat treated, it can be seen that from the point of view of modulus of elasticity, the best answer is presented by the material formed with type H resin, while the material formed with type L resin has the poorest answer. The H1 material has a higher modulus of elasticity than C1 by 45.5%, higher than E1 by 45.8% and higher than L1 by 78.1%.

In the case of pseudo-laminates not modified with NMP, but heat treated, it can be observed that from the point of view of the modulus of elasticity, the best answer is presented by all the material formed with type H resin, while the material formed with type C resin has the

weakest answer. The H2 material has a higher modulus of elasticity than C2 by 19.6%, higher than E2 by 10.4% and higher than L2 by 18.1%.

In the case of pseudo-laminates modified with NMP and not heat-treated, it can be seen that from the point of view of modulus of elasticity, the best answer is presented by the material formed with type L resin, followed extremely closely by the material formed with type C resin, while the material formed with type E resin shows the weakest response. The L3 material has a higher modulus of elasticity than C3 by 0.7%, higher than E3 by 22.7% and higher than H3 by 4.4%.

In the case of pseudo-laminates modified with NMP, but heat treated, it can be seen that from the point of view of modulus of elasticity, the best answer is presented by the material formed with type L resin, while the material formed with type E resin has the weaker response. The L4 material has a higher modulus of elasticity than C4 by 14.1%, higher than E4 by 43.0% and higher than H4 by 9.8%.

In the case of pseudo-laminates unmodified with NMP and not heat treated, it can be seen that from the point of view of the force at maximum load, the best answer is the material formed with type L resin, while the material formed with type H resin has the weaker response. Material L1 has a maximum loading force 6.4% higher than the pseudo-laminate with the weakest response, H1. Compared to other materials, L1 has a force 5.9% higher than C1, and 4.1% higher than E1.
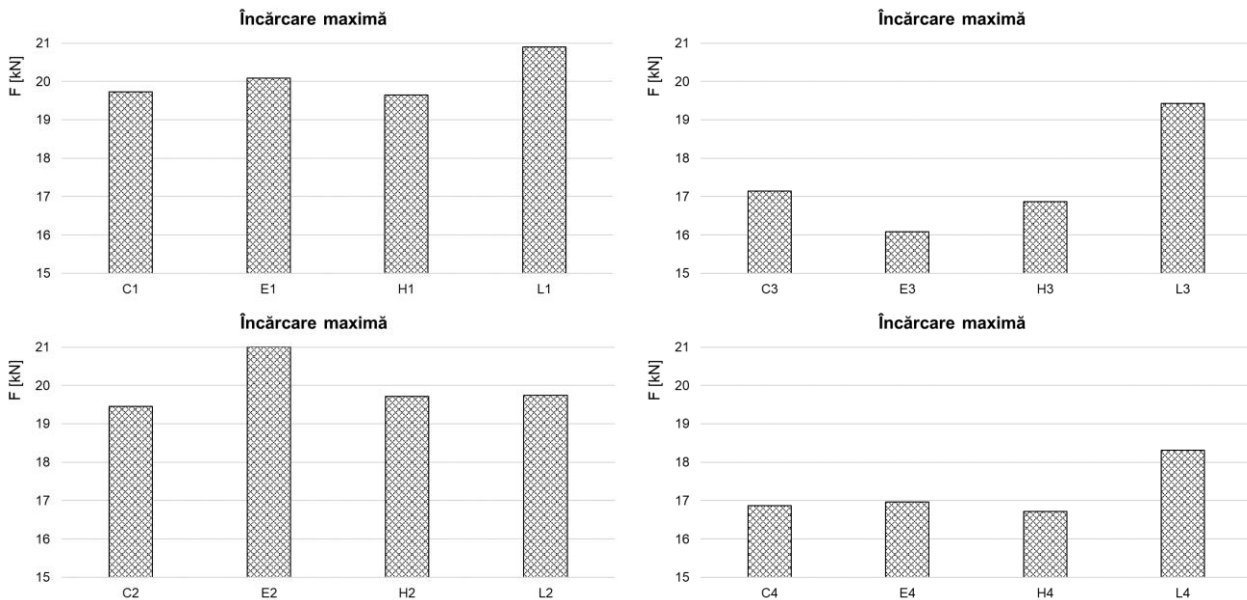
Fig. 170 Maximum load, comparative analysis

In the case of pseudo-laminates not modified with NMP, but heat treated, it can be seen that from the point of view of the force at maximum load, the best answer is the material formed with type E resin, while the material formed with type C resin has the weakest answer. Material E2 has a maximum loading force 7.9% higher than the pseudo-laminate with the weakest response, C2. Compared to other materials, E2 has a strength 6.5% higher than H2, and 6.4% higher than L2.

In the case of pseudo-laminates modified with NMP and naturally polymerized, it can be seen that from the point of view of the force at maximum load, the best answer is the material formed with type L resin, while the material formed with type E resin has the weaker response. The L3 material has a maximum loading force 20.7% higher than the pseudo-laminate with the weakest response, E3. Compared to other materials, L3 has a strength 15.1% higher than H3, and 13.3% higher than C3.

Table 22 Variation of the breaking temperature value from the environment.

Material	Average temperature of the selected specimens [°C]	Increase in temperature value		Material	Average temperature of the selected specimens [°C]	Increase in temperature value

C1	24.39		C1	24.39	
C3	21.53	-11.7%	C2	22.40	-8.1%
C2	22.40		C3	21.53	
C4	15.30	-31.7%	C4	15.30	-28.9%
E1	22.22		E1	22.22	
E3	21.14	-4.9%	E2	25.00	12.5%
E2	25.00		E3	21.14	
E4	23.78	-4.9%	E4	23.78	12.5%
H1	22.45		H1	22.45	
H3	17.76	-20.9%	H2	24.79	10.4%
H2	24.79		H3	17.76	
H4	19.01	-23.3%	H4	19.01	7.0%
L1	29.65		L1	29.65	
L3	25.80	-13.0%	L2	22.09	-25.5%
L2	22.09		L3	25.80	
L4	20.10	-9.0%	L4	20.10	-22.1%

In the case of pseudo-laminates modified with NMP, but heat treated, it can be seen that from the point of view of the force at maximum load, the best answer is the material formed with type L resin, while the material formed with type H resin has the weakest answer. The L4 material has a maximum loading force 9.5% higher than the pseudo-laminate with the weakest response, H4. Compared to other materials, L4 has a strength 8.5% higher than C4, and 7.9% higher than E4.

In general, it can be seen how the average temperature value is slightly higher in the case of heat-treated materials. Exceptions are materials C3 and C4, which have a small temperature difference, as well as L1 and L3, which have a higher average temperature value for the naturally cured material.

From previous studies, it is known that polymers H and L (epoxy resin H and epoxy resin L) are more rigid than the other two resins and an explanation could be found in this regard, which means that micro fractures of the matrix lead to local heating of materials, resulting in a higher temperature at the end.

From **Table 22**, it can be seen that the modifying of the matrix with the NMP solvent, led to a decrease in the value of the recorded temperature, which may be associated with a weaker mechanical response, in terms of tensile stress. This observation is valid both for the average temperature of the eight specimens and for the selected specimens. However, the hypothesis that we can corroborate the increase of the temperature value with some mechanical effects induced by the tensile stress, is not confirmed when analyzing the results presented in **Table 22**.

4.6.   Conclusions – tensile tests

In subchapters 4.1 - 4.4, images corresponding to the moment of rupture of the specimens (rupture of the outer package formed with the carbon fiber fabric) are presented, both front image, side image and an image in the infrared spectrum (the latter being included to highlight the temperature maximum obtained). Also, depending on the orientation of the thermal imaging camera, it was only possible to view high temperature areas that correspond mainly to the outer reinforcement layers (carbon fiber packages), for all studied materials. In the subchapters mentioned above, the values of the breaking temperature are presented for all the tested specimens, and these values should be interpreted as mechanical effects, following in a subsequent study to be compared with the values of the specific thermal capacity of the materials.

Also, prior to breaking the specimen, I observed acoustic emissions [249]–[251] that may be associated with the release of individual fibers from the matrix, for all tested materials. This method of non-destructive testing of the specimen may provide information on defects in the material, such as fracturing, delamination, rupture of fibers or peeling of the layers. This hypothesis was studied and certain frequencies emitted by a material subjected to a mechanical stress were correlated with a possible defect.

Regarding the heating of the specimens for fracturing, we could assume that the final value of the temperature is related both to the quality of the matrix-reinforcement interface and to the amount of energy released when the carbon fibers break.

Another important point in the characterization of the material is the thermographic analysis of the surface of the specimens, during the tensile stresses. This was done with the same equipment mentioned above, Fluke Ti400. Thus, analyzing the images from the infrared spectrum, small heated areas can be observed that appear before the fracture of the matrix. These areas may anticipate, to some extent, the location where the specimen will fracture, as these increases in the surface temperature of the material may represent either detachment of the fiber matrix (interface failure effect) or discharge areas of energy. The material has a plastic deformation, a deformation that will discharge all the energy into the environment, due to the fact that the material does not have an elastic component. Thus, when the discharge takes place in the environment, the first consequence is the local heating of the area where it fractured. This phenomenon is repeated for all materials tested in this study but requires a more detailed follow-up, possibly using a high-resolution thermal imaging camera.

Materials formed with type C, E and L resin but not modified with NMP, can be subjected to a heat treatment, as they lead to better results. While, only materials formed with H and L type resin modified with NMP, can be subjected to a heat treatment, as they lead to better results.

The L4 material, the one modified with NMP and heat treated, presents, from the point of view of the modulus of elasticity, the best response to tensile stress. It has the modulus of (calculated) elasticity higher than E4 by 43.0%, higher than C4 by 14.1%, and higher than H4 by 9.8%.

5. Analysis of mechanical properties of materials - bending tests (three points)

5.1. Mechanical properties of composite materials formed with type C resin

5.1.5. Conclusions pseudo-laminate C
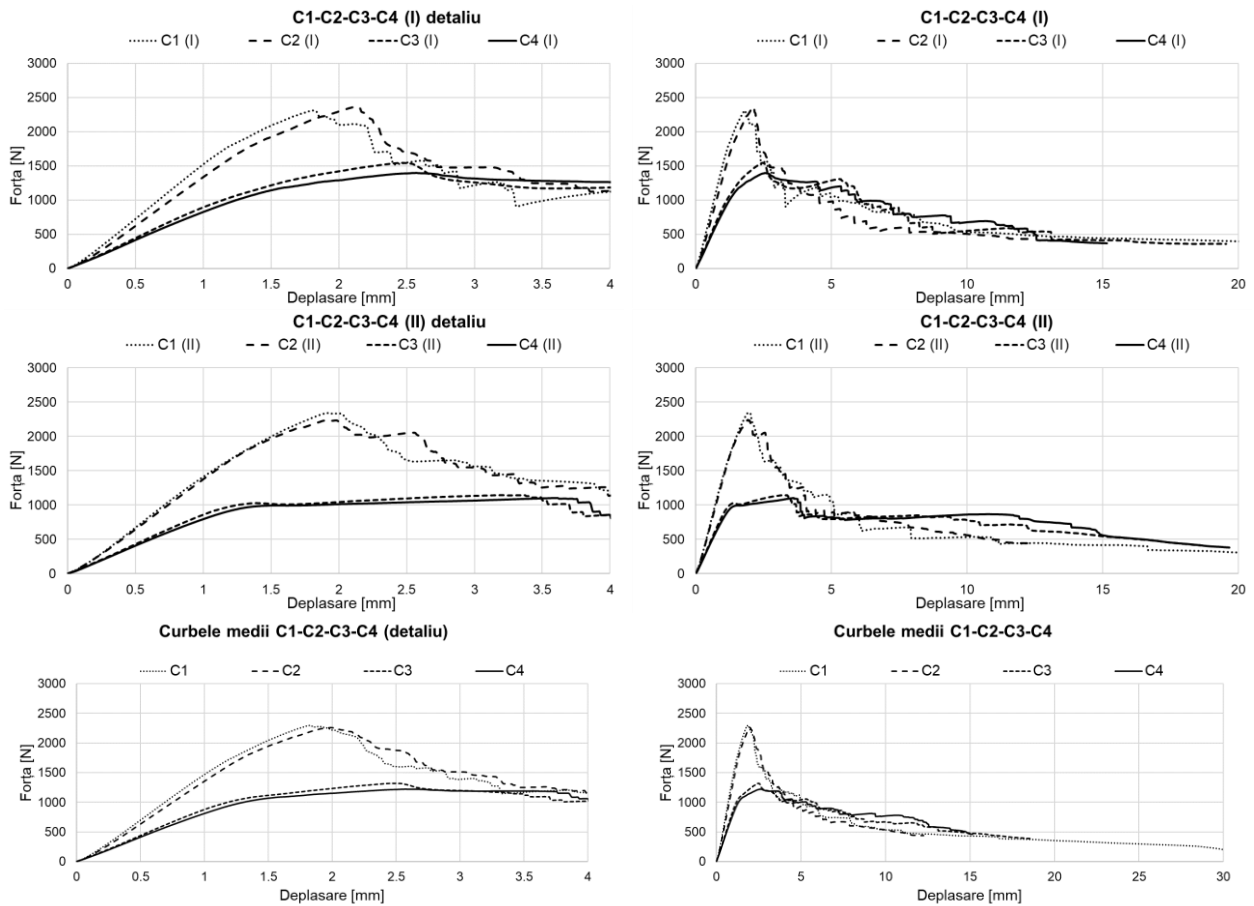
Fig. 221 Comparison between materials C1-C2-C3 și C4 on average bending curves

In the graph of the mean curves C1, C2, C3 and C4, the results obtained from the bending test are compared. Following a quantitative analysis, we can conclude that the heat treatment applied influences the performance of the composite in a negative way, but only to a small extent, although the heat treatment benefits the polymerization of the composite, in the sense that it uniformizes the bonds between layers and matrices.
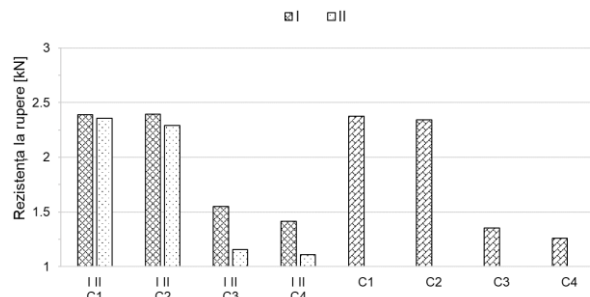
Fig. 222 Breaking stress for type C resin materials

In **Fig. 222**, it can be seen how the breaking stress has different values for the case when the force is applied on the surface I, compared to the case when the force is applied on

the surface II. Thus, only for naturally polymerized material, C1, the average breaking stress on surface I is 1.4% higher than the average on surface II. In the case of heat-treated material, C2, the average breaking stress on surface I is 4.4% higher than the average on surface II. For the material modified with NMP solvent and naturally polymerized, C3, the average breaking stress on surface I is 34.3% higher than the average on surface II. In the case of heat-treated material modified with NMP solvent, C4, the average breaking stress on surface I is 27.5% higher than the average on surface II.

