

IOSUD – „DUNAREA DE JOS” UNIVERSITY OF GALATI

Doctoral School of Mechanical and Industrial Engineering



DOCTORAL THESIS

ABSTRACT

SYNERGETIC EFFECT OF WEAR AND FATIGUE ON THE DEGRADATION OF POLYMERIC MATERIALS

PhD Student,

Eng. Costel HUMELNICU

Scientific coordinator,

Prof. dr. eng. Ec. Elena MEREUTA

Series I6: Mechanical Engineering No. 59

GALATI

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Chapter 1

Introduction

1.1 Motivation on the choice of theme and objectives of the doctoral thesis

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We are witnessing a leap in the development of technology, created due to the development of polymeric materials and the boom that this industry has taken in recent years.

Making a comparison between established materials, such as ferrous or non-ferrous metals, wood, ceramics, stone, etc. and polymeric materials, we have observed that the latter have a number of advantages such as: low density, optimized electrical conductivity, good thermal insulators, high resistance to corrosion and chemical agents, low price, etc.

When it comes to thermal stress behaviour, the polymeric materials fall into two categories: thermoplastic polymeric materials (which can change their shape and condition when heated) and thermo-reactive polymeric materials, also called thermosetting (which can no longer be melted and reused after casting).

Epoxy resins are part of the thermosetting polymers, and due to their superior mechanical, electrical and chemical properties, they are among the most used in the production of composite materials. The properties of composites containing epoxy resins depend on the hardening agent, as well as the volume or mass used, but also on the materials used for reinforcement.

In this paper, we studied the lifespan of epoxy polymeric materials, under the simultaneous action of fatigue and wear. The study aimed to determine the degree of influence of wear on fatigue or how much and how the lifespan of the studied materials is influenced by the simultaneous action of the two stresses.

In this study, cylindrical samples made of pure epoxy resin and samples added with aramid powder or mineral oil were used and were subjected to heat treatments. The results of the study are used as input data in a software that uses neural networks to predict the lifespan of studied materials.

In order to achieve the main objective of this doctoral thesis, we have established a series of subsequent objectives:

- analysis of the current research on thermoset polymeric materials;
- creation of an experimental stand for fatigue and tribo-fatigue tests;
- preparation of epoxy resin samples;
- addition of epoxy resin in order to improve the mechanical properties;
- design and validation of a neural model for the prediction and optimization of fatigue and tribo-fatigue behaviour by wearing thermosetting epoxy resins.

In order to obtain results and conclusions, with implications on the synergistic effect of wear and fatigue processes on the degradation of polymeric materials, we applied a series of wider research methods, but also specific ones for engineering:

- the documentation method, for the study of research in the field of polymeric materials;

- the observation method, to observe and analyse the behaviour of fatigue and tribo-fatigue of thermosetting epoxy resins;
- specific mathematical methods, in order to identify and characterize the behaviour of the test specimens in fatigue and tribo-fatigue;
- experimental methods for the prediction and optimization of fatigue and tribo-fatigue behaviour with thermosetting epoxy resins.

1.2 Current research in the field of thermosetting polymeric materials

1.2.1. Thermosetting polymeric materials

Thermosetting polymers are formed by a crosslinking reaction that facilitates chemical bonding between macromolecular chains, creating a three-dimensional network [1]. These polymers, once formed, cannot be reused or remodelled by heating, as is the case with thermoplastic polymers. Thermosetting polymeric materials are often found in liquid form and are among the most used in the creation of composites reinforced with wires and fibres, powders, etc.[2], [3],[4],[5].

Some of the most used thermosetting polymers are:

- Phenols;
- Polyamines;
- Polyimides;
- Polyurethanes;
- Epoxy resins.

1.2.1.1 Phenols

Phenols are a class of organic compounds that have an aromatic nucleus. Their structure may include one or more hydroxyl (-OH) groups attached (grafted) on the nucleus. By combining phenol with formaldehyde and through a process called the condensation reaction, the first polymeric material appeared, having an opaque colour, also known as Bakelite, after the person who invented it, Leo Baekeland in 1907.[6], [7].

1.2.1.2 Polyamines

Polyamines are polymers that have in their composition an amino group (-NH₂), but through the condensation reaction between the hydrogen atoms in each group with the aldehyde molecules results in a polymeric material with totally different properties. Aminoplasts consist of urea-formaldehyde (UF), melamine-formaldehyde (MF) and melamine-urea-formaldehyde (MUF) [8], [9].

UF, MF or MUF polyamine resins can be mixed with various additives, pigments or fillers such as cellulose, wood or sawdust [10]. The finished products are obtained by heating the mixture and pouring it into moulds by injection, pressing or sintering. [8],[9].

1.2.1.3 Polyimides

Polyimides (PI) are synthetic resins, belong to the category of heat-resistant polymers and are used as matrix resins in fibre-reinforced composites [13]. The advantages of polyimide-based resins and composites are:

- high strength / weight ratio [10];
- thermal stability at temperatures up to 500o C [11];
- high mechanical properties [12],[13];
- good dielectric properties [14],[15];
- resistant to light and water absorption [16].

1.2.1.4 Polyurethanes

Polyurethanes (PUs) are organic polymers, which are formed by the reaction of a polyol (an alcohol with more than two reactive hydroxyl groups per molecule) and a polymeric diisocyanate or isocyanate, in the presence of suitable additives and catalysts [21]. Polyurethanes are a versatile class of polymers with special control over their physicochemical properties based on their chemical composition. [17].

1.2.1.5 Epoxy resins

Epoxy resins are reactive chemicals, synthetic in nature, in a liquid state, and in order to achieve finished products they must be cross-linked (cured) through polymerization, together forming a three-dimensional network. After polymerization, there is a high degree of cross-linking, so the nature of the interchain bonds gives the epoxy special characteristics [23]. The cross-linking is done with the help of compatible hardening agents or hardeners, resulting a solid compound, from which heat-resistant products with predetermined properties can be made.

Epoxy resins are used as matrices in a large number of composites, due to their adhesion properties with various materials and the large number of compounds that can react with the epoxy ring to form resin systems with a very wide range of properties.

Table 1.1 summarizes the advantages and disadvantages of epoxy resins.

Table 1. 1. Advantages and disadvantages of epoxy resins [18]

Advantages	Disadvantage
Increased adhesion on a wide variety of materials	Requires a long curing time
Low shrinkage compared to other materials (approximately 1% compared to 6% polyester)	Sensitive to cracks
It has good properties, especially in a humid environment	High price
High chemical resistance	Very strict conditions of use
Good resistance to high temperatures (150-190°C)	Aggressive products for the skin
Good mechanical properties (superior to high diffusion resins, such as polyester)	Sanding dust is toxic

1.2.2 Uses of thermosetting polymer materials

Due to the technical characteristics and mechanical properties of thermosetting polymeric materials, they are used more and more frequently in the aeronautical industry, the aerospace industry, the automotive industry, the energy industry, the naval industry etc.

Sustainability issues related to pollutant emissions are an important secondary benefit of epoxy resins by reducing their structural mass. Emissions of pollutants, and in particular CO₂, are being targeted by regulators and are becoming increasingly restrictive. Thus, electric vehicles, as well as their manufacture, will exponentially increase the demand for composite materials, which will counterbalance the weight of very heavy battery packs, resulting in lower emissions. [19],[20].

The versatility of epoxy resins results from their ability to be chemically and physically compatible with a wide variety of compounds [27], including polyamides, thiols, imidazoles [28] and, more recently, ionic liquids. [21].

1.2.3 The main loads faced by thermosetting polymeric materials

The stresses on thermoset polymeric materials [30] have long been studied on "conventional" materials [31], [32]. Many of these studies have been and are being carried out in the field of mechanical testing [33], [34], in order to determine whether the materials in question meet the operating and exploiting conditions as a finished product. In addition to these demands, there are also fatigue demands cumulated with wear, studied in this thesis.

1.2.3.1 Fatigue loads

Engineering practice has shown that the components of machines and gears are subjected, over time, to variable loads. This results in the rupture of those parts, although the maximum stresses acting on them is lower than the breaking strength or even the elastic limit of the material. The phenomenon is called fatigue, the respective mechanical characteristics - fatigue limits or fatigue strength, and damage is called fatigue rupture [22],[23].

For metals, the fatigue process is generally well understood, as it is known that displacements in metals occur at the level of the crystalline structure, and research in this field is at an advanced stage. Unfortunately, the same cannot be said about the fatigue of polymeric materials, as their molecular structure is completely different and, therefore, it is very difficult to obtain a pattern in the process of crack initiation. In theory it can be stated that from the moment a crack is initiated, its subsequent propagation is similar to metals. [24].