For materials formed with type C resin (when it's considered the average on the entire laminate being composed of both the average on surface I and the average on surface II), it can be observed, from the point of view of breaking stress, that the heat treatment has negatively influenced the properties of materials. Thus, the average breaking stress for C1 pseudo-laminate is 1.3% higher than C2. For the NMP-modified material, the same behavior can be observed, namely that the solvent significantly reduced the breaking stress compared to the non-NMP-modified material. Regarding the heat treatment applied to the two pseudo-laminates modified with NMP, it can be seen that here too the mechanical properties were negatively influenced, the average breaking stress for pseudo-laminate C3 is higher than C4. Also, a comparison between the materials presented in the figure above, can also highlight the influence of the NMP solvent on the properties of the materials. Thus, the breaking stress for C1 pseudo-laminate is 75.3% higher than C3. In the case of material C2, there was a value of breaking stress 85.9% higher than C4.

Therefore, analyzing the average results of the breaking stress of **Fig. 222** of the materials formed with type C resin, we can observe that from this point of view, the best pseudo-laminate was the material that was not modified with NMP and was not heat treated, C1. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that following the bending tests (in three points), the best material was C3, i.e. the material formed with type C resin, modified with NMP solvent and which wasn't subjected to a heat treatment, but only naturally cured. Material C3 has a breaking stress value of 7.4% higher than material C4.
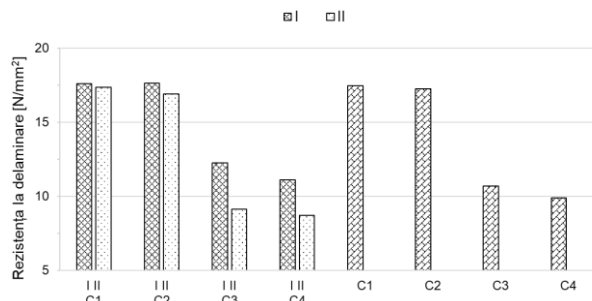
Fig. 223 Delamination resistance for type C resin materials

In **Fig. 223**, it can be seen how the resistance to delamination has different values for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. With regard to the average on surface I, compared to surface II, the same percentage values shall be retained as in the case of breaking stress.

For materials formed with type C resin (when we consider the average of the entire pseudo-laminate being composed of both the average on surface I and the average on surface II), it can be observed, from the point of view of resistance to delamination, that the thermal treatment negatively influenced the properties of the materials. Thus, the average delamination resistance for C1 pseudo-laminate is 1.1% higher than C2. For the NMP-modified material, the same behavior can be observed, namely that the solvent significantly reduced the delamination resistance compared to the non-NMP-modified material. Regarding the heat treatment applied to the two pseudo-laminates modified with NMP, it can be seen that here too the mechanical properties were negatively influenced, the average delamination resistance for pseudo-laminate C3 is higher than C4. Also, a comparison between the materials presented in the figure above, can also highlight the influence of the NMP solvent on the properties of the materials. Thus, the delamination resistance for C1 pseudo-laminate is 63.4% higher than C3. In the case of material C2, a delamination resistance value was recorded by 74.4% higher than C4.

Therefore, analyzing the average results of the delamination resistance in **Fig. 223** of the materials formed with type C resin, we can observe that from this point of view, the best pseudo-laminate was the material that was not modified with NMP and was not heat treated, C1, the result being similar to that of the breaking stress. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that following the bending tests (performed in three points), the best material was C3, i.e. the material formed with type C resin, modified with NMP solvent and which was not subjected to a heat treatment, but only naturally cured. Material C3 has a value of delamination resistance 8.0% higher than material C4.
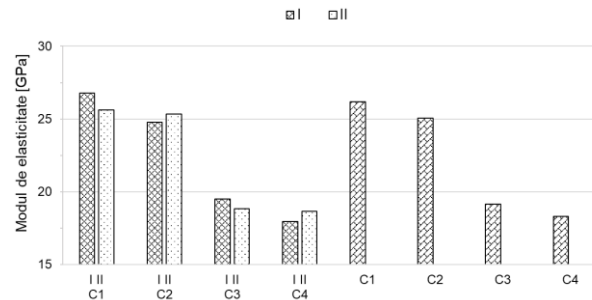

Fig. 224 Modulus of elasticity for materials formed with type C resin

In **Fig. 224**, it can be seen how the modulus of elasticity has different values for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. Thus, only for the naturally polymerized material, C1, the average modulus of elasticity on surface I is 4.5% higher than the average on surface II. In the case of heat-treated material, C2, the average modulus of elasticity on surface II is 2.2% higher than the average on surface I.

The influence of the heat treatment applied, in the case of the modulus of elasticity, seems to be reversed for the material C2 compared to C1. Thus, for the pseudo-laminate C1, a higher value of the modulus of elasticity on the surface I was registered, while, for the material C2, a higher value of the modulus of elasticity on the surface II was registered. It is known that heat treatment brings a benefit to the polymerization of the composite, in the sense that it uniformizes the bonds between layers and matrices, and we would have expected the modulus of elasticity to be higher in the case of heat-treated materials.

For the material modified with NMP solvent and naturally polymerized, C3, the average modulus of elasticity on surface I is 3.5% higher than the average on surface II. In the case of heat-treated material modified with NMP solvent, C4, the average modulus of elasticity on surface II is 4.0% higher than the average on surface I.

For materials formed with type C resin (when we consider the average on the entire laminate being composed of both the average on surface I and the average on surface II), it can be observed, from the point of view of the modulus of elasticity, that the heat treatment negatively influenced the properties of materials. Thus, the average modulus of elasticity for pseudo-laminate C1 is 4.5% higher than C2. For NMP-modified materials, the same behavior can be observed, namely that the solvent significantly reduced the modulus of elasticity compared to non-NMP-modified materials. Regarding the heat treatment applied to the two pseudo-laminates modified with NMP, it can be seen that here too the mechanical properties were negatively influenced, the average modulus of elasticity for the pseudo-laminate C3 is higher than C4. Also, a comparison between the materials presented in the figure above, can also highlight the influence of the NMP solvent on the properties of the materials. Thus, the average modulus of elasticity for C1 pseudo-laminate is 36.6% higher than C3. In the case of C2 material, a modulus value of 37.0% higher than C4 was recorded.

Therefore, analyzing the average results of the modulus of elasticity of **Fig. 224** of the materials formed with type C resin, we can observe that from this point of view, the best pseudo-laminate was the material that was not modified with NMP and was not heat treated, C1, the result being similar to that of breaking stress. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that following the bending tests (performed in three points), the best material was C3, i.e.

the material formed with type C resin, modified with NMP solvent and which was not subjected to a heat treatment, but only naturally cured. Material C3 has a modulus of elasticity 4.7% higher than material C4.

Tabelul 26 Variația valorii temperaturii, față de mediul ambiant, pentru C1

Epruveta / Eveniment			1	2	3	4	5	6
1	I / II	T [°C]	5.3 / 2.3	10.7 / 4.3	8.2 / 4.9	8.9 / 3.5	9.5 / 3.5	6.6 / 3.7
		t [s]	31.7 / 40.0	50.0 / 30.5	32.0 / 31.0	39.0 / 31.0	31.0 / 32.0	29.0 / 30.0
2	I / II	T [°C]	7.8 / 2.5	3.4 / 3.9	4.1 / 8.9	5.6 / 3.9	3.6 / 3.3	4.5 / 5.6
		t [s]	33.2 / 44.0	55.0 / 36.0	98.0 / 36.0	65.0 / 38.0	64.0 / 32.0	75.0 / 37.0
3	I / II	T [°C]	3.7 / 11.4	3.8 / 9.7	- / 5.5	5.2 / 9.5	3.7 / 4.0	4.0 / 5.6
		t [s]	43.3 / 67.1	138.0 / 63.0	- / 42.0	135.0 / 59.7	73.0 / 42.5	202.0 / 53.0
4	I / II	T [°C]	3.9 / -	- / -	- / -	- / -	- / 8.0	- / 8.9
		t [s]	77.1 / -	- / -	- / -	- / -	- / 56.0	- / 58.0
Temperatura mediului ambiant					20.3°C			
Delaminare axială			Rupere C240 strat inferior			Delaminare laterală		

Tabelul 30 Variația valorii temperaturii, față de mediul ambiant, pentru C3

Epruveta / Eveniment			1	2	3	4	5	6
1	I / II	T [°C]	4.9 / 2.4	0.9 / 5.4	5.4 / 8.1	1.2 / 3.9	3.1 / 6.9	1.4 / 4.6
		t [s]	70.0 / 45.8	70.0 / 44.9	74.5 / 47.3	78.0 / 48.6	77.0 / 44.9	65.0 / 47.0
2	I / II	T [°C]	-0.9 / 5.9	-2.2 / 5.4	0.3 / 4.6	-0.5 / -	-1.2 / -	3.4 / 3.1
		t [s]	84.5 / 47.8	81.0 / 46.2	92.5 / 48.8	160.0 / -	77.5 / -	67.0 / 47.9
3	I / II	T [°C]	- / -	-1.7 / 1.6	- / -	- / -	- / -	0.8 / -
		t [s]	- / -	102.0 / 106.5	- / -	- / -	- / -	97.0 / -
4	I / II	T [°C]	- / -	- / -	- / -	- / -	- / -	- / -
		t [s]	- / -	- / -	- / -	- / -	- / -	- / -
Temperatura mediului ambiant					19.8°C			
Delaminare axială			Rupere C240 strat inferior			Delaminare laterală		

Tabelul 28 Variația valorii temperaturii, față de mediul ambiant, pentru C2

Epruveta / Eveniment			1	2	3	4	5	6
1	I / II	T [°C]	3.9 / 1.23	3.3 / 7.6	- / 8.1	4.7 / 2.3	1.2 / 8.5	5.5 / 5.2
		t [s]	33.5 / 37.0	43.5 / 39.5	- / 37.5	32.0 / 37.0	38.0 / 35.5	46.0 / 38.5
2	I / II	T [°C]	0.3 / 8.8	3.5 / 2.0	- / 1.9	1.1 / 2.7	11.3 / 4.6	2.0 / 3.2
		t [s]	87.5 / 51.5	45.5 / 55.0	- / 54.5	54.0 / 45.0	50.5 / 37.5	69.0 / 52.5
3	I / II	T [°C]	1.6 / 2.2	0.1 / -	- / -	2.1 / 13.1	1.3 / 3.4	- / -
		t [s]	101.0 / 63.0	70.5 / -	- / -	80.5 / 66.0	60.0 / 57.5	- / -
4	I / II	T [°C]	- / -	0.7 / -	- / -	- / -	- / 2.6	- / -
		t [s]	- / -	75.5 / -	- / -	- / -	- / 75.5	- / -
Temperatura mediului ambiant					16.4°C			
Delaminare axială			Rupere C240 strat inferior			Delaminare laterală		

Tabelul 32 Variația valorii temperaturii, față de mediul ambiant, pentru C4

Epruveta / Eveniment			1	2	3	4	5	6
1	I / II	T [°C]	6.9 / 4.0	0.5 / 6.2	0.5 / 4.1	3.5 / 9.1	1.2 / 6.7	0.9 / 6.2
		t [s]	86.0 / 52.0	39.0 / 50.0	56.5 / 47.5	90.0 / 49.0	57.0 / 50.5	71.0 / 47.0
2	I / II	T [°C]	0.6 / 3.0	1.3 / 2.5	1.4 / 7.1	5.4 / -	0.8 / -	1.9 / 7.9
		t [s]	133.0 / 178.0	116.0 / 173.0	73.0 / 48.5	152.0 / -	67.0 / -	89.0 / 50.0
3	I / II	T [°C]	5.7 / -	- / -	2.5 / -	- / -	4.9 / -	7.7 / 4.3
		t [s]	152.0 / -	- / -	143.5 / -	- / -	113.0 / -	142.0 / 146.6
4	I / II	T [°C]	- / -	- / -	- / -	- / -	5.3 / -	7.7 / -
		t [s]	- / -	- / -	- / -	- / -	119.0 / -	145.0 / -
Temperatura mediului ambiant					19.8°C			
Delaminare axială			Rupere C240 strat inferior			Delaminare laterală		

Fig. 225 The variation of the temperature value, compared to the environment, for C1, C2, C3, C4

In **Fig. 225**, it can be observed how the variation of the temperature value presents different values as well as different behavior, for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. This method of presentation is very easy because the increases in temperature values can be immediately correlated with the identified mechanical effects (axial delamination, lateral delamination, relative slip of the layers, fractures of the layers).

5.2. Mechanical properties of composite materials formed with type E resin

5.2.5. Conclusions pseudo-laminate E

In the graph of the mean curves E1, E2, E3 and E4, the results obtained from the bending test are compared. Following a quantitative analysis, we can conclude that the heat treatment applied influences the performance of the composite in a negative way, but only to a small extent, although the heat treatment benefits the polymerization of the composite, in the sense that it uniformizes the bonds between layers and matrices.
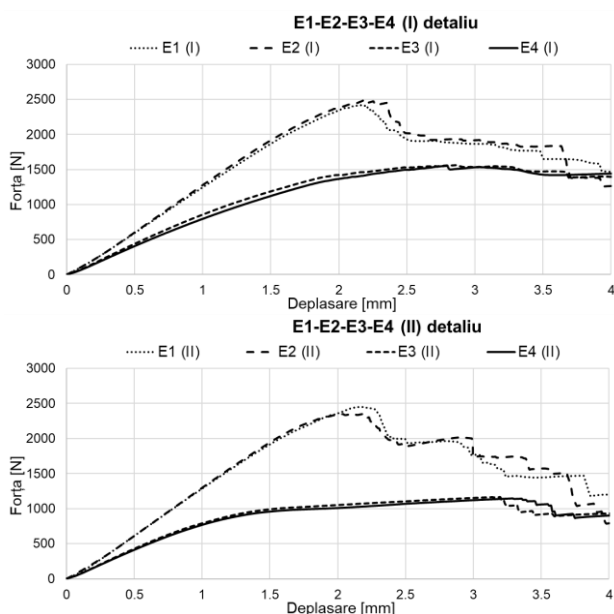
Fig. 276 Comparison between materials E1-E2-E3 și E4 on average bending curves

Fig. 277 Breaking stress for type E resin materials

In **Fig. 277**, it can be seen how the breaking stress has different values for the case when the force is applied on the surface I, compared to the ca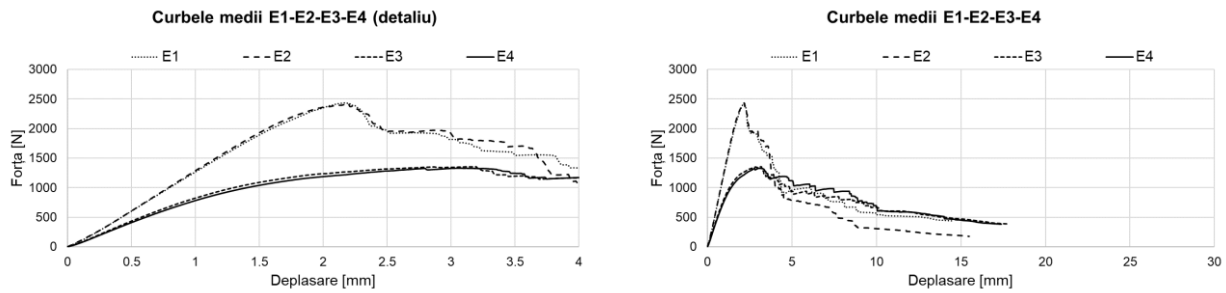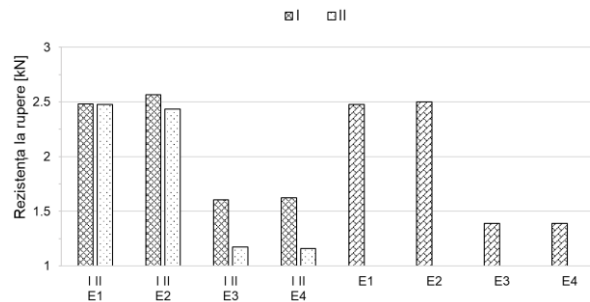se when the force is applied on the surface II. Thus, for the only naturally polymerized material, E1, the average breaking stress on surface I is 0.2% higher than the average on surface II. In the case of heat-treated material, E2, the average breaking stress on surface I is 5.4% higher than the average on surface II. For the material modified with NMP solvent and naturally polymerized, E3, the average breaking stress on surface I is 36.6% higher than the average on surface II. In the case of heat-treated material modified with NMP solvent, E4, the average breaking stress on surface I is 40.0% higher than the average on surface II.

For materials formed with type E resin (when we consider the average on the entire laminate being composed of both the average on surface I and the average on surface II), it can be observed, from the point of view of breaking stress, that the heat treatment has positively influenced the properties of materials, but only to a small extent. Thus, the average breaking strength for pseudo-laminate E2 is 0.9% higher than E1. For NMP-modified materials, the same behavior can be observed, namely that the solvent has significantly reduced the breaking stress compared to non-NMP-modified materials. Regarding the heat treatment applied to the two pseudo-laminates modified with NMP, it cannot be observed that the mechanical properties were influenced, the average breaking stress for the pseudo-laminate E3 being approximately equal to E4. Also, a comparison between the materials presented in the figure above, can also highlight the influence of the NMP solvent on the properties of the materials. Thus, the breaking stress for pseudo-laminate E1 is 78.4% higher than E3. In the case of E2 material, a higher value of breaking stress was registered, the average being 79.9% higher than E4.

Therefore, analyzing the average results of the breaking stress of **Fig. 277** of the materials formed with type E resin, we can observe that from this point of view, the best pseudo-laminate was the material that was not modified with NMP but was heat treated, E2. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, I can say that following the bending tests (performed in three points), the best material was E4, i.e. the material formed with type E resin, modified with NMP solvent and subjected to heat treatment. Material E4 has a value of breaking stress 0.1% higher than material E3, the difference being insignificant.

In **Fig. 278**, it can be seen how the resistance to delamination has different values for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. With regard to the average on surface I, compared to surface II, the same percentage values are kept as in the case of breaking strength.
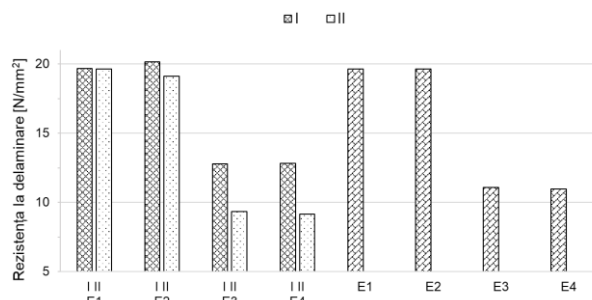
Fig. 278 Delamination strength for type E resin materials

For materials formed with type E resin (when we consider the average on the entire laminate being composed of both the average on surface I and the average on surface II), it can be observed, from the point of view of delamination resistance, that the heat treatment has negatively influenced the properties of materials. Thus, the average delamination resistance for pseudo-laminate E1 is 0.05% higher than E2. For NMP-modified materials, the same behavior can be observed, namely that the solvent significantly reduced the delamination resistance compared to non-NMP-modified materials. Regarding the heat treatment applied to the two pseudo-laminates modified with NMP, it can be seen that here too the mechanical properties were negatively influenced, the average delamination resistance for pseudo-laminate E3 is 0.7% higher than E4. Also, a comparison between the materials presented in the figure above, can also highlight the influence of the NMP solvent on the properties of the materials. Thus, the delamination resistance for pseudo-laminate E1 is 77.6% higher than E3. In the case of E2 material, a higher value of delamination resistance was registered, the average being 78.8% higher than E4.

Therefore, analyzing the average results of the delamination resistance in **Fig. 278** of the materials formed with type E resin, we can observe that from this point of view, the best pseudo-laminate was the material that was not modified with NMP and was not heat treated, E1. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that following the bending tests, the best material was E3, i.e. the material formed with type E resin, modified with the NMP solvent and which was not subjected to a heat treatment, but only was naturally polymerized. Material E3 has a delamination resistance value 0.7% higher than material E4, but the difference is not significant.
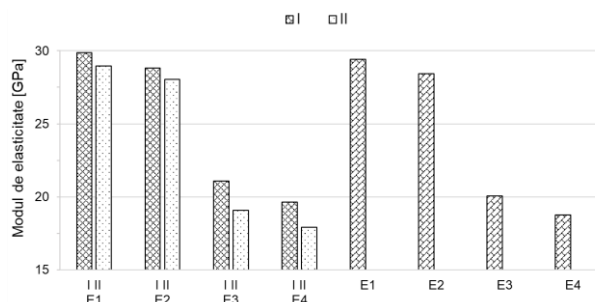
Fig. 279 Elastic modulus for materials formed with type E resin

In **Fig. 279**, it can be seen how the modulus of elasticity has different values for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. Thus, the average area I for material E1 is 3.2% higher than the average area II. In the case of material E2, a higher value of the modulus of elasticity on surface I was registered, being 2.8% higher than the average on surface II. The average area I for material E3 is 10.6% higher than the average area II. In the case of material E4, a higher value of the modulus of elasticity on surface II was registered, being 9.7% higher than the average on surface I.

The influence of the heat treatment applied, in the case of the modulus of elasticity, seems to be reversed for the material E2 compared to E1. Thus, for the pseudo-laminate E1, a higher value of the modulus of elasticity on the surface I was registered, as well as in the case

of the material E2. It is known that heat treatment brings a benefit to the polymerization of the composite, in the sense that it uniformizes the bonds between layers and matrices, and, we would have expected the modulus of elasticity to be higher in the case of heat-treated materials.

For the material modified with NMP solvent and naturally polymerized, E3, the average modulus of elasticity on surface I is 10.6% higher than the average on surface II. In the case of heat-treated material modified with NMP solvent, C4, the average modulus of elasticity on surface I is 9.7% higher than the average on surface II.

For materials formed with type E resin (when we consider the average on the entire laminate being composed of both the average on surface I and the average on surface II), it can be observed, from the point of view of the modulus of elasticity, that the heat treatment has negatively influenced the properties of materials. Thus, the average modulus of elasticity for pseudo-laminate E1 is 3.5% higher than E2. For NMP-modified materials, the same behavior can be observed, namely that the solvent significantly reduced the modulus of elasticity compared to non-NMP-modified materials. Regarding the heat treatment applied to the two pseudo-laminates modified with NMP, it can be seen that here too the mechanical properties were negatively influenced, the average modulus of elasticity for the pseudo-laminate E3 is 6.9% higher than E4. Also, a comparison between the materials presented in the figure above, can also highlight the influence of the NMP solvent on the properties of the materials. Thus, the average modulus of elasticity for pseudo-laminate E1 is 46.6% higher than E3. In the case of E2 material, a higher value of the modulus of elasticity was registered, the average being higher by 51.5% compared to E4.

Therefore, analyzing the average results of the modulus of elasticity of **Fig. 279** of the materials formed with type C resin, we can observe that from this point of view, the best pseudo-laminate was the material that was not modified with NMP and was not heat treated, E1, the result being similar to that of the delamination resistance. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that following the bending tests (in three points performed), the best material was E3, i.e. the material formed with type E resin, modified with the NMP solvent and which was not subjected to a heat treatment, but only was naturally polymerized. Material E3 has a modulus of elasticity 6.9% higher than material E4.

Tabelul 34 Variația valorii temperaturii, față de mediul ambient, pentru E1

Eveniment	I/II		1	2	3	4	5	6
1	I/II	T [°C]	6.8 / 2.6	10.1 / 9.7	11.7 / 2.8	6.1 / 9.5	9.0 / 7.8	9.9 / 7.2
		t [s]	30.0 / 33.1	32.0 / 31.6	32.5 / 32.1	33.0 / 32.4	30.6 / 32.0	31.0 / 31.1
2	I/II	T [°C]	12.0 / 4.1	4.7 / 1.2	4.0 / 4.7	4.0 / 2.0	4.1 / 1.5	4.5 / 2.6
		t [s]	32.0 / 34.5	110.0 / 41.0	86.0 / 34.1	56.0 / 57.5	51.0 / 56.0	55.0 / 38.3
3	I/II	T [°C]	4.8 / -0.1	8.4 / 1.6	- / 0.9	- / -	- / -	5.5 / 2.4
		t [s]	101.5 / 42.5	124.0 / 50.0	- / 55.1	- / -	- / -	77.1 / 48.5
4	I/II	T [°C]	- / 1.4	- / -	- / -	- / -	- / -	- / -
		t [s]	- / 56.1	- / -	- / -	- / -	- / -	- / -
Temperatura mediului ambient			19.1°C					

Delaminare axială | Rupere C_{240} strat inferior | Delaminare laterală

Tabelul 36 Variația valorii temperaturii, față de mediul ambient, pentru E2

Eveniment	I/II		1	2	3	4	5	6
1	I/II	T [°C]	7.5 / 5.9	6.6 / 8.5	9.2 / 6.6	8.7 / 6.8	8.9 / 8.7	- / 9.6
		t [s]	33.0 / 33.1	29.1 / 30.2	31.1 / 32.1	31.0 / 27.5	31.3 / 28.5	- / 29.5
2	I/II	T [°C]	3.4 / 6.4	6.2 / 4.4	8.2 / 4.2	3.7 / 10.0	9.6 / 3.2	- / 12.1
		t [s]	52.0 / 51.1	50.0 / 38.4	54.5 / 43.9	93.4 / 47.1	46.2 / 46.7	- / 47.4
3	I/II	T [°C]	- / -	- / -	- / 3.2	3.1 / -	- / 3.6	- / -
		t [s]	- / -	- / -	- / 50.0	105.2 / -	- / -	- / -
4	I/II	T [°C]	- / -	- / -	- / -	- / -	- / -	- / -
		t [s]	- / -	- / -	- / -	- / -	- / -	- / -
Temperatura mediului ambient			19.8°C					

Delaminare axială | Rupere C_{240} strat inferior | Delaminare laterală

Tabelul 38 Variația valorii temperaturii, față de mediul ambient, pentru E3

Eveniment	I/II		1	2	3	4	5	6
1	I/II	T [°C]	3.1 / 11.5	3.2 / 12.2	2.9 / 10.4	3.7 / 11.1	4.1 / 15.4	4.5 / 12.5
		t [s]	80.0 / 40.0	52.0 / 42.1	46.2 / 42.1	52.0 / 39.2	63.0 / 42.5	72.0 / 45.0
2	I/II	T [°C]	3.9 / 7.0	3.8 / 6.6	3.1 / 6.0	4.0 / 6.0	4.0 / 6.7	4.5 / 8.0
		t [s]	96.0 / 105.0	56.0 / 107.1	60.2 / 109.1	61.0 / 108.1	78.4 / 109.1	85.0 / 118.1
3	I/II	T [°C]	- / -	8.5 / -	4.2 / -	8.9 / 7.1	- / 7.1	- / -
		t [s]	- / -	141.5 / -	175.0 / -	149.0 / 117.0	- / 123.0	- / -
4	I/II	T [°C]	- / -	- / -	- / -	- / -	- / -	- / -
		t [s]	- / -	- / -	- / -	- / -	- / -	- / -
Temperatura mediului ambient			13.9°C					

Delaminare axială | Rupere C_{240} strat inferior | Delaminare laterală

Tabelul 40 Variația valorii temperaturii, față de mediul ambient, pentru E4

Eveniment	I/II		1	2	3	4	5	6
1	I/II	T [°C]	9.3 / 17.5	6.5 / 13.3	6.5 / 13.0	7.6 / 13.1	6.5 / 11.3	7.1 / 12.3
		t [s]	107.2 / 45.6	61.1 / 45.1	63.2 / 43.1	63.0 / 44.0	104.7 / 42.3	92.0 / 47.1
2	I/II	T [°C]	6.3 / -	6.0 / 9.4	6.5 / -	6.6 / 9.7	6.4 / 9.4	6.9 / 8.4
		t [s]	171.2 / -	76.7 / 107.1	79.1 / -	78.7 / 108.2	123.1 / 112.1	103.0 / 120.0
3	I/II	T [°C]	- / -	- / -	- / -	10.0 / -	7.6 / -	- / -
		t [s]	- / -	- / -	- / -	118.4 / -	222.1 / -	- / -
4	I/II	T [°C]	- / -	- / -	- / -	- / -	- / -	- / -
		t [s]	- / -	- / -	- / -	- / -	- / -	- / -
Temperatura mediului ambient			13.9°C					

Delaminare axială | Rupere C_{240} strat inferior | Delaminare laterală

Fig. 280 The variation of the temperature value, compared to the environment, for E1, E2, E3, E4

In **Fig. 280**, it can be observed how the variation of the temperature value presents different values as well as different behavior, for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. This method of presentation is a very easy one because the increases in temperature values can be immediately correlated with the identified mechanical effects (axial delamination, lateral delamination or layer fractures).