The lifespan of a part is usually estimated by the number of cycles performed to break and is denoted by N . The lifespan is determined by subjecting a batch of specimens to cyclic stress, the first of which is at a maximum voltage lower than the breaking strength of the material R_m , the others being charged with a maximum voltage lower than the previous ones. Showcasing in a graph the values σ_{max} and N , we obtain the Wöhler curve, also called the fatigue durability curve, similar to the one shown in figure 1.1.

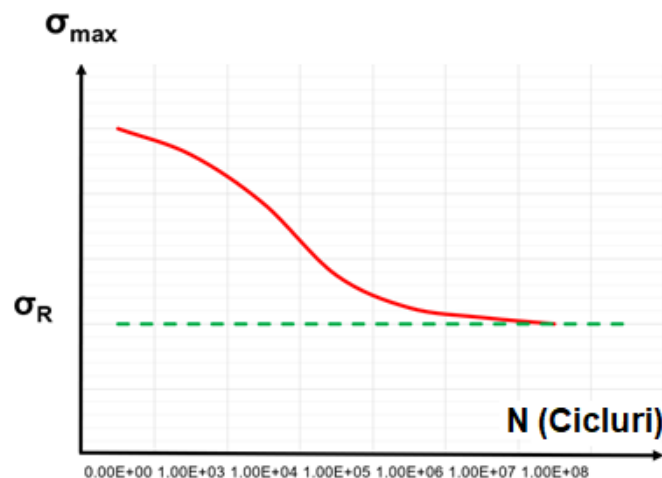


Figure 1. 1. Wöhler curve specific to some steels. [25]

In the case of fibre-reinforced polymeric composites, the study of fatigue is more complex than in the case of metals, due to the fact that fatigue destruction occurs in four stages, namely: matrix cracking, interface decomposition, delamination and fibre breakage [39]. These conclusions are based on the performance of many fatigue investigation tests, and based on them, several models of linear and nonlinear destruction have been developed. [26],[27],[28],[29],[30],[31].

Fatigue tests performed on a sample of laminated materials, composed of nanocomposites composed of epoxy / carbon and modified with nylon nanofibers 66 [46], showed that the rate of crack formation decreased significantly, which leads to an increase in lifespan of the nanocomposites in question.

1.2.3.2 Wear loads

The wear resistance of a material is as important as the mechanical properties of that material. Wear is characterized by a loss of material on the contact surface of a part when it is subjected to a relative movement in contact with another part. [32].

When it comes to thermoplastic polymeric materials, several wear mechanisms have been identified, namely adhesive wear, abrasive wear, surface fatigue, chemical wear and thermal wear [48], [49]. In the case of thermosetting polymers, the most discussed wear mechanisms are abrasion and adhesion.

The most relevant tribological parameters, which are most often used in wear calculations, are the wear rate and the coefficient of friction [50]. The wear rate is the loss of volume per unit distance and is independent of the applied load. The coefficient of friction is the ratio between the slip resistance force and the load force and determines the torques and loads in contact.

Through different techniques for measuring and analysing the parameters of wear, it is possible to achieve a classification and an evaluation of tribosystems according to their characteristics of durability and reliability [51]. Due to the fact that wear occurs as a result of the interaction between two components, part of a tribosystem, the parameters of wear are not related to a single material, but are always related to the pair of materials, in fact, to the whole tribosystem [52]. Usually, the complexity of the wear processes leads to the impossibility of modeling its parameters, but they can be determined experimentally.

1.2.3.3 Compound tribo-fatigue loads

Wear and tear are factors that make a significant contribution to the deterioration and destruction of a mechanical systems. The academic literature offers numerous explanations on how these phenomena act on metallic materials, them being studied and researched independently. From 1985-1986 a group of researchers in Belarus began to study the effects of damage to mechanical systems due to the simultaneous action of wear and tear. This science, which is a subdiscipline of mechanics, has been called "Tribo-fatigue". [33].

Types of damage caused by wear and tear:

- *Fatigue-slipping*
- *Fatigue-running*
- *Fatigue-fretting*
- *Corrosion fatigue*
- *Fatigue-erosion*
- *Fatigue-corrosion-erosion*

The analysis of the basic integral characteristics of fatigue strength, meaning the fatigue curve, is based on the following three criteria [53]: volume deterioration; surface damage; damage and destruction caused by wear and tear.

Volume damage

According to the model of a deformable body, subjected to tribo-fatigue, damages occur when the strength limits, corresponding to the two loads, both individually and simultaneously, are reached at a dangerous level.

Surface damage

Surface damage occurs during the interaction of contact between two solid bodies pressed by a contact load and in a process of relative motion to each other. These bodies are a friction pair.

Determination of the characteristics of damage and destruction caused by tribo-fatigue

The specific phenomenon of wear and tear occurs when at least one of the members of a friction pair undergoes a cyclic deformation of the volume, so that the contact stresses as well as the non-contact stresses are induced simultaneously in the friction zone.

Most current machines and equipment have mechanical systems with which movements, forces and moments can be transmitted.

Determination of the characteristics of damage and destruction caused by wear and tear of materials or even structures can also be done using finite element analysis software [54]. These software make it possible to design and test virtual elements or even assemblies, thus saving time, space, materials etc.

1.3 Partial Conclusions

- Studying the specialized literature in the field of polymers, it is observed that there are a multitude of study materials (books, magazines, conferences, etc.) that describe research and experiments conducted in this field, thus confirming the topicality, increased interest and development potential.
- With the advent of polymers, new composite materials have been developed, with mechanically improved properties, advantages that recommend them for use in all industries.
- Recycling of thermosetting polymers is much more difficult because after polymerization they can no longer be subjected to second processing and no viable methods have been found to achieve technologies that allow their reuse, as is the case with thermoplastic polymers.
- The polymeric composite materials can have their properties “modelled” from the design-preparation phase, so that a series of advantages can be obtained such as: low density; high tensile strength; very low coefficient of expansion; high shock resistance; high durability etc.
- Polymer composites have a number of disadvantages, such as: high cost; does not show ductility; they cannot be easily reconverted into the raw material from which they were generated; inorganic components of composite materials cannot be reused; high flammability etc.
- Depending on the materials used, polymer composites may behave similarly, or better, than metals. For example, graphite composites have an increased resistance to frictional wear (better than bronze).
- For polymeric composite materials it is difficult to perform a modelling or simulation of the wear parameters due to the complexity of these processes.
- Significant reduction of the coefficient of friction and the wear rate can be done by adding a coating on the polymer composite, so as to provide a self-lubricating feature.
- Although the literature offers a wide range of studies on the determination of cumulative tribo-fatigue of metallic materials, in terms of thermoset polymeric materials, there are few studies to determine the fatigue limit, but no studies to highlight the cumulative effects of tribo-fatigue on these materials.

Chapter 2.

Methodology for testing epoxy resin-based polymer materials at compound wear fatigue stresses

Through the experiments conducted in this thesis, I analysed a series of polymeric materials, which were subjected to concomitant tribo-fatigue to determine how wear influences the lifespan of a material. Several samples of the specimens were tested, in a first phase to fatigue, after which they were also subjected to tribo-fatigue, caused by sliding friction.

2.1. Methods of investigating and determining fatigue

The ability of a material to withstand breaking when subjected to time-varying stresses is called fatigue strength.

Compared to the fractures produced by static stresses, the fatigue rupture has a specific appearance with two areas: a glossy area and a grainy area with sharp crystals resulting from the sudden breaking of a fracture.

Studying the academic literature, the most used method for determining the fatigue of materials for the symmetrical and pulsating alternating cycle is the one based on the analysis of stresses having as a basic element the tracing of the Wöhler curve.

Sosnovskiy L. A. explained in his book [55], starting from practical examples, how the performance of a specific mechanical system is evaluated, which is frequently exposed to damage caused by tribo-fatigue.

Another method of determining the fatigue limit was used by Fabio Giudice *et al.* [56], who tried to define the fatigue parameters of a commercial steel (C 45) under static and cyclic loading conditions $R = 0$, using a thermographic method and comparing the results with other tests performed by other methods.

Another approach in identifying the causes that lead to the destruction of parts and subassemblies produced from various materials and subjected to variable cyclic stress is by the method of detecting microcracks in those materials, using acoustic emissions [57].

In another study, Solberg, K. *et al.* [58] analysed the fatigue behaviour of mechanically processed, finished and subsequently heat-treated Inconel 718 samples, mechanically produced, finished and thermally treated.

Laurent Gornet *et al.* [59] tested the fatigue limit of an epoxy matrix composite reinforced with unidirectional carbon fibres. They also used another method of estimating the fatigue limit, the so-called "self-heating test" method.

The same method for determining the fatigue limit was used by Catherine Peyrac *et al.* [60], testing a composite material consisting of a thermoplastic polymer matrix (PA66) and reinforced with carbon fibre fabric.

Other methods that are used to determine the fatigue of materials from the design phase of some elements and subassemblies are those that use dedicated computer design software (CAD Computer-Aided Design).

Using the CAD - SOLIDWORKS program, I designed two planetary shafts used for Dacia Pick-up vans with the results being published in the article 'Re-Engineering of a planetary shaft based on lifespan' [61]. The purpose of this research was to subject two different types of car planetary gears (one with a flange on the wheel hub and the other with grooves, "rod type") to various demands to determine their durability. The idea from which this study started was that the first planetary gear used in the side transmission of Dacia Pick-up cars, the one with flange, had an inconvenience, reason for which it was replaced. After modelling the two types of planetary gears in the SolidWorks program to their accurate dimensions and shapes, they were subjected to a finite element analysis simulation. The simulation consisted in subjecting them to real bending forces and torsional moments, of static, cyclic constant and cyclic variable variables. The results showed that both types of planetary gears have unlimited durability, but from a mass point of view, one had a mass about 50% lower than the other.

The conclusions of this study showed that both types of planetary gears meet the theoretical conditions of design, sizing and choice of manufacturing material and, theoretically, have unlimited durability. Another conclusion was that the "rod" type planetary has about half the weight of the other planetary. This results in many advantages for using a lighter planetary, primarily financial (low weight = small volume and size, simpler processing = lower cost). If the planetary gear is smaller, the other subassemblies of the transmission (differentially, the wheel hub) have automatically been resized, reducing their weight, all this leading to a reduction in the total weight of the vehicle and an increase in its load capacity.

Another study I conducted to determine the lifespan of some materials using a CAD program, was presented at the international conference IManEE 2018 and published in the article entitled '*Fatigue life investigation on a MAC engine piston*' [62]. The study aimed to investigate the influence of the shape of the head, as well as the materials from which the pistons used in diesel engines are made, as well as their optimization when it came to material fatigue. To achieve these goals, the finite element analysis method was used, which is an integral part of the SolidWorks software. First, two pistons were designed, with two different shapes of the head, one having the combustion chamber in the centre of the piston head, and the second having the combustion chamber offset from its vertical axis. Two different materials were also used, namely aluminium alloy and alloy steel. After design, these pistons were subjected to fatigue simulations and kept for comparison. A copy of them was subjected to repeated fatigue simulations, and after each simulation changes on the dimension were made and, as far as possible, on the shape (without affecting the basic characteristics of the pistons and the required dimensions), up to the point in which, the finite element analysis showed that they no longer correspond in terms of material durability.

The conclusions showed that after undergoing fatigue simulations of the four pistons, two made of aluminium alloy and two alloy steel, the aluminium pistons have similar characteristics (lifespan and minimum safety factor). Regarding the alloy steel pistons, it was observed that all the resulting parameters for the pistons for which the shape was optimized, are much improved and, more importantly, its mass was considerably reduced by about 40%.

Using a design software has many advantages such as: the program provides an intuitive interface, does not require many operators, at any stage of the model can resize a certain segment or even the entire part, designing and testing parts from the drawing phase, existence a library where you can choose a wide range of materials with all their characteristics, it creates a detailed report that includes all the information about the projected part, etc.

2.1.1. Devices used for fatigue tests

Fatigue tests are performed on materials which, during usage, are subjected to repeated stresses in order to reduce their rigidity, their strength and to determine the total number of duty cycles to destruction.

For performing fatigue tests, depending on the type of demands, a wide range of stands are used, some with a simple construction, being dedicated only to this type of testing and used for laboratory tests or others with a more complex construction, which can perform a diverse range of tests.

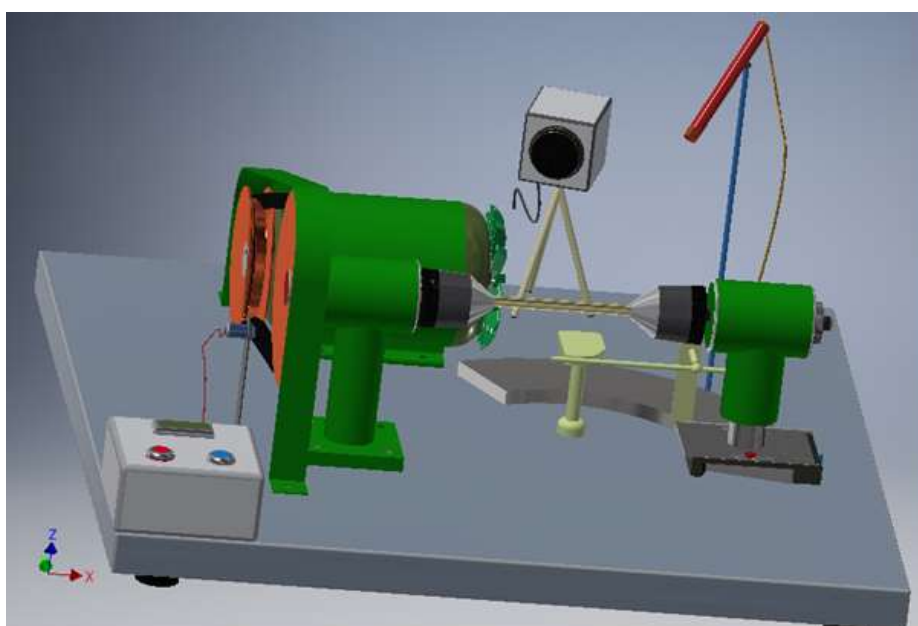
We can mention: the Amsler 150 HFP 5100 stand produced and marketed by ZWICK / ROELL [63], the one produced by ZWICK / ROEL, the fatigue test stand, developed and delivered by Amsler Prüfsysteme (Schweiz) AG, which is a [64] UBM stand 200tC, a laboratory stand used to determine the fatigue strength of circular cross-section metal specimens subjected to alternating stresses by rotary bending in accordance with DIN 50113, which regulates this type of tests [64]. We can also mention the fatigue testing device of thermosetting polymeric materials designed and made by Gheorghe V. and Purcărea R [65]. Ujjwal Makkar *et al.* [66] determined the Wöhler curve for two composite materials, the first material being an epoxy resin, reinforced with glass fibres, and the second material made of epoxy resin and carbon fibres. To achieve this, the study authors used cylindrical specimens, which were subjected to rotational bending fatigue tests.

2.1.2. Proposed constructive solution

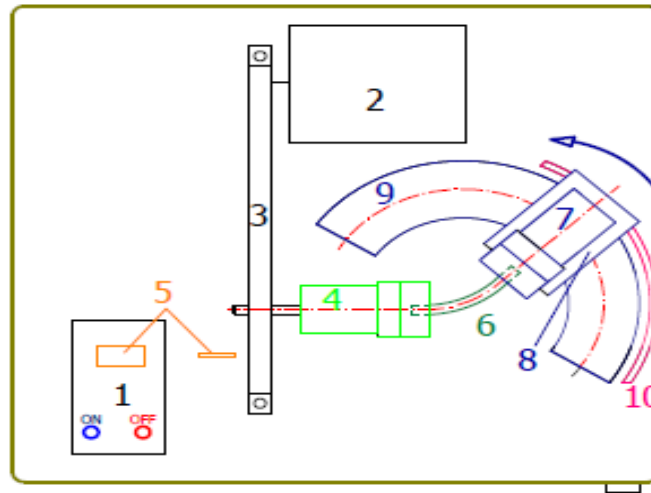
For fatigue testing, in laboratory conditions, of cylindrical samples, a stand was created with the help of which several samples were subjected to variable rotational bending stresses, and the obtained results were used to determine the Wöhler curve, tension- number of cycles (SN) specific to the tested material.

The stand consists of a metal platform made of rectangular pipe of 30x20x1.5 mm, has a square shape with a side of 550 mm and is covered with 5 mm sheet, is supported on 4 rubber legs, and the total height is 50 mm. The other component parts that make up the stand are arranged on the platform, respectively: a control panel, an electric motor, a transmission belt provided with a metal guard, two mandrels, a sliding surface of one of the mandrels, a graduated ladder.

Figure 2.1a shows the sketch of the stand, with the test piece at rest, and figure 2.1b shows the sketch of the stand with the test samples inclined at a certain angle.



a)



b)

Figure 2. 1. Sketch of the stand: a) with the sample at rest, b) with the inclined sample. [67]

The operation principle of the stand and the operation mode are performed in a way that allows the performance of fatigue tests with minimal financial resources and easy handling. The device is put into operation by turning the "ON" button located on the control panel (1), at which time the electric motor (2) is powered. Through a belt which is protected by a housing (3), it transmits the rotational movement to the shaft on which the fixed chuck is located (4), the rotation is recorded by a device provided with a magnet attached to the shaft pulley, an electromagnetic contact and a digital display screen (5). One end of the sample (6) is fixed in the fixed chuck and the other end in the movable chuck (7), the chuck rotates due to the sample and when it breaks the mobile chuck stops. In turn, the mandrel 7 is gripped by a bolt, which ensures that it rotates around the vertical axis on the sliding support (8).