5.3. Mechanical properties of composite materials formed with type H resin

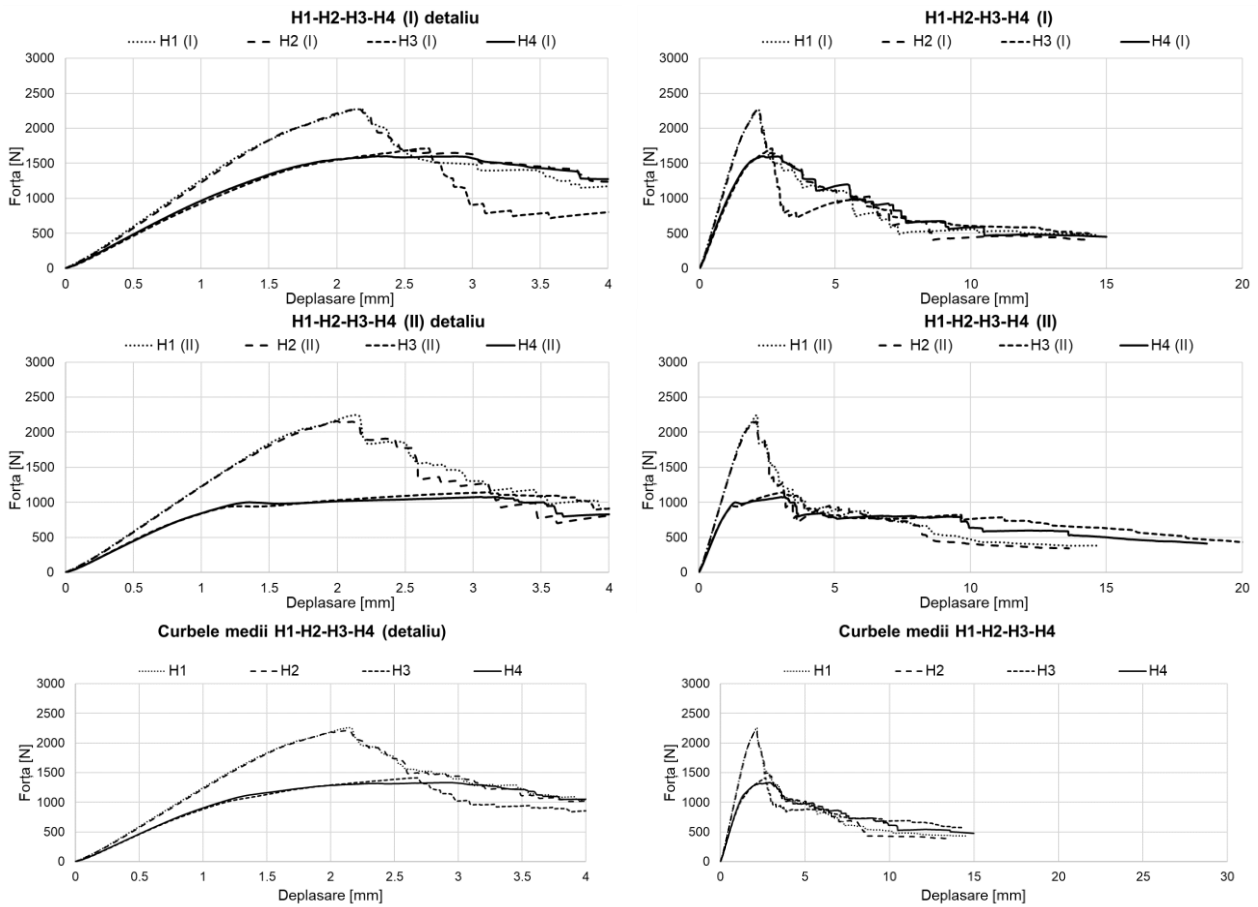### 5.3.5. Conclusion pseudo-laminate H

Fig. 331 Comparison between materials H1-H2-H3 și H4 on average bending curves

In the graph of the mean curves H1, H2, H3 and H4, the results obtained after the bending test are compared. Following a quantitative analysis, we can conclude that the heat treatment applied influences the performance of the composite in a negative way, but only to a small extent, although the heat treatment benefits the polymerization of the composite, in the sense that it uniformizes the bonds between layers and matrices.
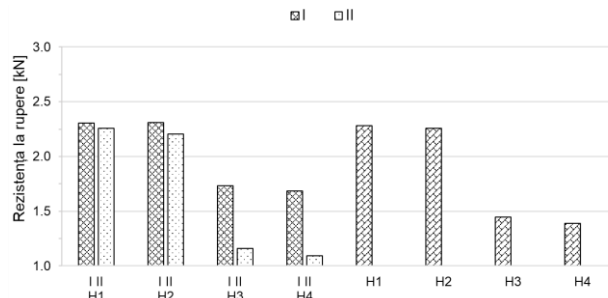
Fig. 332 Breaking stress for H-type resin-formed materials

In **Fig. 332**, it can be seen how the breaking stress has different values for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. Thus, for the only naturally polymerized material, H1, the average breaking stress on surface I is 2.2% higher than the average on surface II. In the case of heat-treated material, H2, the average breaking stress on surface I is 5.0% higher than the average on surface II. For the material modified with NMP solvent and naturally polymerized, H3, the average breaking

stress on surface I is 49.3% higher than the average on surface II. In the case of heat-treated material modified with the solvent NMP, H4, the average breaking stress on surface I is 54.6% higher than the average on surface II.

For materials formed with type H resin (when we consider the average on the entire laminate being composed of both the average on surface I and the average on surface II), it can be observed, from the point of view of breaking stress, that the heat treatment has negatively influenced the properties of materials, but only to a small extent. Thus, the average breaking stress for H1 pseudo-laminate is 1.0% higher than H2. For NMP-modified materials, the same behavior can be observed, namely that the solvent has significantly reduced the breaking stress compared to non-NMP-modified materials. Regarding the heat treatment applied to the two pseudo-laminates modified with NMP, it can be seen that the mechanical properties were negatively influenced, the average breaking stress for pseudo-laminate H3 is 4.1% higher than H4. Also, a comparison between the materials presented in the figure above, can also highlight the influence of the NMP solvent on the properties of the materials. Thus, the breaking stress for H1 pseudo-laminate is 57.8% higher than H3. In the case of H2 material, a higher value of breaking stress was registered, the average being higher by 62.6% compared to H4.

Therefore, analyzing the average results of the breaking stress of **Fig. 332** of the materials formed with H-type resin, we can see that from this point of view, the best pseudo-laminate was the one that was not modified with NMP and was not heat treated, H1. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that following the bending tests (performed in three points), the best material was H3, i.e. the material formed with type H resin, modified with the NMP solvent and which was not subjected to a heat treatment, but only was naturally polymerized. Material H3 has a breaking stress value 4.1% higher than material H4.
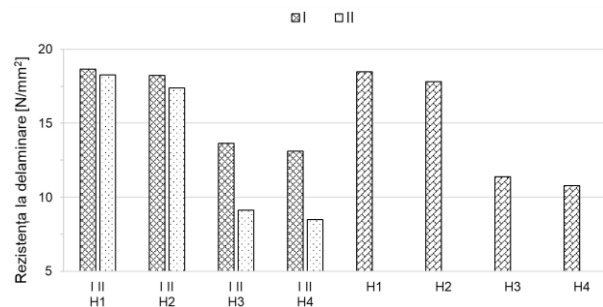

Fig. 333 Delamination resistance for H-type resin-formed materials

In **Fig. 333**, it can be seen how the resistance to delamination has different values for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. With regard to the average on surface I, compared to surface II, the same percentage values are kept as in the case of breaking strength.

For materials formed with type H resin (when we consider the average of the entire laminate being composed of both the average on surface I and the average on surface II), it can be observed, from the point of view of resistance to delamination, that the heat treatment has negatively influenced the properties of materials. Thus, the average delamination resistance for pseudo-laminate H1 is 3.6% higher than H2. For NMP-modified materials, the same behavior can be observed, namely that the solvent significantly reduced the delamination resistance compared to non-NMP-modified materials. Regarding the heat treatment applied to the two pseudo-laminates modified with NMP, it can be seen that here too the mechanical properties were negatively influenced, the average delamination resistance for the H3 pseudo-laminate is 5.5% higher than H4. Also, a comparison between the materials presented in the figure above, can also highlight the influence of the NMP solvent on the properties of the materials. Thus, the delamination resistance for H1 pseudo-laminate is 62.1% higher than H3. In the case of H2 material, a higher value of delamination resistance was registered, the average being 65.1% higher than H4.

Therefore, analyzing the average results of the delamination resistance in **Fig. 333** of the materials formed with type H resin, we can observe that from this point of view, the best pseudo-laminate was the material that was not modified with NMP and was not heat treated,

H1, the result being similar to that of to breaking stress. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that following the bending tests, the best material was H3, i.e. the material formed with type H resin, modified with the NMP solvent and which was not subjected to a heat treatment, but only was naturally polymerized. Material H3 has a delamination resistance value 5.5% higher than material H4.
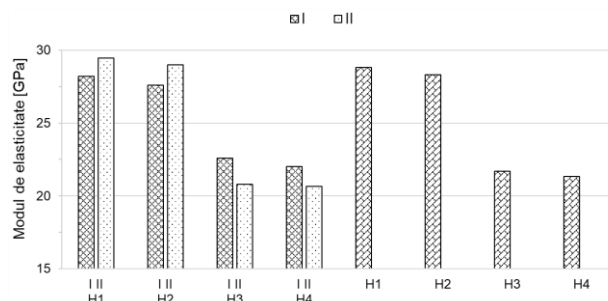

Fig. 334 Modulus of elasticity for materials formed with type H resin

In **Fig. 334**, it can be seen how the modulus of elasticity has different values for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. Thus, the average on surface II for material H1 is higher by 4.4% compared to the average on surface I. In the case of material H2, a higher value of the modulus of elasticity was also registered on surface II, being higher by 5.1% compared to of the average on surface I. The average on surface I for material H3, is 8.6% higher than the average on surface II. In the case of material H4, a higher value of the modulus of elasticity on surface I was registered, being higher by 6.6% compared to the average on surface II.

The influence of the heat treatment applied, in the case of the modulus of elasticity, seems to be reversed for the material H2 compared to H1. Thus, for the pseudo-laminates H1 and H2, a higher value of the modulus of elasticity was registered on the surface I, the average being higher for the non-heat-treated material. The same observation is valid for the comparison on surface II of the two materials. It is known that heat treatment brings a benefit to the polymerization of the composite, in the sense that it uniformizes the bonds between the layers and the matrix, and, we would have expected the modulus of elasticity to be higher in the case of heat-treated materials.

For materials formed with type H resin (when we consider the average on the entire laminate being composed of both the average on surface I and the average on surface II), it can be observed, from the point of view of the modulus of elasticity, that the heat treatment has negatively influenced the properties of materials. Thus, the average modulus of elasticity for H1 pseudo-laminate is 1.9% higher than H2. For NMP-modified materials, the same behavior can be observed, namely that the solvent significantly reduced the modulus of elasticity compared to non-NMP-modified materials. Regarding the heat treatment applied to the two pseudo-laminates modified with NMP, it can be seen that here too the mechanical properties were negatively influenced, the average modulus of elasticity for the pseudo-laminate H3 is 1.6% higher than H4. Also, a comparison between the materials presented in the figure above, can also highlight the influence of the NMP solvent on the properties of the materials. Thus, the average modulus of elasticity for H1 pseudo-laminate is 33.0% higher than H3. In the case of H2 material, a higher value of the modulus of elasticity was registered, the average being higher by 32.6% compared to H4.

Therefore, analyzing the average results of the modulus of elasticity of **Fig. 334** of the materials formed with type H resin, we can observe that from this point of view, the best pseudo-laminate was the material that was not modified with NMP and was not heat treated, H1, the result being similar to that of to breaking strength. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that following the bending tests (in three points performed), the best material was H3, i.e. the material formed with type H resin, modified with the NMP solvent and which was not subjected to a heat treatment, but only was naturally polymerized. The H3 material has a modulus of elasticity 1.6% higher than the H4 material.

In **Fig. 335**, it can be observed how the variation of the temperature value presents different values as well as different behavior, for the case when the force is applied on the

surface I, compared to the case when the force is applied on the surface II. This method of presentation is a very easy one because the increases in temperature values can be immediately correlated with the identified mechanical effects (axial delamination, lateral delamination or layer fractures).

Tabelul 42 Variația valorii temperaturii, față de mediul ambiant, pentru H1

Epruveta Eveniment		1	2	3	4	5	6
1	I / II	T [°C] 11.6 / 1.1	8.9 / 6.3	8.2 / 10.1	9.1 / 8.1	3.8 / 6.5	7.7 / 6.4
		t [s] 49.1 / 34.0	32.5 / 35.1	46.1 / 34.8	32.3 / 33.3	39.0 / 34.0	48.0 / 34.1
2	I / II	T [°C] 3.2 / 8.7	4.7 / 1.9	6.4 / 2.1	3.6 / 6.7	6.3 / 9.0	3.4 / 2.4
		t [s] 92.2 / 43.2	72.7 / 40.0	83.2 / 40.0	88.1 / 38.1	45.2 / 35.3	71.5 / 37.0
3	I / II	T [°C] - / 1.6	3.5 / 2.2	- / 2.4	- / 2.6	4.7 / 2.3	- / 2.4
		t [s] - / 66.1	126.6 / 59.1	0.0 / 47.1	0.0 / 52.0	70.1 / 40.3	0.0 / 45.0
4	I / II	T [°C] - / -	- / -	- / -	- / -	- / 2.5	- / -
		t [s] - / -	- / -	- / -	- / -	- / 50.2	- / -
Temperatura mediului ambiant			19.1°C				
Delaminare axială			Rupere C240 strat inferior		Delaminare laterală		

Tabelul 46 Variația valorii temperaturii, față de mediul ambiant, pentru H3

Epruveta Eveniment		1	2	3	4	5	6
1	I / II	T [°C] 4.1 / 10.9	5.1 / 11.8	4.4 / 10.7	5.2 / 10.9	5.1 / 16.6	5.0 / 10.2
		t [s] 78.0 / 50.0	37.0 / 51.0	39.0 / 49.0	37.1 / 41.3	39.3 / 49.0	39.1 / 47.2
2	I / II	T [°C] 4.4 / 10.1	8.9 / -	5.2 / -	5.2 / -	4.7 / -	4.9 / -
		t [s] 89.0 / 56.0	86.8 / -	41.1 / -	43.3 / -	40.7 / -	46.1 / -
3	I / II	T [°C] 8.4 / -	- / -	7.3 / -	8.9 / -	10.2 / -	8.6 / -
		t [s] 157.1 / -	- / -	97.1 / -	95.0 / -	168.0 / -	94.5 / -
4	I / II	T [°C] - / -	- / -	- / -	- / -	- / -	- / -
		t [s] - / -	- / -	- / -	- / -	- / -	- / -
Temperatura mediului ambiant			13.9°C				
Delaminare axială			Rupere C240 strat inferior		Delaminare laterală		

Tabelul 44 Variația valorii temperaturii, față de mediul ambiant, pentru H2

Epruveta Eveniment		1	2	3	4	5	6
1	I / II	T [°C] 5.3 / 9.3	3.9 / 2.9	7.8 / 12.5	8.2 / 3.4	6.8 / 4.2	11.5 / 7.7
		t [s] 35.3 / 34.1	32.0 / 34.2	31.3 / 34.1	29.0 / 34.0	31.0 / 31.8	29.4 / 33.0
2	I / II	T [°C] 2.1 / 2.9	1.9 / 3.3	2.0 / 3.2	2.8 / 3.2	2.5 / 3.8	3.6 / 6.6
		t [s] 87.0 / 37.0	64.0 / 41.3	48.0 / 38.1	47.0 / 40.0	39.1 / 40.9	80.5 / 43.6
3	I / II	T [°C] 5.6 / 3.0	2.2 / 8.3	3.0 / 3.5	3.0 / 12.4	4.3 / 13.0	4.2 / -
		t [s] 103.1 / 45.1	87.1 / 62.0	79.2 / 46.4	77.0 / 69.0	63.1 / 64.2	105.0 / -
4	I / II	T [°C] - / -	- / -	- / -	- / -	- / -	- / -
		t [s] - / -	- / -	- / -	- / -	- / -	- / -
Temperatura mediului ambiant			16.4°C				
Delaminare axială			Rupere C240 strat inferior		Delaminare laterală		

Tabelul 48 Variația valorii temperaturii, față de mediul ambiant, pentru H4

Epruveta Eveniment		1	2	3	4	5	6
1	I / II	T [°C] 1.3 / 6.6	1.8 / 8.7	1.5 / 9.5	8.4 / 10.1	9.0 / 9.2	2.2 / 6.6
		t [s] 113.2 / 48.1	55.0 / 47.1	92.0 / 46.2	73.1 / 46.1	69.0 / 42.9	48.0 / 41.1
2	I / II	T [°C] 2.5 / 4.2	3.1 / 4.2	- / 4.1	3.3 / 4.6	2.2 / 6.9	2.2 / 7.8
		t [s] 130.1 / 88.2	70.2 / 63.3	- / 121.0	94.0 / 167.9	89.0 / 128.2	54.0 / 45.0
3	I / II	T [°C] - / -	2.2 / -	- / -	- / -	- / -	7.6 / 5.7
		t [s] - / -	114.0 / -	- / -	- / -	- / -	86.0 / 122.1
4	I / II	T [°C] - / -	- / -	- / -	- / -	- / -	- / -
		t [s] - / -	- / -	- / -	- / -	- / -	- / -
Temperatura mediului ambiant			13.9°C				
Delaminare axială			Rupere C240 strat inferior		Delaminare laterală		

Fig. 335 The variation of the temperature value, compared to the environment, for H1, H2, H3, H4

5.4.   Mechanical properties of composite materials formed with type L resin

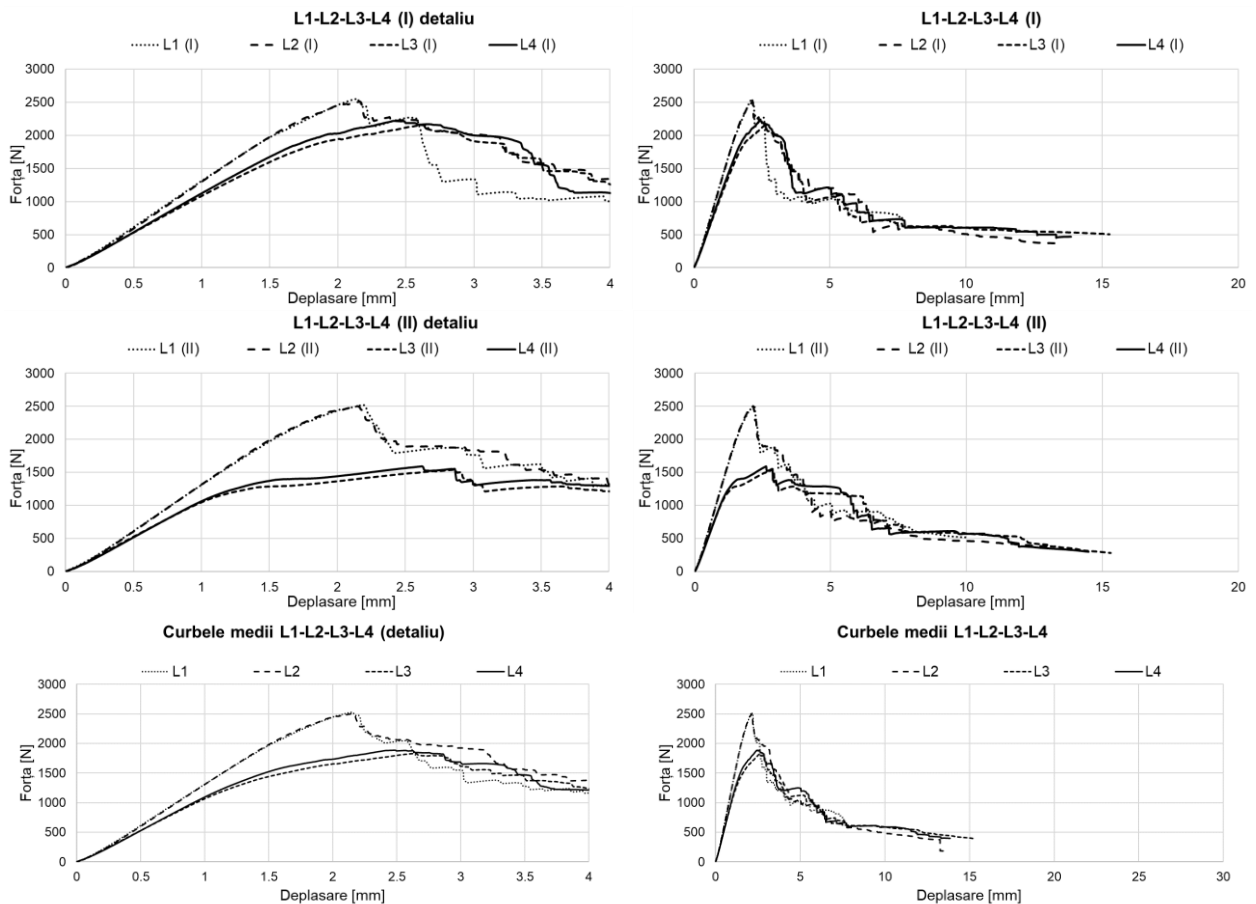### 5.4.5.   Conclusion pseudo-laminate L

Fig. 386 Comparison between materials L1-L2-L3 și L4 on average bending curves

In the graph of the average curves L1, L2, L3 and L4, the results obtained after the bending test are compared. Following a quantitative analysis, we can conclude that the heat treatment applied, for F (I), does not influence the performance of the composite, while for F (II), the heat treatment is beneficial. It is well known that heat treatment brings a benefit to the polymerization of the composite, in the sense that it uniformizes the bonds between the layers and the matrix.
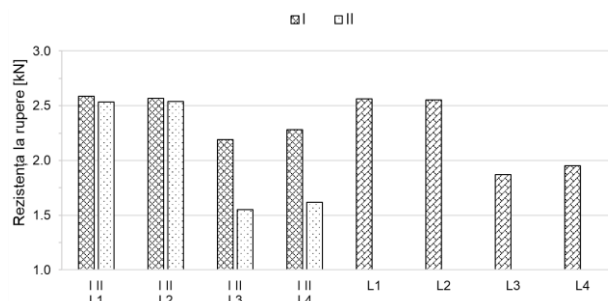

Fig. 387 Breaking stress for L-shaped resin materials

In **Fig. 387**, it can be seen how the breaking stress has different values for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. Thus, for the only naturally polymerized material, L1, the average breaking stress on surface I is 2.0% higher than the average on surface II. In the case of heat-treated material, L2, the average breaking stress on surface I is 1.2% higher than the average on surface II. For the material modified with NMP solvent and naturally polymerized, L3, the average breaking stress on surface I is 41.0% higher than the average on surface II. In the case of heat-treated material modified with NMP solvent, L4, the average breaking stress on surface I is 40.9% higher than the average on surface II.

For materials formed with type L resin (when we consider the average on the entire laminate being composed of both the average on surface I and the average on surface II), it can be observed, from the point of view of breaking stress, that the heat treatment has negatively influenced the properties of materials, but only to a small extent. Thus, the average breaking stress for L1 pseudo-laminate is 0.4% higher than L2. For NMP-modified materials, the same behavior can be observed, namely that the solvent has significantly reduced the breaking stress compared to non-NMP-modified materials. Regarding the heat treatment applied to the two pseudo-laminates modified with NMP, it can be seen that the mechanical properties were positively influenced, the average breaking stress for L4 pseudo-laminate is 4.2% higher than L3. Also, a comparison between the materials presented in the figure above, can also highlight the influence of the NMP solvent on the properties of the materials. Thus, the breaking stress for L1 pseudo-laminate is 36.9% higher than L3. In the case of L2 material, a higher value of breaking stress was registered, the average being 30.8% higher than L4.

Therefore, analyzing the average results of the breaking stress of **Fig. 387** of the materials formed with L type resin, we can observe that from this point of view, the best pseudo-laminate was the material that was not modified with NMP and was not heat treated, L1. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that following the bending tests (performed in three points), the best material was L4, i.e. the material formed with L-type resin, modified with NMP solvent and heat treated. The L4 material has a breaking stress value 4.2% higher than the L3 material
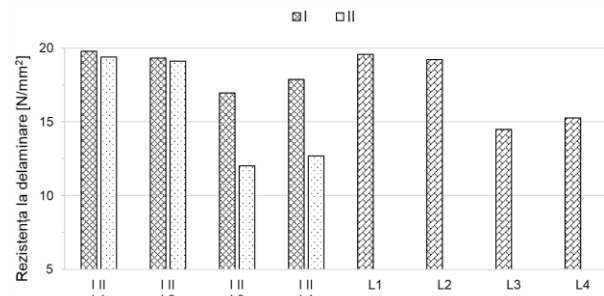
Fig. 388 Delamination resistance for L-shaped resin materials

In **Fig. 388**, it can be seen how the resistance to delamination has different values for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. With regard to the average on surface I, compared to surface II, the same percentage values are kept as in the case of breaking strength.

For materials formed with type L resin (when we consider the average on the entire laminate being composed of both the average on surface I and the average on surface II), it can be observed, from the point of view of resistance to delamination, that the heat treatment has negatively influenced the properties of materials. Thus, the average delamination resistance for L1 pseudo-laminate is 2.0% higher than L2. For NMP-modified materials, the same behavior can be observed, namely that the solvent significantly reduced the delamination resistance compared to non-NMP-modified materials. Regarding the heat treatment applied to the two pseudo-laminates modified with NMP, it can be seen that the mechanical properties were positively influenced, the average delamination resistance for the L4 pseudo-laminate is 5.5% higher than L3. Also, a comparison between the materials presented in the figure above, can also highlight the influence of the NMP solvent on the properties of the materials. Thus, the delamination resistance for L1 pseudo-laminate is 35.2% higher than L3. In the case of L2 material, a higher value of delamination resistance was registered, the average being 25.7% higher than L4.

Therefore, analyzing the average results of the delamination resistance in **Fig. 388** of the materials formed with L-type resin, we can see that from this point of view, the best pseudo-laminate was the material that was not modified with NMP and was not heat treated, L1, the result being similar to that of to breaking stress. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that following the bending tests (performed in three points), the best material was L4, i.e. the material formed with L-type resin, modified with NMP solvent and heat treated. L4 material has a 5.5% higher delamination resistance value than L3 material.
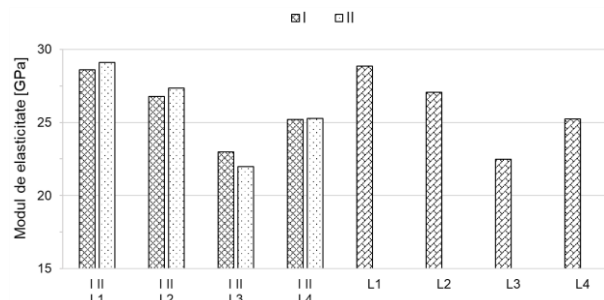

Fig. 389 Modulus of elasticity for materials formed with type L resin

In **Fig. 389**, it can be seen how the modulus of elasticity has different values for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. Thus, the average on surface II for the L1 material is 1.8% higher than the average on surface I. In the case of L2 material, a higher value of the modulus of elasticity on surface II was registered, being 2.2% higher than the average on surface I. The average on surface I for material L3, is 4.6% higher than the average on surface II. In the case of L4 material, a higher value of the modulus of elasticity on surface II was registered, being 0.2% higher than the average on surface I.

The influence of the heat treatment applied, in the case of the modulus of elasticity, seems to be reversed for the material L2 compared to L1. Thus, for the pseudo-laminates L1 and L2, a higher value of the modulus of elasticity was registered on surface II, the average being higher for the non-heat-treated material. The same observation applies to the comparison on surface I of the two materials. It is known that heat treatment brings a benefit to the polymerization of the composite, in the sense that it uniformizes the bonds between layers and matrices, and, we would have expected the modulus of elasticity to be higher in the case of heat-treated materials.

For materials formed with type L resin (when we consider the average on the entire laminate being composed of both the average on surface I and the average on surface II), it can be observed, from the point of view of the modulus of elasticity, that the heat treatment negatively influenced the properties of materials. Thus, the average modulus of elasticity for L1 pseudo-laminate is 6.6% higher than L2. For NMP-modified materials, the same behavior can be observed, namely that the solvent significantly reduced the modulus of elasticity compared to non-NMP-modified materials. Regarding the heat treatment applied to the two pseudo-laminates modified with NMP, it can be seen that the mechanical properties were positively influenced, the average modulus of elasticity for the L4 pseudo-laminate is 12.3% higher than L3. Also, a comparison between the materials presented in the figure above, can also highlight the influence of the NMP solvent on the properties of the materials. Thus, the average modulus of elasticity for L1 pseudo-laminate is 28.4% higher than L3. In the case of L2 material, a higher value of the modulus of elasticity was registered, the average being higher by 7.3% compared to L4.

Therefore, analyzing the average results of the modulus of elasticity of **Fig. 389** of the materials formed with L-type resin, we can observe that from this point of view, the best pseudo-laminate was the material that was not modified with NMP and was not heat treated, L1, the result being similar to that of to breaking stress. Since the purpose of the study is to identify a material with elasticity gradient, analyzing the four pseudo-laminates from this point of view, we can say that following the bending tests (in three points performed), the best material was L4, i.e. the material formed with L-type resin, modified with NMP solvent and heat treated. The L4 material has a value of modulus of elasticity 12.3% higher than the L3 material.

Tabelul 50 Variația valorii temperaturii, față de mediul ambiant, pentru L1

Epruveta / Eveniment		1	2	3	4	5	6
1	I / II T [°C]	8.4 / 6.7	9.4 / 6.4	10.6 / 13.7	7.3 / 9.1	8.4 / 9.2	9.6 / 5.6
	t [s]	30.5 / 33.0	29.0 / 32.0	30.0 / 31.0	29.5 / 31.5	30.6 / 30.3	29.5 / 30.0
2	I / II T [°C]	4.0 / 3.0	3.6 / 2.8	6.7 / 2.8	4.6 / 3.9	5.3 / 4.5	5.6 / 9.4
	t [s]	52.2 / 41.5	37.0 / 40.1	35.0 / 39.7	34.2 / 45.4	43.6 / 64.0	39.0 / 31.0
3	I / II T [°C]	3.8 / 4.8	- / 3.3	- / 3.4	- / 3.6	4.2 / -	- / 3.8
	t [s]	57.0 / 51.0	- / 53.5	- / 53.0	- / 56.5	71.5 / -	- / 46.2
4	I / II T [°C]	- / -	- / -	- / -	- / -	4.1 / -	- / 4.3
	t [s]	- / -	- / -	- / -	- / -	94.5 / -	- / 70.5
Temperatura mediului ambiant				19.1°C			

Delaminare axială | Rupere C₂₄₀ strat inferior | Delaminare laterală

Tabelul 54 Variația valorii temperaturii, față de mediul ambiant, pentru L3

Epruveta / Eveniment		1	2	3	4	5	6
1	I / II T [°C]	2.6 / 11.9	3.1 / 6.0	2.5 / 7.5	6.6 / 8.9	3.2 / 8.7	3.2 / 11.5
	t [s]	46.0 / 42.5	47.5 / 40.1	70.0 / 41.1	50.0 / 42.1	52.0 / 39.5	53.0 / 39.0
2	I / II T [°C]	6.4 / 6.4	2.5 / 8.0	6.4 / 5.5	7.4 / 6.7	5.6 / 7.9	6.1 / 7.0
	t [s]	75.0 / 98.4	55.5 / 81.1	92.0 / 90.4	53.0 / 84.1	73.0 / 81.1	78.0 / 77.0
3	I / II T [°C]	- / -	7.4 / -	- / -	2.6 / -	6.1 / -	6.5 / -
	t [s]	- / -	76.0 / -	- / -	70.0 / -	97.0 / -	91.0 / -
4	I / II T [°C]	- / -	- / -	- / -	- / -	- / -	- / -
	t [s]	- / -	- / -	- / -	- / -	- / -	- / -
Temperatura mediului ambiant				19.5°C			

Delaminare axială | Rupere C₂₄₀ strat inferior | Delaminare laterală

Tabelul 52 Variația valorii temperaturii, față de mediul ambiant, pentru L2

Epruveta / Eveniment		1	2	3	4	5	6
1	I / II T [°C]	4.8 / 10.2	7.1 / 5.7	6.5 / 8.9	8.5 / 10.5	4.9 / 12.2	8.4 / 10.1
	t [s]	32.0 / 33.0	29.0 / 29.0	28.5 / 29.0	28.5 / 30.3	28.0 / 29.0	28.0 / 29.1
2	I / II T [°C]	1.0 / 4.4	1.8 / 5.0	2.5 / 8.4	2.1 / 4.9	1.9 / 4.8	2.5 / 5.0
	t [s]	54.0 / 46.0	49.2 / 41.5	65.2 / 53.0	45.0 / 47.0	80.5 / 41.0	40.3 / 42.4
3	I / II T [°C]	3.2 / 4.8	3.3 / 4.7	2.5 / -	4.8 / 6.3	2.7 / 6.3	- / 5.9
	t [s]	79.1 / 59.0	75.1 / 50.2	81.1 / -	79.5 / 62.5	110.0 / 53.0	- / 50.0
4	I / II T [°C]	- / -	- / -	- / -	1.8 / -	- / -	- /
	t [s]	- / -	- / -	- / -	92.5 / -	- / -	- / -
Temperatura mediului ambiant				19.8°C L2 (I) și 16.4°C L2 (II)			

Delaminare axială | Rupere C₂₄₀ strat inferior | Delaminare laterală

Tabelul 56 Variația valorii temperaturii, față de mediul ambiant, pentru L4

Epruveta / Eveniment		1	2	3	4	5	6
1	I / II T [°C]	- / 5.8	2.1 / 10.3	5.2 / 7.1	4.8 / 3.7	6.7 / 8.5	3.6 / -
	t [s]	- / 36.7	65.5 / 40.1	76.5 / 39.1	82.0 / 36.2	70.2 / 37.0	46.0 / -
2	I / II T [°C]	- / 3.7	5.3 / 9.3	- / 1.6	- / 11.9	4.4 / 8.5	3.5 / -
	t [s]	- / 91.5	80.9 / 82.5	- / 82.1	- / 39.5	97.8 / 74.0	47.5 / -
3	I / II T [°C]	- / -	2.2 / -	- / -	- / 9.1	4.4 / -	8.6 / -
	t [s]	- / -	165.0 / -	- / -	- / 73.5	146.2 / -	70.1 / -
4	I / II T [°C]	- / -	- / -	- / -	- / -	- / -	- / -
	t [s]	- / -	- / -	- / -	- / -	- / -	- / -
Temperatura mediului ambiant				16.2°C L4 (I) și 19.5°C L4 (II)			

Delaminare axială | Rupere C₂₄₀ strat inferior | Delaminare laterală

Fig. 390 The variation of the temperature value, compared to the environment, for L1, L2, L3, L4

In **Fig. 390**, it can be observed how the variation of the temperature value presents different values as well as different behavior, for the case when the force is applied on the surface I, compared to the case when the force is applied on the surface II. This method of presentation is a very easy one because the increases in temperature values can be immediately correlated with the identified mechanical effects (axial delamination, lateral delamination or layer fractures).