The stress in the sample is achieved through bending, this is being done by moving the support 8 on the sliding surface in the form of a semicircle (9), and the degree of inclination is shown on the graduated scale (10) [67]

In order to monitor the ambient temperature and the samples during the tests, the stand is equipped with an Optris PI 160 thermal imaging camera (figure 2.2 a), and to observe the behaviour of the samples at wear, the stand also has a camera from Supereyes (figure 2.2 b). Even more, to observe some elements in detail, a digital microscope was used whose lenses could adjust the 1X-20X optical zoom.



a)



b)

Figure 2. 2. Monitoring devices: a) thermal imaging camera, b) video camera.

Figure 2.3 shows the stand with all the modifications made, in order to eliminate the inconveniences found during the first tests.



Figure 2. 3. Fatigue test stand.

1- Control panel; 2 - Electric motor; 3 - Belt housing; 4 - Fixed chuck; 5 - Device for reading and displaying rotations; 6 - Test tube; 7 - Mobile chuck; 8, 9 - Sliding device of the movable chuck; 10 - Device that facilitates the concentration of stress in the middle of the samples; 11- Thermal imaging camera; 12 - Video camera

2.2. Tribo-fatigue test methodology

Wear occurs as a result of interactions between surfaces in contact and is manifested by the deformation and removal of a piece of material from one or even both surfaces.

To perform simultaneous tests, wear and tear, new classes of test equipment have been designed and developed [68], [69], [70]. Figure 2.4 exemplifies the principles by which these test stands were developed and the methods by which fatigue wear tests can be performed when the main source of mechanical fatigue occurs as a result of repeated and concomitant bending and rotating processes, which they are found in almost all modern machines and equipment [71], [72], [55].

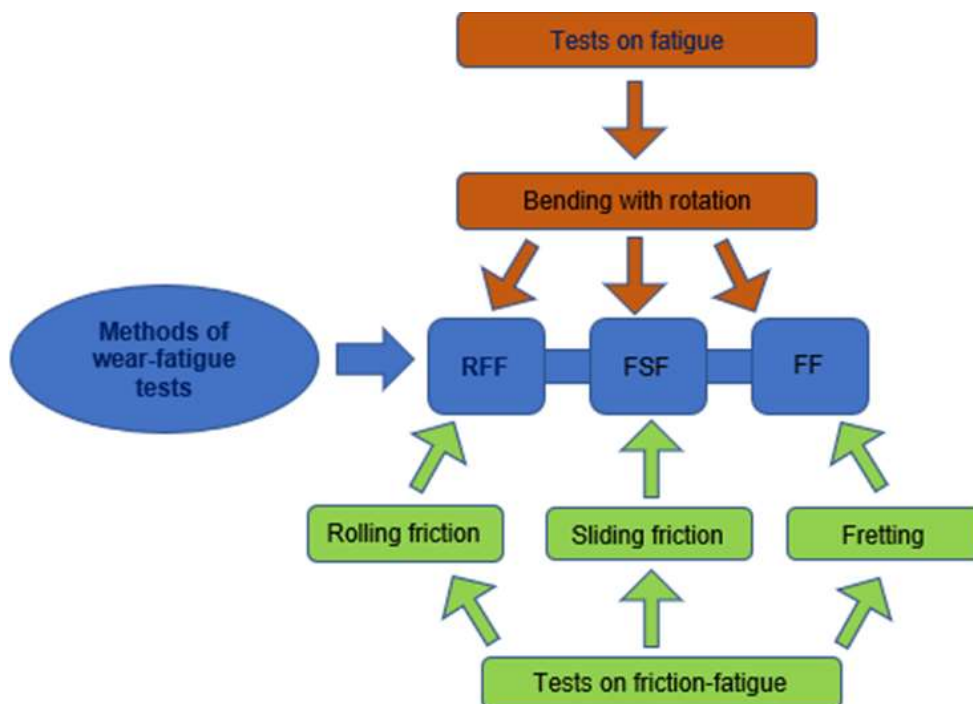


Figure 2. 4. Development of methods for wear-fatigue during the main rotational movement: OFR - rolling friction fatigue; OFA - fatigue with slipping friction; OF - fretting fatigue [55], [70]

2.2.1 Stands used for compound stress tests

In the early 1990s, a test stand called "SI-OM" was designed (after the initials of the names of those who made it: Sosnovskiy and Indman), and later several modifications were made to improve its performance.

2.2.2. Proposed constructive solution

The previous chapter has shown various methods, stands and research conducted both in terms of fatigue and wear of polymeric materials and composites based on polymeric resins.

Analysing the academic literature, it can be seen that no studies are performed on the tribo-fatigue of polymeric materials or composites based on thermosetting polymeric resins or, rather, on how wear affects the lifespan of thermoset polymeric materials.

In order to perform these types of test, a device (figure 2.5) was attached to the stand made for fatigue tests, described in subchapter 2.1.2, which allows a sliding friction being performed on the surface of the sample.

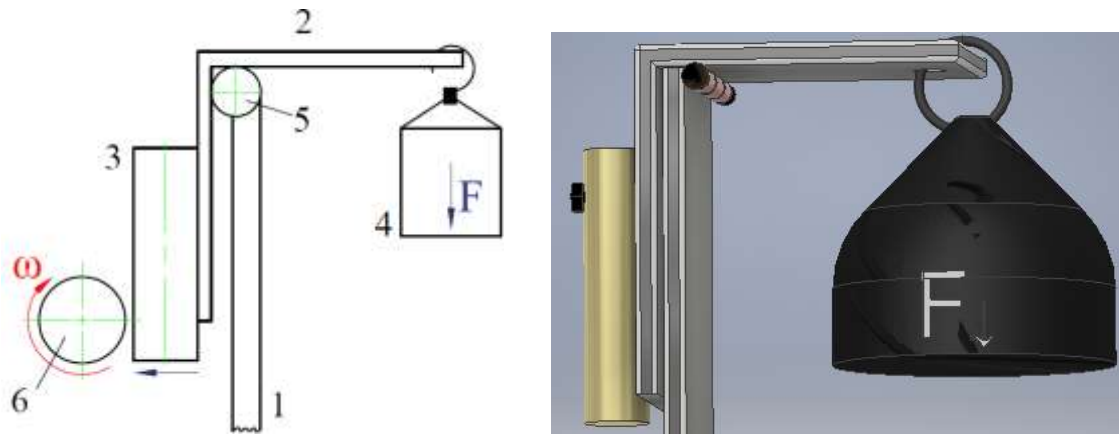


Figure 2. 5. Device for achieving wear by sliding friction.

Figure 2.6 shows the stand that has attached the device with which the frictional wear is performed.



Figure 2. 6. Device for generating wear.

Counterpart 3 can be made of various materials, has a circular section, a diameter of 6 mm and a length of 25 mm.

2.3. Materials and processes used

2.3.1. Materials to be tested

The samples necessary for this study were made of epoxy polymeric resin Epiphen RE 4020 (reactive prepolymer with epoxy groups, with 75 ... 78% bisphenol A), in a proportional mixture with the hardener recommended by the manufacturer, DE 4020 (modified aliphatic amines), and in the case of composite materials, the matrix was made of this resin, which is produced by R&G Gmb Waldenbuch.

Additive epoxy polymeric materials

In these studies, powder or liquid additives were used to improve the mechanical properties of the epoxy resin used.

Aramid powder additive

To create a composite material with improved mechanical properties, we opted for the addition of Epiphen RE 4020 epoxy resin and aramid powder hardener DE 4020, produced by Dhingra Plastic & Plastiscisers Pvt. Ltd. Some characteristics of aramid powder are: high tensile strength, good shock-absorbing properties, increased fatigue strength, low density and zero thermal expansion [73], which should improve its mechanical and thermal properties.

Synthetic oil additive

In order to achieve a composite material with improved mechanical properties, both for fatigue and wear, Epiphen RE 4020 epoxy resin with hardener DE 4020 was introduced, an additive based on synthetic oil, in various proportions, produced by TOTAL ROMANIA SA.

2.3.2. Description of the process for obtaining the samples

The process of forming epoxy matrix composites must go through several steps: [74]

1. analysis of the properties and characteristics of resins;
2. establishing the dispersion method;
3. determining the fraction;
4. establishing the necessary samples;
5. establishing the type of moulds;
6. completion of the process;
7. selection of samples.