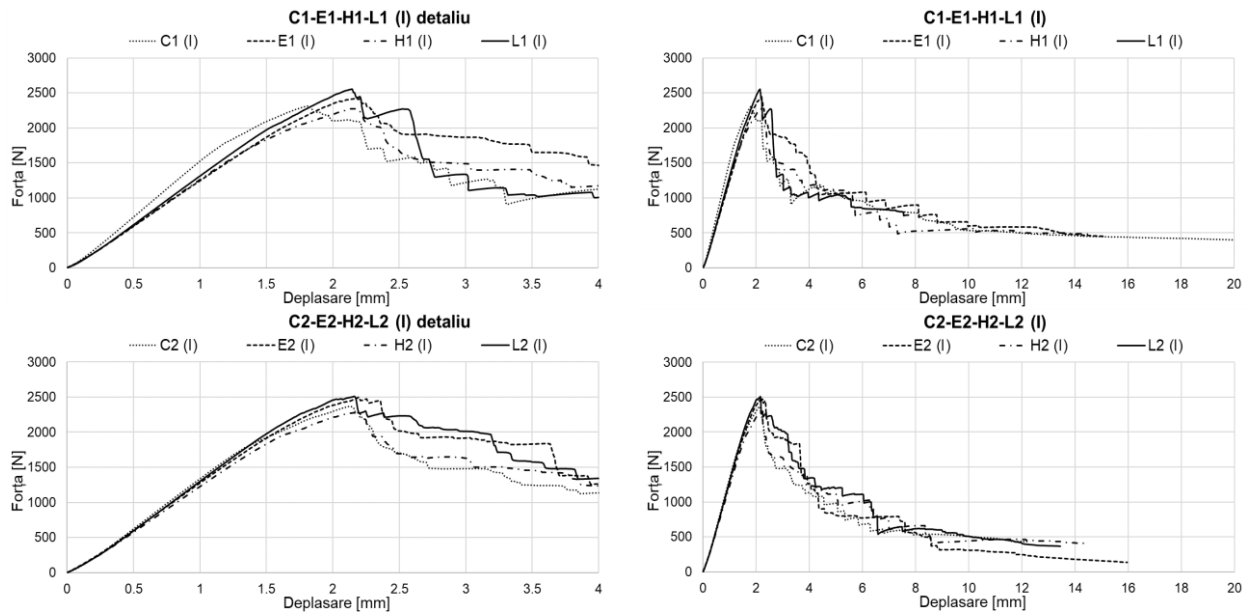## 5.5.   Comparative analysis of pseudo-laminates

Fig. 391 Comparative analysis of the average curves F (I) of heat treated/untreated materials

When the force is applied on surface (I), for the naturally polymerized pseudo-laminates and not modified with NMP, it can be observed that from the point of view of the average curves, the best answer is presented by the material formed with L-type resin, while the material formed with type C resin shows the weakest response, followed very closely by the material formed with type H resin.

When the force is applied on surface (I), for the pseudo-laminates not modified with NMP, but heat treated, it can be observed that from the point of view of the average curves, the best answer is presented by the material formed with type L resin, being followed extremely close to E-type resin material, while H-type resin material has the weakest response.
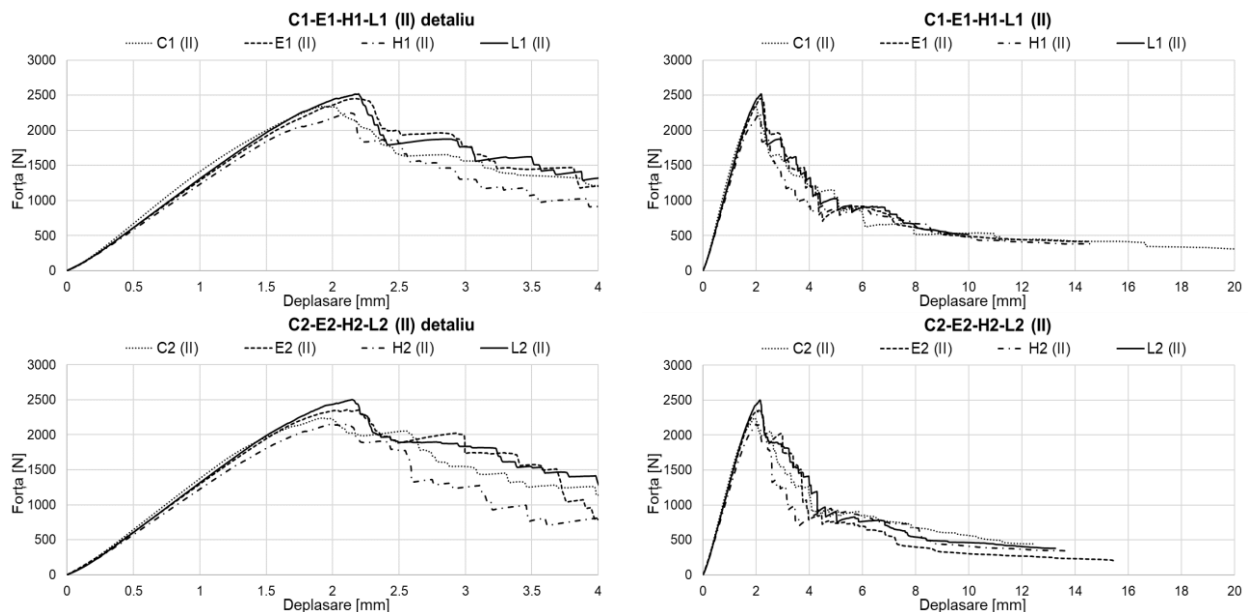

Fig. 392 Comparative analysis of the average curves F (II) of heat treated / untreated materials

When the force is applied on surface (II), for the naturally polymerized pseudo-laminates and not modified with NMP, it can be observed that from the point of view of the average

curves, the best answer is the whole material formed with L-type resin, while the material formed with type H resin shows the weakest response.

When the force is applied on surface (II), for the pseudo-laminates not modified with NMP, but heat treated, it can be observed that from the point of view of the average curves, the best answer is presented by the material formed with type L resin, being followed extremely close to E-type resin material, while H-type resin material has the weakest response.

In the case of pseudo-laminates naturally polymerized and not modified with NMP, when we consider the average over the entire laminate being composed of both the average on surface I and the average on surface II, it can be seen that in terms of average curves, the best the answer is found at the material formed with type L resin, while the material formed with type H resin has the weakest answer, followed very closely by the material formed with type C resin.

For pseudo-laminates not modified with NMP, but heat treated, when we consider the average over the entire laminate being composed of both the average on surface I and the average on surface II, it can be seen that from the point of view of the average curves, the best response is presented by the material formed with type L resin, being followed extremely close by the material formed with type E resin, while the material formed with type H resin has the weakest response.
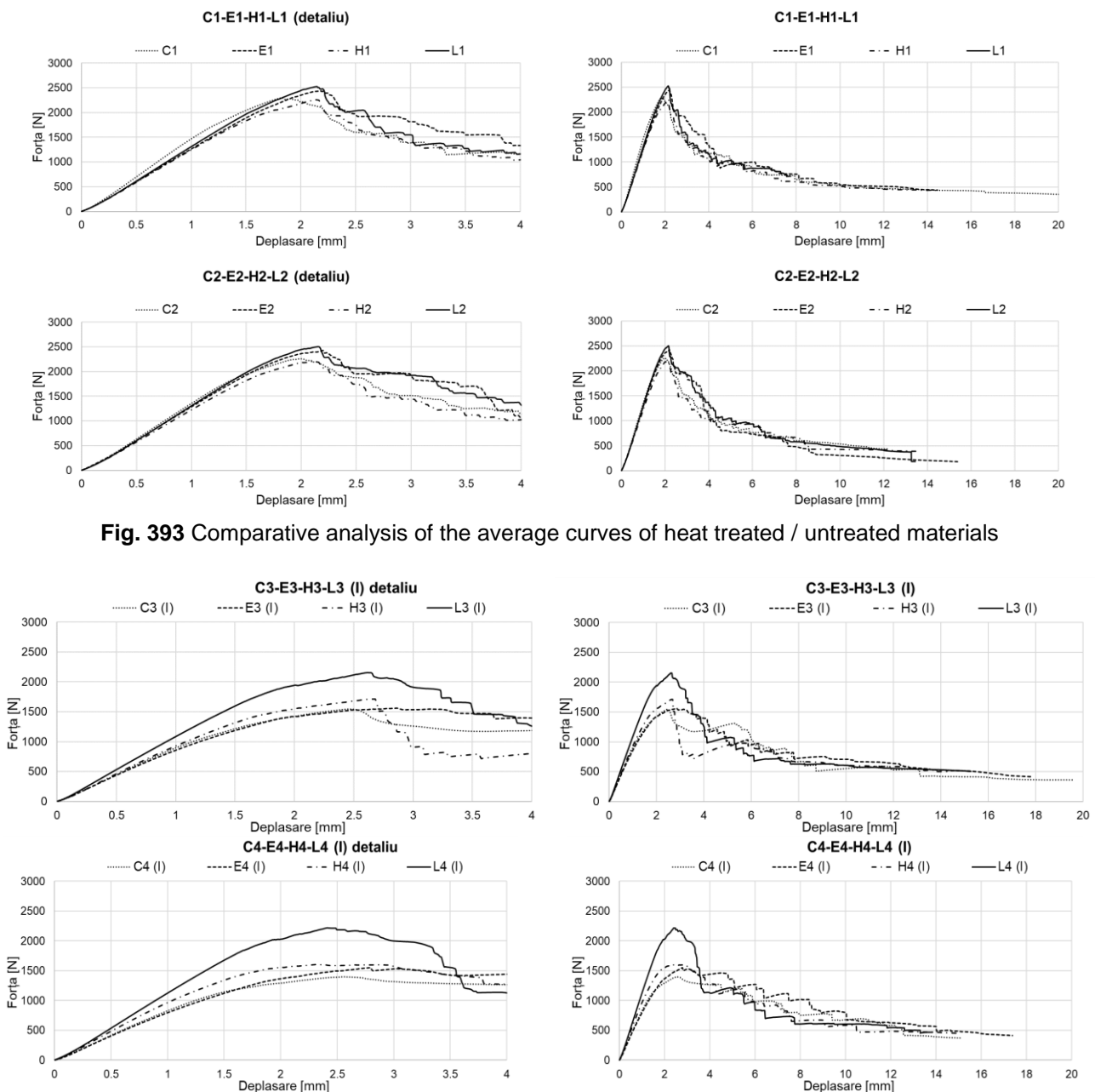

Fig. 393 Comparative analysis of the average curves of heat treated / untreated materials

Fig. 394 Comparative analysis of mean curves F (I) of NMP-modified and heat-treated/untreated materials

When the force is applied on surface (I), for the pseudo-laminates modified with NMP, but not heat treated, it can be observed that from the point of view of the average curves, the best answer is presented by the material formed with type L resin, while the material formed with type C resin shows the weakest response.

When the force is applied on surface (I), for the pseudo-laminates modified with NMP and heat treated, it can be observed that from the point of view of the average curves, the best answer is presented by the material formed with L-type resin, while the material formed with type C resin shows the weakest response.