In the forming process, the polymer matrix passes through three transformation phases (figure 2.7): in the first phase, it is a fluid / liquid material (initial state); in the second phase it is transformed into a gel-like material (polymerization period); In the final phase of the polymerization process it is transformed into a glassy material (final state). [75]

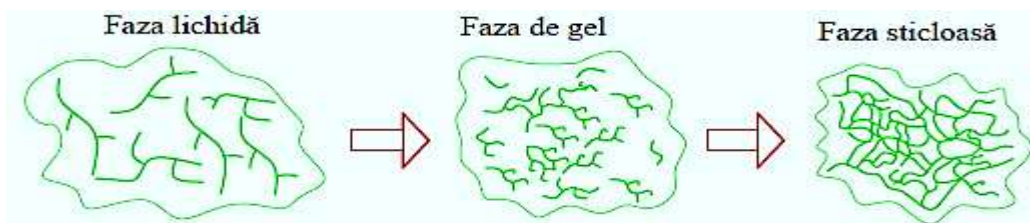


Figure 2. 7. The phases of transformation of the polymer matrix. [75]

Polymerization of epoxy matrices is fast, occurs at ambient temperature, without the release of volatile by-products.

Applying of heat treatment is an important step, because this operation has the role of eliminating the residual stresses in the volume of the material formed, giving it a state of equilibrium, as well as improving its mechanical and thermal properties.

The mixing time of the two components (figure 2.8) was 10 minutes and was performed using a mixer, at a speed of 60-70 rpm (a higher speed would introduce air into the composition). These parameters were established based on tests previously performed at different speeds and times. After mixing, the mixture was not stirred for 15 minutes to facilitate air removal. Given that the handling time of the mixture is a maximum of 40 minutes from the start of the mixture, the moulding procedure should be fast but, as far as possible, without introducing air inside, as its shape and length do not elimination of air bubbles.



Figure 2. 8. Hardener DE 4020 (A), Epiphen RE 4020 (B).

The samples used in this study were cast in tubular moulds, have a length of 120 mm and a diameter of 8 mm. The clamping of the samples in the mandrels was performed on a length of 20 mm, at both ends, so that the effective test length was 80 mm.

To make a polymeric composite, a certain amount of additive material (in volume or mass proportions) must be introduced into the polymer resin. In the case of epoxy resins with hardener, the additive may be dispersed in one of the two components or in the pre-polymer mixture [76], [77].

2.4. Planning and conducting tests

The current research aims to determine the influence of wear on the fatigue of thermosetting polymeric materials or thermosetting polymeric composites. In order to achieve this goal, a series of samples made of Epiphen RE 4020 epoxy resin, with DE 4020 hardener, as well as samples obtained by the addition of this resin, with additives in the form of powders or liquids, being subjected to tests, five specimens from each sample, for each degree of bending of the samples.

More than 1,000 samples were created, most of which were used for actual fatigue and tribo-fatigue.

The tests took place over 17-18 months, taking into account that at an inclination of the mobile chuck of 8 degrees, the duration of a single test is about 22 hours.

Determination of the standard and influence of the polymerization method

In order to perform these studies, first of all, a series of samples were created only from Epiphen RE 4020 epoxy resin, with hardener DE 4020. These samples were subjected to fatigue tests, performed respecting the same temperature conditions, between 24^o C and 25^o C.

To determine whether the polymerization method has a significant influence on fatigue stress, three polymerization schemes were applied, of which those subjected to three-

stage heat treatment are clearly superior to those naturally polymerized at room temperature, or those to which they were treated. the heat treatment scheme was applied in a single step.

Therefore, the results obtained for the heat-treated three-stage samples were used as a benchmark to compare the results of subsequent fatigue tests as well as those of tribo-fatigue.

Determining the influence of additives

Two types of additives were used to obtain composite materials with improved properties at fatigue stress and the accumulated tribo-fatigue, the first being aramid powder and the second a synthetic oil.

During the study, several specimens were created, made of epoxy resin Epiphen RE 4020, with hardener DE 4020 and added with aramid powders in different mass proportions (4%, 3% and 2.5%, the latter having results conclusive), which have been subjected to different polymerization methods.

Another composite material tested was made of Epiphen RE 4020 epoxy resin with DE 4020 hardener, added with synthetic oil. Preliminary tests showed that the best fatigue performance was obtained when the amount of additive added was 3%. (mass amounts of oil were inserted: 5%, 4%, 3%).

Determining the influence of wear on fatigue

All materials used in this study were primarily subjected to fatigue stress, after which they were subjected to tribo-fatigue tests, in order to determine to what extent, or whether, wear influences the tribo-fatigue strength of that material.

Tribo-fatigue tests were performed on two sliding friction couplings: a polymer-polymer coupling (figure 2.9 a) and a polymer-metal coupling (figure 2.9 b) and were performed with the normal force of 5N (force at which the results were conclusive), respecting the same temperature conditions (24°C - 25°C.).

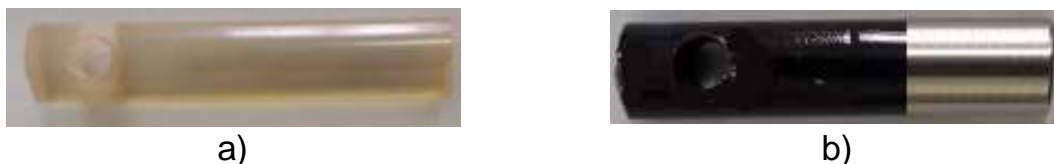


Figure 2. 9 Friction counterparts a) made of polymer b) made of steel.

2.5. Partial conclusions

- Fatigue testing of epoxy polymeric materials is useful because it can anticipate the lifespan of a product made of that material.
- The stand made for fatigue tests, using the method of subjecting the samples to rotational bending stresses, is compact, offers easy manoeuvrability and has smaller dimensions, compared to stands that use weights to induce fatigue stress.
- The speed of rotation of the sample must be adapted in such a way as to avoid self-heating of the material due to the internal stresses formed.
- The implementation of a device made of a rod, a cam and a spring, made it possible to concentrate the stresses in the middle of the samples.
- Making fine cuts, with a cutting device, aims to concentrate the tensions in the middle of the length of the samples, when the angle of inclination is greater than 15 degrees.
- As a result of the improvements made, the stand can be considered suitable for performing fatigue and cumulative tests – tribo-fatigue, on epoxy materials.
- The materials used to add the epoxy resin (aramid powder, synthetic oil) interacted favourably with it, due to the optimal dosage and the way of mixing,

- Following the preliminary tests, a standard could be developed, which was later used to compare the other results.
- Following the preliminary tests, both the optimal quantities of additives that were introduced into the epoxy resin and the maximum pressing force of the friction counterpart were determined.

Chapter 3.

Obtained results

3.1. Tests in fatigue

3.1.1. The influence of the polymerization method on fatigue strength

The first tests were performed to determine the extent to which fatigue strength is influenced when different polymerization methods are applied. For this purpose, two sets of specimens made of simple epoxy resin (Epiphen RE 4020 with hardener DE 4020) which were prepared under similar conditions but polymerized through different methods. For the first set, the natural polymerization method was applied, hereinafter referred to as RNP (the polymerization process was carried out at room temperature, 22-23° C), and the time difference between casting and the date when they were tested was 60 days. The second set, after polymerization at room temperature, was subjected to a three-stage heat treatment, and the fatigue test was performed 21 days after the application of the heat treatment (hereinafter referred to as RTT).

The methods used, the data obtained and the conclusions developed in this subchapter, entitled: The influence of the polymerization method on fatigue resistance, were presented at the SGEM 2020 international conference in the article "Reduction of polymeric waste by applying heat treatments during the polymerization period" [67] and published in the conference volume.

One of the first observations, after casting and before the start of testing, was that the heat-treated specimens had a darker shade than those naturally polymerized (Figure 3.1).



Figure 3. 1. RTT sample (A), RNP sample (B).

Using the thermal imaging camera, the evolution of the temperature of the sample subjected to fatigue was monitored. Temperature monitoring during testing is important because thermosetting polymeric materials self-heat due to internal friction that occurs as a result of the stresses to which they are subjected to [78].

One aspect, found during the fatigue tests of the RTT samples, is that at an angle of inclination of the movable mandrel greater than 15-16 degrees, the rupture has the shape of a chip, visible in Figure 3.2, a sign that the tension is high and the rupture occurs suddenly.

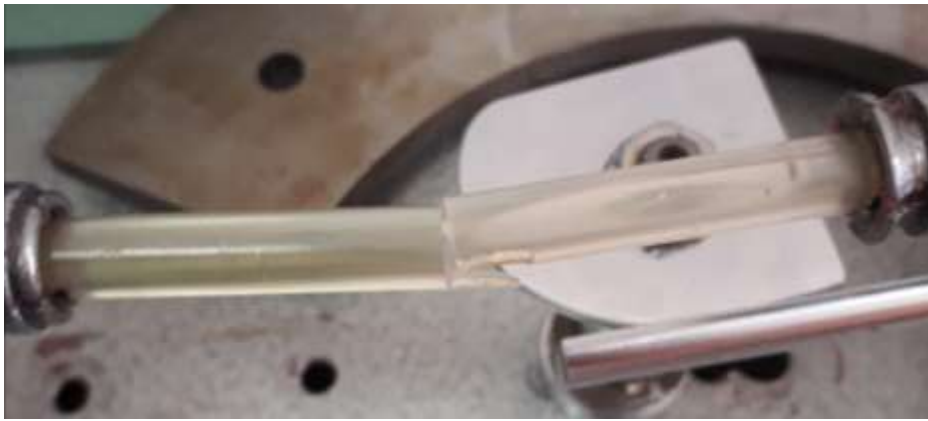


Figure 3. 2. The shape of the rupture of the RTT specimens, bent at over 15-16 degrees.

To determine the Wöhler curve, a series of samples were tested for each angle of inclination. The angles at which the samples were inclined at, as well as the rounded average values of the resulting number of cycles are shown in Table 3.1.

Table 3. 1. Average of the results obtained after performing the fatigue test.

Angle of inclination (°)	RNP (cycles)	RTT (cycles)	σ_{\max} (MPa)
8	96000	1250000	35.8
10	37000	132000	47.7
11	22000	75000	57.3
12	19500	29000	59.7
13	8000	25500	63.2
14	5500	15500	65.6
15	4100	12000	69.2
16	3000	9500	74.1
17	2200	6500	77.6
18	1700	4700	78.8
19	1200	2900	87.2
20	650	1500	90.7

No tests were performed below the angle of 8 degrees because the heat-treated samples passed 10^6 cycles, and this is considered acceptable for polymeric materials [79], [80].

The determination of the maximum transverse stress σ_{\max} was possible knowing the force applied for the bending of the samples, and the results are presented in table 3.1 and are shown in MPa.

Based on the results obtained and the data in Table 3.1, the Wöhler curve, voltage - number of cycles (S-N), was drawn for the two types of samples subjected to the fatigue test.

Figure 3.3 shows the S-N curves for the RNP and RTT samples.

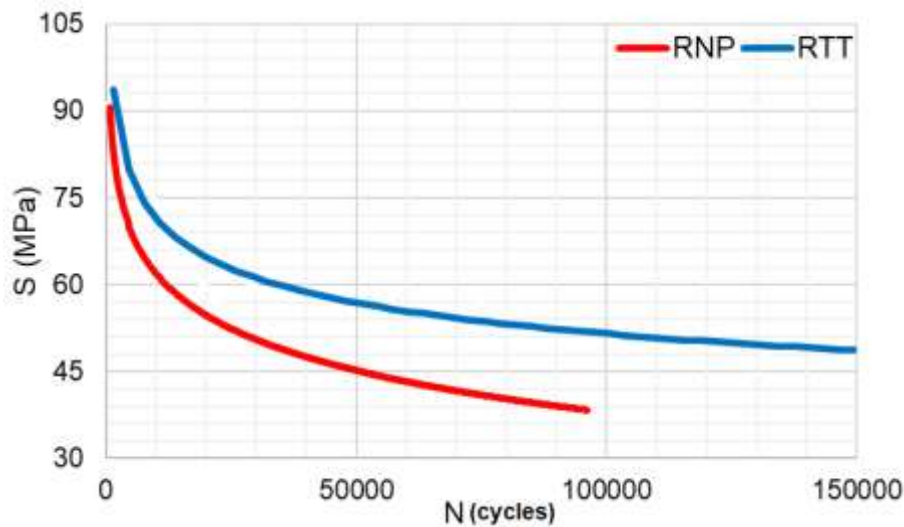


Figure 3. 3. Wöhler curves for RNP and RTT samples.

3.1.2. The influence of additives on fatigue strength

Aramid powder additives

Aramid powder in a mass proportion of 4%.

The methods used, the data obtained, and the conclusions of these tests were published in the article "Fatigue Polymeric Materials - Air Pollution Factor" [81], an article presented at a conference and published in its volume. The study started from the premise that the polymer materials industry has experienced a great growth due to the advantages they have, but if these materials are not recyclable or recycled, they can cause very great damage to the environment. Therefore, the article addressed the reduction of pollution caused by these materials, through laboratory studies, which will find more possibilities to increase the life of parts and subassemblies made of epoxy materials, so that the destruction caused by fatigue is delayed as much as possible.

The preparation of the samples is similar to that described in subchapter 2.3, with the exception that 4% weight of aramid powder was added after the epoxy resin and hardener had been mixed for 10 minutes (hereinafter referred to as RA 4%). After natural polymerization, the samples were subjected to an 8-hour heat treatment at a temperature of 60° C [82].

Table 3.2 shows the results obtained from the fatigue testing of the two types of samples, as well as the maximum stresses, depending on the angle of inclination.

Table 3. 2. Average of results obtained for RP and RA 4%.

Angle of inclination (°)	RP (cycles)	RA 4% (cycles)	σ_{max} (MPa)
15	2500000	650000	47,694
16	1250000	293000	48,887
17	850000	150000	51,271
18	250000	43000	56,836
20	31000	11600	62,639

From the data presented in table 3.2 it is observed that the RP samples have on average a longer lifespan than the RA 4%, by approximately 23.5%. The data obtained and presented in table 3.2 were used to draw the S-N curves, for the two types of samples, curves visible in figure 3.4.

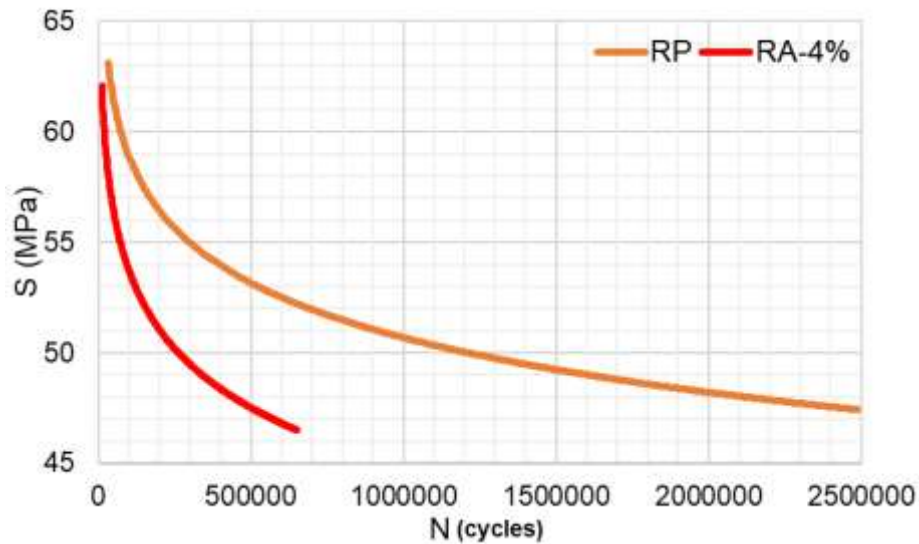


Figure 3. 4. Wöhler curves for RP and RA 4%.

Aramid powder in a mass proportion of 2.5%.

Another experiment performed in the thesis, was to obtain a composite material having as matrix the epoxy resin Epiphen RE 4020, with hardener DE 4020, added with aramid powder. Unlike the RA 4% specimens, the three-stage polymerization scheme was used for the newly created ones because, as it was found in the previous tests (RTT and RA 4%), this scheme led to the increase of fatigue resistance.

To achieve this goal, several samples were created, with different mass proportions of aramid powder: 5%, 4%, 3% and 2.5%. As the preliminary tests performed on the 5%, 4% and 3% samples had nonlinear results, the following studies were performed on the 2.5% aramid powder additive sample, hereinafter referred to as the 2.5% RA.

The averages of the results obtained are presented in table 3.3.

Table 3. 3. Average of fatigue results for RA 2.5%.2,5%.

Angle of inclination (°)	RA 2,5% (cycles)	σ_{max} (MPa)
8	1110000	35,8
10	121800	47,7
11	69700	57,3
12	25400	59,7
13	22800	63,2
14	14300	65,6
15	8300	69,2
16	6000	74,1
17	4600	77,6
18	3300	78,8
19	2100	87,2
20	780	90,7

Figure 3.5 shows Wöhler curves for the composite material obtained from epoxy resin Epiphen RE 4020 with hardener DE 4020, added with 2.5% aramid powder (RA 2.5%) and RTT.