When the force is applied on surface (II), for the pseudo-laminates modified with NMP, but not heat treated, it can be observed that from the point of view of the average curves, the best answer is presented by the material formed with type L resin, while the material formed with type E resin shows the weakest response, but the difference is very small compared to H and C.
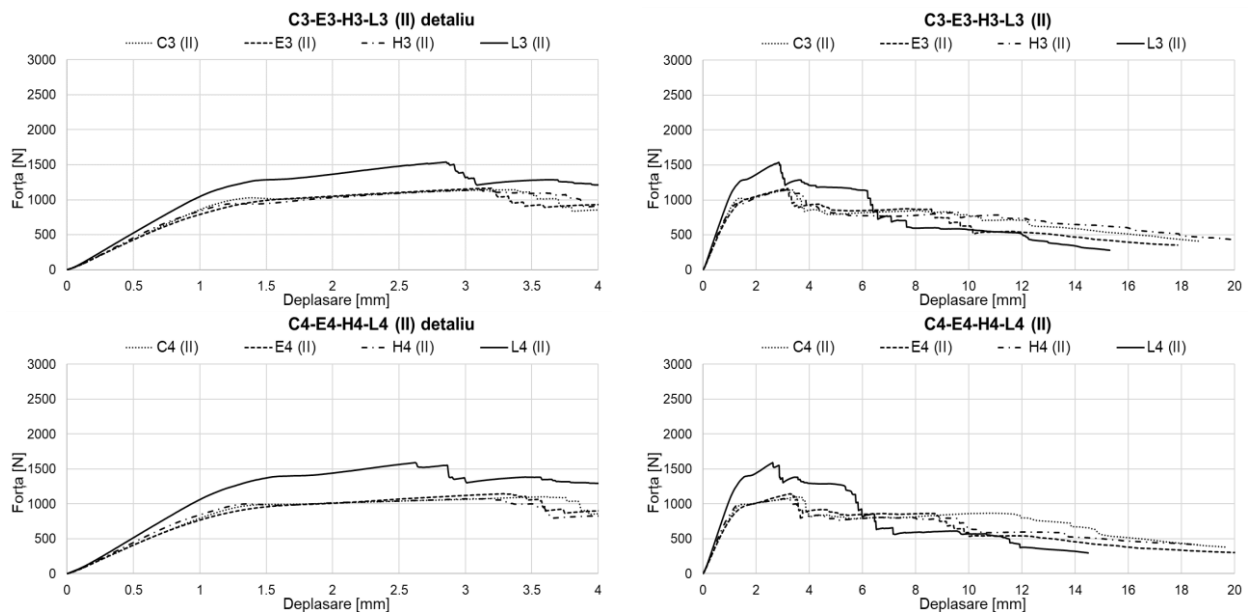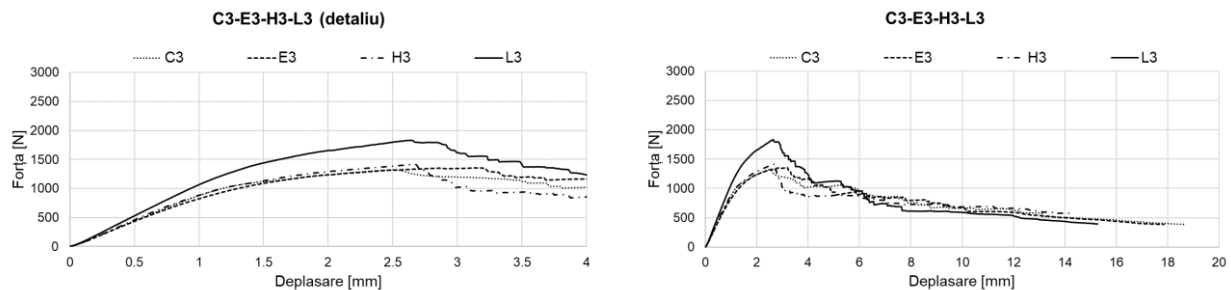

Fig. 395 Comparative analysis of F (II) mean curves of NMP-modified and heat-treated/untreated materials

When the force is applied on surface (II), for pseudo-laminates modified with NMP and heat treated, it can be observed that from the point of view of the average curves, the best answer is presented by the material formed with type L resin, while the material formed with type E resin has the weakest response, but the difference is very small compared to H and C.
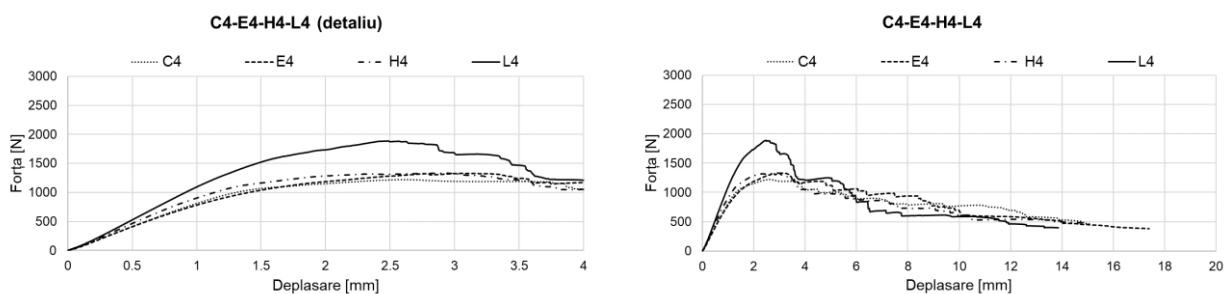
Fig. 396 Comparative analysis of the average curves of NMP-modified and heat-treated materials

In the case of pseudo-laminates modified with NMP, but not heat treated, when we consider the average over the entire laminate being composed of both the average on surface I and the average on surface II, it can be seen that in terms of average curves, the best answer is the material formed with type L resin, while the material formed with type E resin has the weakest answer, but the difference is very small compared to H and C.

For pseudo-laminates modified with NMP and heat treated, when we consider the average over the entire laminate being composed of both the average on surface I and the average on surface II, it can be seen that from the point of view of average curves, the best answer it is presented by the material formed with type L resin, while the material formed with type E resin has the weakest response, but the difference is very small compared to C.
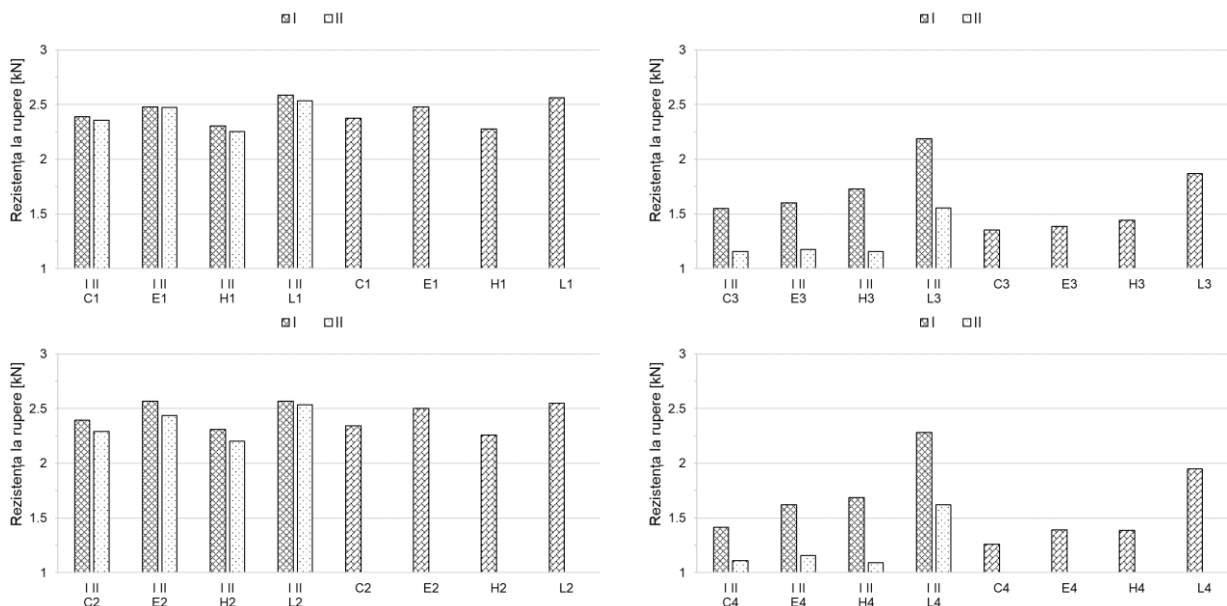

Fig. 397 Comparison between materials C-E-H and L on breaking stress

In the case of non-modified NMP pseudo-laminates and not heat-treated, it can be seen that from the point of view of breaking stress, the best answer is the material formed with type L resin, while the material formed with type H resin has the highest poor answer. The L1 material has a breaking stress 12.4% higher than the pseudo-laminate with the weakest response, H1. Compared to other materials, L1 has a higher breaking stress by 7.9% compared to C1, and 3.4% compared to E1.

In the case of pseudo-laminates not modified with NMP, but heat treated, it can be seen that in terms of breaking stress, the best answer is the material formed with type L resin, while the material formed with type H resin has the weaker response. The L2 material has a breaking stress 13.0% higher than the pseudo-laminate with the weakest response, H2. Compared to other materials, L2 has a higher breaking stress by 8.9% compared to C2, and 2.0% compared to E2.

In the case of pseudo-laminates modified with NMP and naturally polymerized, it can be seen that in terms of breaking stress, the best response is the material formed with type L resin, while the material formed with type C resin has the poorest answer. The L3 material has a breaking stress 38.2% higher than the pseudo-laminate with the weakest response, C3.

Compared to other materials, L3 has a higher breaking stress by 29.6% compared to H3, and by 34.7% compared to E3.
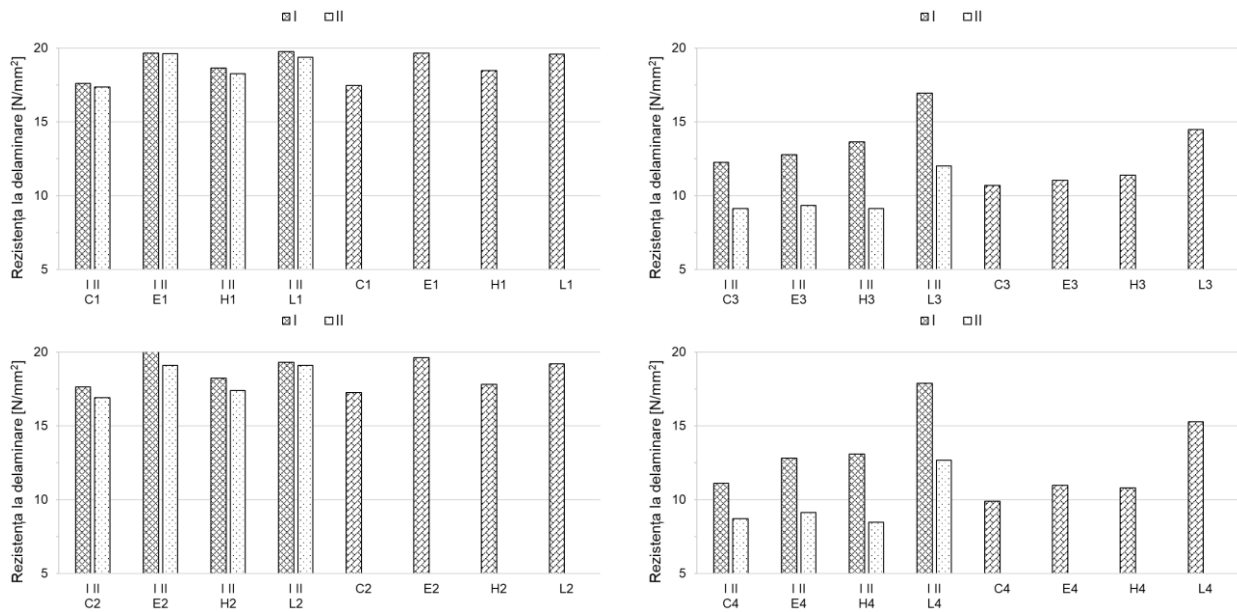

Fig. 398 Comparison between C-E-H and L materials on delamination resistance

In the case of pseudo-laminates modified with NMP, but heat treated, it can be seen that in terms of breaking stress, the best response is the material formed with type L resin, while the material formed with type C resin has the weaker response. The L4 material has a breaking stress 54.7% higher than the pseudo-laminate with the weakest response, C4. Compared to other materials, L4 has a higher breaking stress by 40.5% compared to H4, and by 40.3% compared to E4.

In the case of non-modified NMP pseudo-laminates and not heat-treated, it can be seen that from the point of view of delamination resistance, the best answer is the material formed with type E resin, while the material formed with type C resin has the poorest answer. Material E1 has a delamination resistance 12.4% higher than the pseudo-laminate with the weakest response, C1. Compared to other materials, E1 has a higher delamination resistance by 6.4% compared to H1, and 0.3% compared to L1.

In the case of pseudo-laminates not modified with NMP, but heat-treated, it can be seen that in terms of resistance to delamination, the best answer is the material formed with type E resin, while the material formed with type C resin has the weaker response. Material E2 has a delamination resistance 13.6% higher than the pseudo-laminate with the weakest response, C2. Compared to other materials, E2 has a 10% higher delamination resistance than H2, and 2.2% higher than L2.

In the case of pseudo-laminates modified with NMP and naturally polymerized, it can be seen that in terms of delamination resistance, the best response is the material formed with type L resin, while the material formed with type C resin has the poorest answer. The L3 material has a delamination resistance 35.4% higher than the pseudo-laminate with the weakest response, C3. Compared to other materials, L3 has a higher delamination resistance by 27.2% compared to H3, and 30.9% compared to E3.

In the case of pseudo-laminates modified with NMP, but heat-treated, it can be seen that in terms of resistance to delamination, the best answer is the material formed with type L resin, while the material formed with type C resin has the weaker response. The L4 material has a delamination resistance 54.3% higher than the pseudo-laminate with the weakest response, C4. Compared to other materials, L4 has a higher delamination resistance by 41.6% compared to H4, and 39.2% compared to E4.
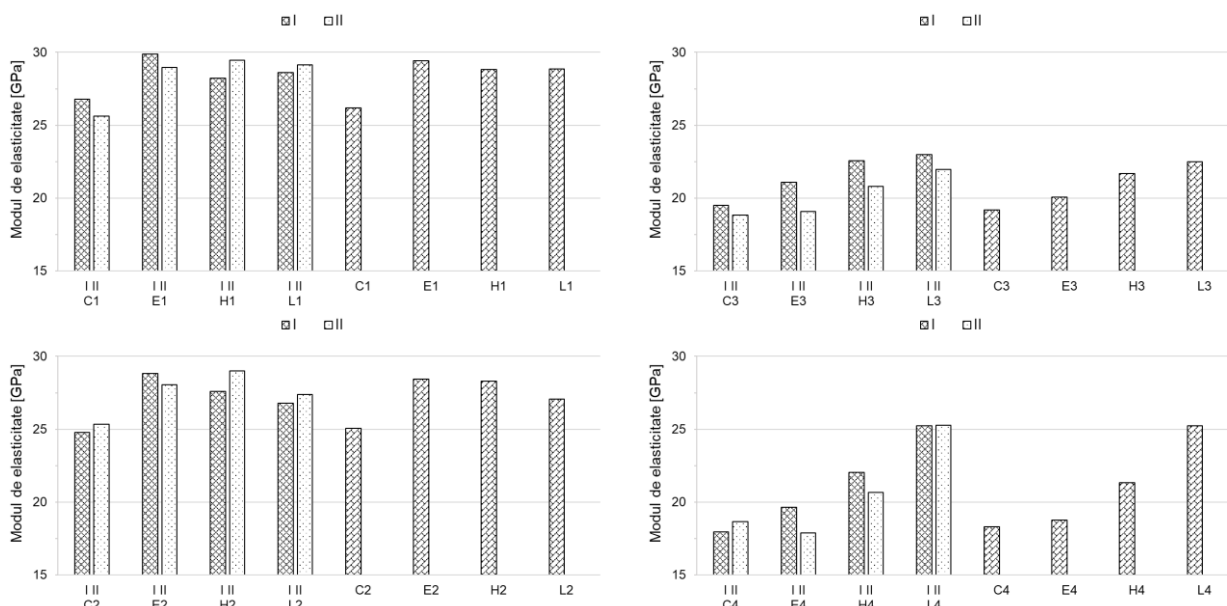
Fig. 399 Comparison between materials C-E-H and L regarding the modulus of elasticity

In the case of non-modified NMP pseudo-laminates and not heat-treated, it can be seen that from the point of view of the modulus of elasticity, the best answer is the material formed with type E resin, while the material formed with type C resin has the poorest answer. Material E1 has a modulus of elasticity 12.3% higher than the pseudo-laminate with the weakest response, C1. Compared to other materials, E1 has a modulus of elasticity 2.0% higher than H1, and 1.9% higher than L1.

In the case of pseudo-laminates not modified with NMP, but heat treated, it can be seen that from the point of view of the modulus of elasticity, the best answer is the material formed with type E resin, while the material formed with type C resin has the weaker response. Material E2 has a modulus of elasticity 13.4% higher than the pseudo-laminate with the weakest response, C2. Compared to other materials, E2 has a modulus of elasticity 0.5% higher than H2, and 5.0% higher than L2.

In the case of pseudo-laminates modified with NMP and naturally polymerized, it can be seen that from the point of view of the modulus of elasticity, the best answer is the material formed with type L resin, while the material formed with type C resin has the poorest answer. The L3 material has a modulus of elasticity 17.3% higher than the pseudo-laminate with the weakest response, C3. Compared to other materials, L3 has a modulus of elasticity 3.7% higher than H3, and 12.0% higher than E3.