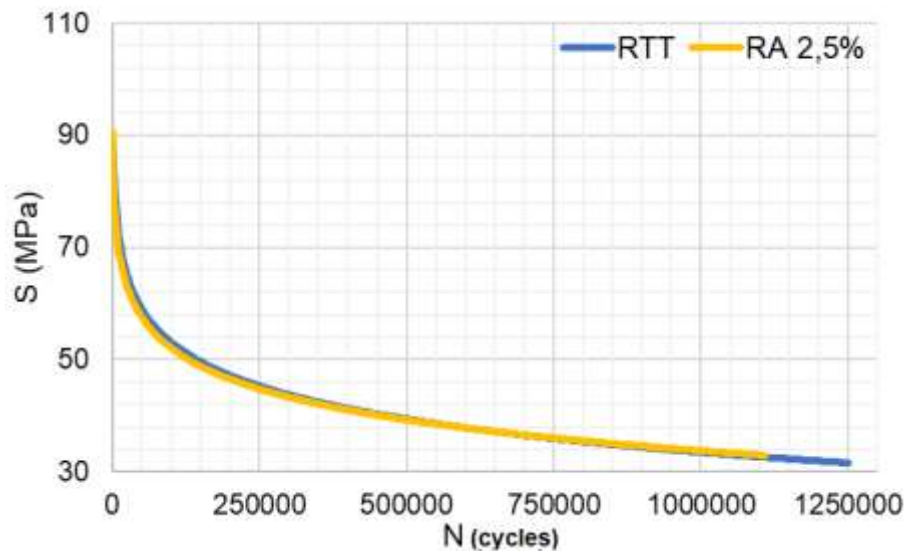


Figure 3.5. Wöhler curves for RTT and RA 2.5%.

Analyzing the graph in figure 3.5, it is observed that, from the point of view of fatigue, both types of samples follow the same S-N curve, there being no major differences between RTT and RA 2.5%.

One aspect observed in the samples containing 2.5% aramid powders was that the temperatures accumulated during the fatigue stress by rotating bending accumulate locally, the dissipation of these temperatures throughout the mass of the specimens is slow.

Figure 3.6 shows a 2.5% RA sample, inclined at an angle of 15 degrees, and from the temperature graph above the sample piece we can observe the temperature distribution in its mass, the highest temperature being recorded in the middle of the samples piece, where the demand is maximum.

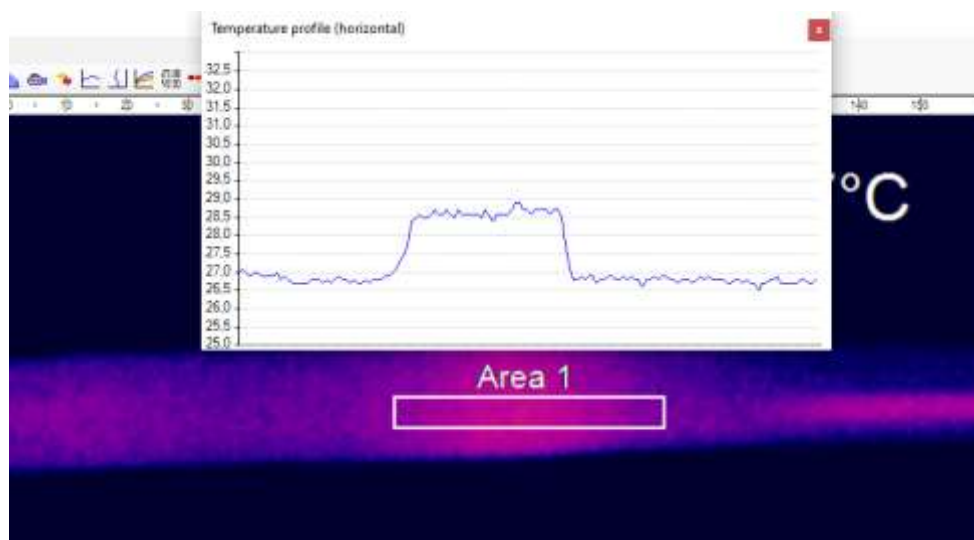


Figure 3. 6. Temperature distribution in the mass of the RA test piece 2.5%.

Synthetic oil additive

In order to obtain a composite material with improved mechanical properties, the matrix formed of epoxy resin Epiphen RE 4020, with hardener DE 4020, was added with synthetic oil with the properties described in subchapter 2.3. The prepolymer mixture was mixed for 10 minutes with the help of the mechanical mixer, after which the synthetic oil was added in a mass proportion of 3%, then the mixing was continued for another 10 minutes. Next, these samples will be graded RU 3%.

The averages of the results of these tests, rounded to the order of hundreds, are presented in table 3.4.

Table 3. 4. Average results of fatigue test for RU 3%.

Angle of inclination (°)	RU 3% (cycles)	σ_{max} (MPa)
8	1250000	35,8
10	325400	47,7
11	113700	57,3
12	78000	59,7
13	48100	63,2
14	21100	65,6
15	12000	69,2
16	6900	74,1
17	4600	77,6
18	3400	78,8
19	2300	87,2
20	1100	90,7

However, the samples inclined at an angle of 8 degrees were stopped from testing, when the number of cycles reached the maximum value resulting from the testing of RTT specimens, respectively 1,250,000 cycles. This option has been chosen in order to reduce test times, as to reach this value, a single test takes about 22 hours.

Figure 3.7 shows the Wöhler curves for RTT and RU 3% materials.

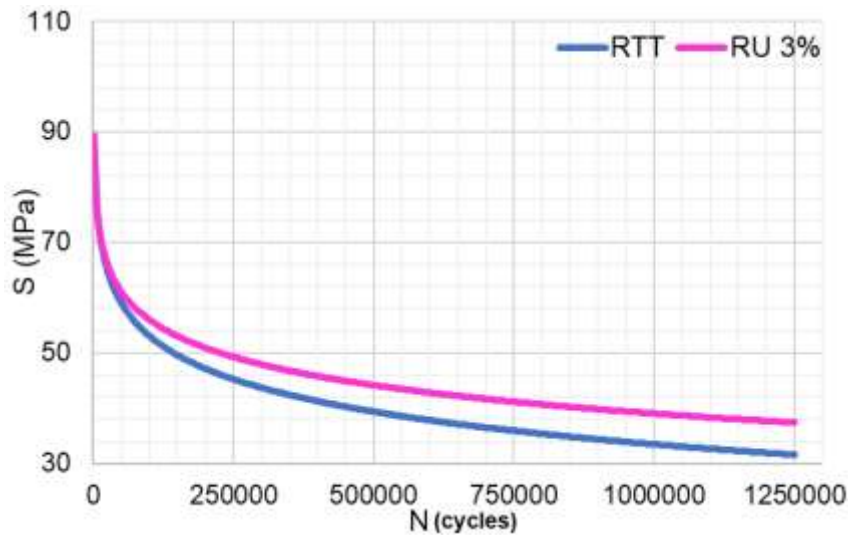


Figure 3. 7. Wöhler curves for RTT and RU 3%.

Figure 3.8 shows the SN curves for RTT, RA 2.5% and RU 3% samples, where it is observed that, from the point of view of fatigue, all samples exceed 106 cycles, but RU 3% can withstand higher voltages at the same number of cycles.

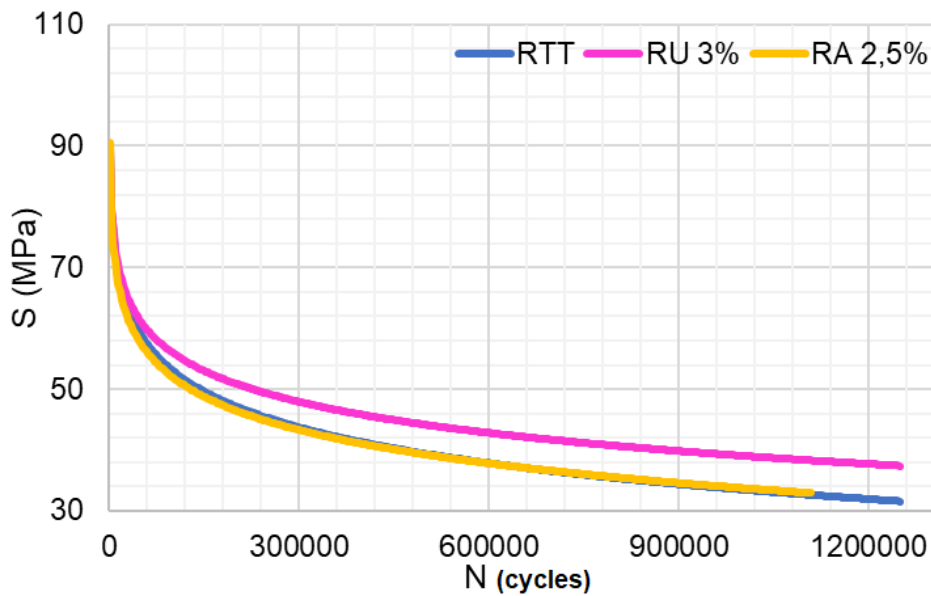


Figure 3. 8. Comparison between RTT, RU 3% and RA 2.5% samples.