In the case of pseudo-laminates modified with NMP, but heat treated, it can be seen that from the point of view of the modulus of elasticity, the best answer is the material formed with type L resin, while the material formed with type C resin has the weaker response. The L4 material has a modulus of elasticity 37.9% higher than the pseudo-laminate with the weakest response, C4. Compared to other materials, L4 has a modulus of elasticity 18.2% higher than H4, and 34.4% higher than E4.

Table 55 The relative increase of the temperature value compared to the environment

Specimen / Material	1	2	3	4	5	6	Average specimens 1-6
C1 (I)	38%	53%	40%	44%	47%	33%	42%
C1 (II)	56%	48%	44%	47%	39%	44%	46%
C2 (I)	24%	21%	-	29%	69%	34%	35%
C2 (II)	54%	46%	49%	80%	52%	32%	52%
C3 (I)	25%	18%	27%	15%	16%	17%	20%
C3 (II)	30%	27%	41%	20%	35%	23%	29%
C4 (I)	35%	7%	12%	27%	27%	39%	24%
C4 (II)	20%	31%	36%	46%	34%	40%	35%
E1 (I)	63%	53%	61%	32%	47%	52%	51%

E1 (II)	30%	51%	25%	50%	41%	38%	39%
E2 (I)	38%	33%	46%	44%	45%	-	41%
E2 (II)	32%	43%	33%	51%	44%	48%	42%
E3 (I)	28%	61%	30%	64%	29%	32%	41%
E3 (II)	83%	88%	75%	80%	111%	90%	88%
E4 (I)	67%	47%	47%	72%	55%	51%	56%
E4 (II)	126%	96%	94%	94%	70%	88%	95%
H1 (I)	61%	47%	43%	48%	33%	40%	45%
H1 (II)	46%	33%	53%	42%	47%	34%	42%
H2 (I)	34%	24%	48%	50%	41%	70%	45%
H2 (II)	57%	51%	76%	76%	79%	51%	65%
H3 (I)	60%	64%	54%	64%	73%	62%	63%
H3 (II)	78%	85%	77%	78%	119%	73%	85%
H4 (I)	18%	22%	12%	60%	65%	55%	39%
H4 (II)	47%	63%	68%	73%	66%	55%	62%
L1 (I)	44%	49%	55%	38%	44%	50%	47%
L1 (II)	35%	34%	72%	48%	48%	49%	48%
L2 (I)	24%	36%	33%	43%	25%	42%	34%
L2 (II)	62%	43%	54%	68%	74%	62%	61%
L3 (I)	33%	38%	33%	38%	31%	31%	34%
L3 (II)	61%	41%	38%	46%	45%	59%	48%
L4 (I)	-	33%	32%	30%	41%	53%	38%
L4 (II)	30%	53%	36%	61%	44%	-	45%

From **Table 55**, there are some obvious differences, especially between the 2 surfaces of the pseudo-laminate, mainly due to the asymmetry. When the force is applied to the surface I, the recorded temperatures have lower values for most pseudo-laminates except E1, H1, L1 and L2 where the recorded temperatures have higher values, but the differences are not significant. Also, these differences are not observed in the case of NMP-modified or NMP-modified and heat-treated materials. The chemical bonds at the interface between fiber S_{163} and fiber C/A_{68} or fiber S_{163} and fiber A_{61}, are not as high quality as the bond between fiber A_{61} and fiber S_{163} due to orientations at different angles of the sheets. This observation was extracted from the analysis of the specimens, which showed delamination between the layers of S_{163} fiber and A_{61} fiber or inside the A_{61} fiber package.

In 5.1, 5.2, 5.3 and 5.4, the bending results (force-displacement) of the tested specimens are presented and it can be seen that there are differences depending on the direction of application of the force.

5.6.   Conclusions – bending tests

Regarding the pseudo-laminates named with the numbers 1 and 2 presented, it shows that the pseudo-laminate L1 (I) has the best response to bending, while H2 (II) has the weakest response. In the case of pseudo-laminates named 3 and 4, the best answer is L4 (I), while H4 (II) has the weakest answer. However, it must be kept in mind that pseudo-laminates are not symmetrical and, in this case, they must be cataloged according to the response of each surface.

Analyzing, the mediated results presented in the figures in chapter 5.5, other conclusions are drawn: the epoxy system Epiphen, does not lead to the best response to bending, as expected, it is represented by the epoxy resin type L, while the epoxy resin type H has the weakest response. As can be seen from the figures shown, the best response to bending stress is presented by type L epoxy resin, followed very closely by type E epoxy resin.

Thus, according to the graphs shown in **Fig. 397**, we can identify that the best response to bending, in terms of breaking stress, had the epoxy resin type L, for all 4 cases (1-normal, 2-heat treated, 3-modified with NMP, 4 -heat treated and modified with NMP), regardless of the direction of action of the force, while the epoxy resin type H, showed the weakest response, for all 4 cases of loading, except when the force acted on the surface I for NMP-modified pseudo-laminates (3 and 4), where the weakest response was recorded at C4 (I).

Also, according to the graphs shown in **Fig. 399**, we can identify that the best response to bending, in terms of modulus of elasticity, was the material formed with epoxy resin type L, for two types of matrix (3-modified with NMP, 4-heat treated and modified with NMP), while type C epoxy resin showed the weakest response, for all 4 loading cases. For the other two types of matrixes (1-normal, 2-heat treated), the best response to bending was the material formed with type E epoxy resin.

Since a large part of the specimens were delaminated inside the A_{61} fiber bundle, or at the interface between the A_{61} fiber adjacent reinforcement bundles, this phenomenon can be explained by the fact that there is a strong influence on the bending behavior of the pseudo-laminate forming technique. Given that the A_{61} fiber fabric is thin, and the forming technique requires impregnation with pre-polymer mixture when placed in the mold, above the C_{240} fabric, it will be affected not only by its own corrugation, but also by the adjacent fabric.

Both the modulus of elasticity and the bending behavior of the materials depend on the direction of force application.

Stratification C_{240}, C/A_{68}, A_{61}, S_{163}, is a beneficial one both because of the unmodified materials and because of the materials modified with NMP.

Stratification of S_{163}, C/A_{68}, A_{61}, C_{240} is not appropriate. There are detachments at the interface between the A_{61}-C_{240} fabrics but also some detachments, less, at the aramid-mixed fabric interface.

As specified in the previous chapter, a solvent was introduced into the matrix of the formed pseudo-laminates to obtain a gradient of elasticity of the material, thus resulting in a pseudo-laminate with controllable strength.

With regard to lateral delamination or slipping of layers or packages, it should be noted that specimens 2, 4 and 6, when referring to surface II, had an addition of material at one free end in order to recover the specimen from cutting, while test specimens 1, 3 and 5, had the same addition, but at both ends. A hacksaw was used to separate the specimens, and this may lead to an explanation of why the aramid layers gave way. Thus, the shear strength of the aramid fibers being high, led to an additional effort induced by the hacksaw, in order to perform the separation of the specimens.

Fracture of the matrix between two layers of reinforcement has consequences at the free ends of the specimen in the relative slip of the remaining reinforcement packages. What is constant is that it never takes place at both ends, but takes place at one end, and, presumably, the end that was cut with the hacksaw is more affected.

6. Conclusions and research directions

6.1. Final conclusions

The main objective of this paper is to establish a methodology for obtaining a new composite material, reinforced with fiber fabric and flexible matrix.

To achieve this goal, the following six specific objectives have been achieved:

- the study of the specialized literature regarding the formation of composite materials reinforced with fiber fabrics (carbon, glass, aramid, mixed fabric) as well as the influence of different types of layering and orientations of the fabric;

- study of the specialized literature regarding the influence of the epoxy matrix in the formation of composite materials reinforced with fabrics;

- study of the literature on the influence of modifying the epoxy matrix with N-methyl-2-pyrrolidinone in the formation of composite materials reinforced with fabrics;

- formation of composite materials reinforced with fabrics and epoxy matrix;

- performing the experimental analysis for the characterization of the formed materials;

- interpretation and study of the results obtained.

In order to achieve the first three specific objectives, the scientific papers referred to in Chapter 1 were analyzed, on the basis of which it was established:

- the architecture of pseudo-laminates in order to obtain a new material, with elasticity gradient;

- the technique for inserting and fixing the fabrics in the mold;

- epoxy resins for the test matrix.

Experimental analyzes were performed to achieve the following two specific objectives. Thus, in Chapter 3 is presented the formation of composite materials with control epoxy matrix, from which pseudo-laminates were formed. **Table 2** shows the fabrics used in the formation of laminates, the orientation of the fabrics at different angles, as well as the concentration of solvent added to each package of fabrics.

16 materials with four different epoxy resins (commercially available) were formed to analyze the effect of matrix strengthening on the stretching and bending properties of materials:

- 4 pseudo-laminates that have not been heat treated or modified with N-methyl-2-pyrrolidinone;

- 4 pseudo-laminates that were subjected to heat treatment but were not modified with N-methyl-2-pyrrolidinone;

- 4 pseudo-laminates that were not subjected to heat treatment but were modified with N-methyl-2-pyrrolidinone;

4 pseudo-laminates that were heat treated and modified with N-methyl-2-pyrrolidinone.

Each material is reinforced with 35 layers of fiber fabrics having in the middle 5 layers of fiberglass, which are covered by aramid fiber fabrics (another five layers), on one side, and on the other side, are covered by mixed fabrics (another five layers), followed by another 5 layers of mixed fabrics (as opposed to the previous layers) and at the end (as outer layers) carbon fiber fabric (another five layers on each side). Four 210x294mm plates were formed for each matrix and two of the plates were heat treated, while the other two were modified with NMP solvent. Mechanical tests were performed one year after the formation of the materials.

In paragraphs 4.1 4.2 4.3 and 4.4 of Chapter 4 and paragraphs 5.1 5.2 5.3 and 5.4 of Chapter 5, the behavior of materials formed at tensile and bending stresses is investigated.

In order to achieve the last specific objective, the comparison of the experimental results presented in paragraphs 4.5 and 4.6 of Chapter 4 and 5.5 and 5.6 Chapter 5 was performed.

The synthetic conclusions reached on the basis of this study with regard to tensile tests are: materials formed with type C, E and L resin but not modified with NMP, can be subjected to a heat treatment, as they lead to better results, while, the heat treatment applied to the materials formed with type H resin, did not lead to viable results. For materials formed with type H and L resin and modified with NMP, they can be subjected to a heat treatment, as they lead to better results, while the heat treatment applied to materials formed with type C and E resin and modified with NMP, did not led to viable results. Thus, the L4 material, the one modified with NMP and heat treated, presents, from the point of view of the modulus of elasticity, the best

response to tensile tests. It has the modulus of elasticity (calculated) higher than E4 by 43.0%, higher than C4 by 14.1%, and higher than H4 by 9.8%.

Also, with regard to bending tests, only materials formed with type L resin and modified with NMP, can be subjected to a heat treatment, as they lead to better results, while the heat treatment applied to materials formed with type C resin, E and H, modified but also those not modified with NMP, did not lead to viable results. Thus, the L4 material, the one modified with NMP and heat treated, presents, from the point of view of the modulus of elasticity, the best response to the bending in three points. It has the modulus of elasticity (automatically) higher than C4 by 37.9%, higher than E4 by 34.4%, and higher than H4 by 12.2%.

Also, with regard to bending tests, only materials formed with L-type resin and modified with NMP can be subjected to a heat treatment, as they lead to better results. Thus, the L4 material, the one modified with NMP and heat treated, presents, from the point of view of the modulus of elasticity, the best response to the bending in three points. It has the modulus of elasticity (automatic), higher than E4 by 34.4%, higher than C4 by 37.9%, and higher than H4 by 12.2%.

6.2. Personal contributions

The aim of this paper is to establish a methodology for obtaining a new optimal composite material, reinforced with fabric and flexible matrix, by introducing in the epoxy matrix an agent for modifying and flexing the material obtained, identifying differences between materials with the same structure reinforcement, but with different types of epoxy resins as matrix.

The statistical analysis of the partial results leads to the conclusion that only a few specimens can be used to characterize the materials and with these results the mean curves σ/ε were evaluated. The specimens that were used for mediation are mentioned on the graphs together with images of the fractured specimens (front and side). Thermal measurement shows that there are differences between heat-treated materials and those that are naturally polymerized, but these values (mediated for the whole batch of specimens or only for specimens with similar mechanical behavior) are not too different and cannot be used to interpret mechanical results. The mechanical behavior of the materials, given by the average curves, has six slopes for each material. Of these slopes, the second corresponds to the elastic response of the composite material, while the sixth represents the elastic response of the fibers. The highest value of the modulus of elasticity is reached for the HT2 epoxy system with HT2 hardener (approximately 7.2Gpa) and corresponds to the matrix that has not been heat treated and has not been modified with NMP. Also, from the point of view of the modification of the matrix with NMP, the highest value of the modulus of elasticity, we find it in the epoxy system L with GL1 hardener (approximately 7.0Gpa).

The working methodology required that the specimens be extracted from the plates formed using a high-pressure water jet machine. The position of each sample was marked to associate any mechanical effects with the position of the specimen. The experimental method included two audio-video recording equipment (one to record a surface of the specimen and one to record an edge of the specimen) to associate mechanical events with visual effects (especially at rupture). A thermal imaging camera was used to record the increase in temperature during the tests, and the results mainly refer to the release of energy (identifiable by the relative increase in temperature relative to ambient temperature) at break.

The following original contributions were made to achieve this goal:
- constructing a working methodology regarding the testing, using at the same time the thermal imaging camera, the 2 digital video recording cameras, as well as the mechanical testing machine;
- formation of epoxy matrix composite materials, based on a new pseudo-laminate architecture, based on previous studies developed at CCDCOMT;
- carrying out experiments in order to establish the mechanical properties in terms of the results obtained from the tensile and bending stresses;
- comparison of the results obtained between matrices with the same epoxy resin, as well as comparison of the results obtained between matrices with different epoxy resin;
- making a material with elasticity gradient.

6.3. Future directions of research

Following the results obtained in the study presented, we found the opportunity to establish the mechanical properties from the perspective of other test methods such as:

- thermomechanical analysis in order to establish the coefficient of linear thermal expansion using TMA;

- Raman analysis by spectrophotometry, which could identify the bonds between polymers due to the increase of NMP concentration in the matrix and could highlight whether other compounds were formed as a result of chemical interactions, at the molecular level.

- SEM analysis of the material that could explain the process of interaction of electrons with pseudo-laminate constituents, and thus we can identify the existence of nanostructures. This method can also be applied to the specimens resulting from this study, without the need for new samples;

- analysis of the specific heat of the formed materials, using a differential scanning calorimeter, DSC. This analysis can identify the value of the glass transition temperature as well as the analysis of the loss of NMP solvent, following the heating of the test piece;

- electromagnetic analysis of pseudo-laminates using RLC-meter, so it could identify the electrical resistance as well as the electrical capacity of the newly formed material;

- analysis in the infrared spectrum of the surface of the specimens during tensile stresses, in order to identify areas with local heating, which is a phenomenon that can anticipate the fracture of the material, using a high-resolution thermal imaging camera;

- analysis of possible defects caused by the forming technique by non-destructive control methods in the infrared spectrum. By applying a heat source to a surface of the pseudo-laminate (before water jet cutting), interphase or interlaminar defects or the accumulation of a surplus of resin in a certain area can be identified.

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