3.2. Tests on wear and tear

Analysing the academic literature, it is found that studies have been performed on the influence of normal force on the friction of polymeric materials [83], [84], [73], [85] or how the friction is influenced by the sliding speed [86], [87], [73], as well as the influence of temperature on friction [88], [89], [73], [90]. However, no studies are known to show how friction or wear, respectively, influence the fatigue of polymeric materials or whether the two stresses act simultaneously on a polymeric material and influence its lifespan.

3.2.1. Polymer-polymer couplings

Polymer-polymer couplings are most often used in mechanisms where the speeds and friction forces are relatively low. However, there are few studies in the literature on wear caused by friction between two polymers and little knowledge about their tribological behaviour [91], [92].

Non-additive materials

A series of samples made of Epyphen RE 4020 epoxy resin, with DE 4020 hardener and subjected to three-stage heat treatment (RTT), were tested for wear and tear (caused by friction with sliding in a polymer-polymer coupling).

The pin (counterpart) that comes into contact with the sample by sliding friction, in the polymer-polymer coupling, has a diameter of 6 mm, a length of 25 mm.

During the wear fatigue tests, a pin pressure force was started on the surface of the 10 N sample, but the samples deteriorated due to local heating, above the glass transition limit (T_g).

Consequently, the following tests were performed with a pin pressing force on the sample of 5 N, and in the case of the polymer-polymer coupling they will be thus referred to as RS-5cpp.

The results obtained from the wear and tear tests of RS-5cpp are presented in Table 3.5.

Table 3. 5. Average results of fatigue test for RS-5cpp samples.

Angle of inclination (°)	RS-5cpp (cycles)	σ_{max} (MPa)
8	34300	35,8
10	21100	47,7
11	13700	57,3
12	9100	59,7
13	7100	63,2
14	5800	65,6
15	5000	69,2
16	4400	74,1
17	3800	77,6
18	2800	78,8
19	2100	87,2
20	1500	90,7

Figure 3.9 shows the microscopic image of wear on a counterpart in contact with slip on an 11-degree bent sample.



Figure 3. 9. Wear on the counterpart.

Analyzing the data in table 3.5 or the SN curves in figure 3.10 (where the longitudinal axis was limited to the value of 50,000 cycles to highlight the difference more clearly) it is observed that the lifespan of RS-5cpp samples is much shorter than in the case RTT, both in terms of fatigue and in terms of breaking stress.

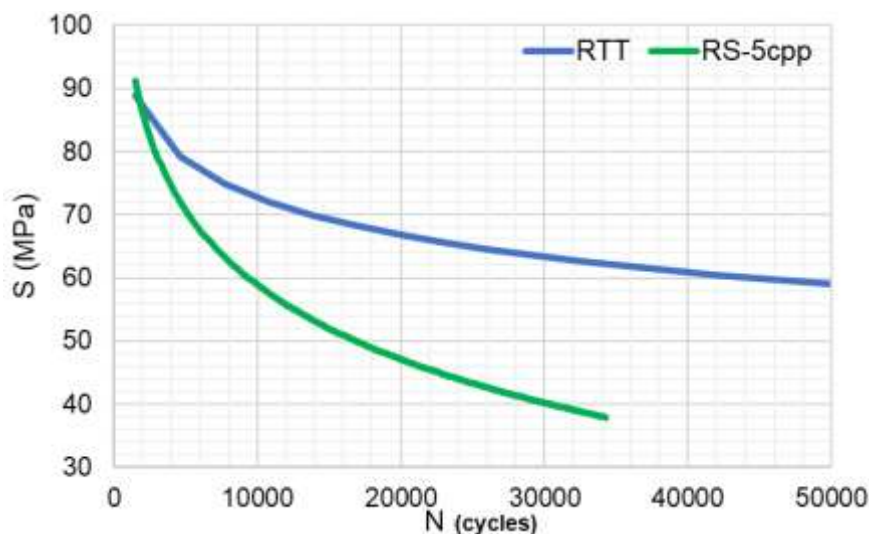


Figure 3. 10. Comparison between the S-N curves of the RTT and RS-5cpp samples.

Additive materials with 2.5% aramid powder

The samples added with 2.5% aramid powder were subjected to wear and tear tests in a polymer-polymer coupling (hereinafter referred to as RA2,5-5 hp), under the same temperature conditions and using the same type of counterpart.

As in the case of the RS-5cpp samples and the RA2.5-5cpp samples, they were subjected to fatigue wear tests caused by slip friction, where the normal force started from 10 N, but following the preliminary tests it was found that they rupture due to reaching the temperature T_g . Consequently, the tests with the normal force of 5 N were continued.

The average of the results obtained from the fatigue wear tests of the RA2,5-5hp samples is shown in Table 3.6.

Table 3. 6. Average of the results of the wear fatigue test for RA2,5-5 hp samples.

Angle of inclination (°)	RA2,5-5cpp (cycles)	σ_{max} (MPa)
8	171400	35,8
10	55200	47,7
11	31100	57,3
12	23300	59,7
13	17200	63,2
14	11400	65,6
15	8200	69,2
16	6500	74,1
17	4400	77,6
18	2700	78,8
19	1600	87,2
20	450	90,7

To observe the differences between the S-N fatigue curves of the RA 2.5% and RA2.5-5hp samples, they were superimposed in a graph visible in Figure 3.11.

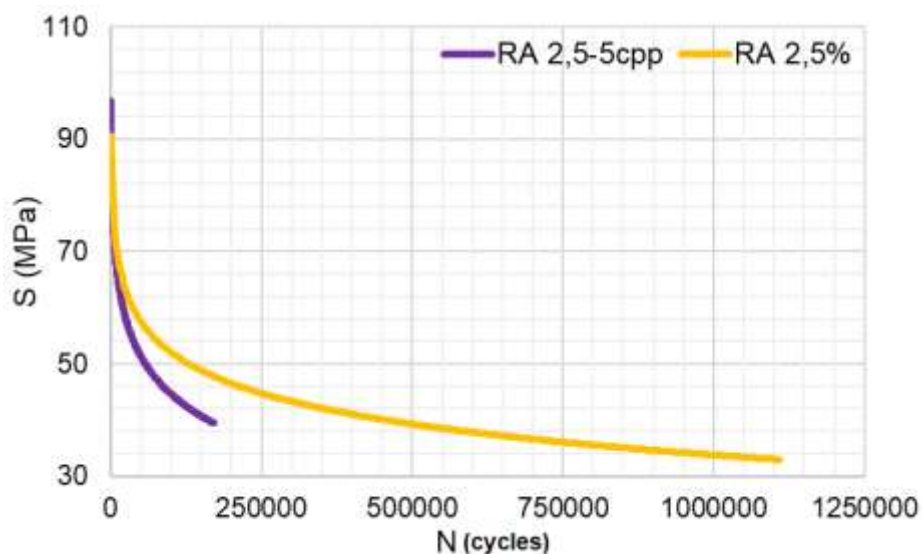


Figure 3. 11. Differences between S-N curves of RA 2.5% and RA2.5-5hp samples.

Materials added with 3% synthetic oil

A samples of RU 3% was tested for wear and tear to determine if and to what extent, tribo-fatigue caused by sliding friction in a polymer-polymer coupling affects the life of that material.

These tests were performed in accordance with the same temperature and pressure conditions on the samples described in the RS-5cpp samples. Epoxy polymeric specimens added with a percentage of 3% synthetic oil used in tests with a normal force of 5 N will be hereinafter referred to as RU3-5cpp.

The average of the results obtained, as a result of the fatigue wear tests of the RU3-5hp samples, are visible in Table 3.7.

Table 3. 7. Average of the results of the wear fatigue test for RU3-5hp samples.

Angle of inclination (°)	RU3-5cpp (cycles)	σ_{max} (MPa)
8	189000	35,8
10	109100	47,7
11	27800	57,3
12	17600	59,7
13	12500	63,2
14	9700	65,6
15	7100	69,2
16	5100	74,1
17	3600	77,6
18	2900	78,8
19	1700	87,2
20	950	90,7

One aspect noted in these tests is that both the counterparts and the samples did not suffer wear and tear with surface damage, as in the case of the counterparts that came into contact with the RS-5cpp or RA2,5-5cpp samples.

Figure 3.12 shows the SN curves of the composite material made of epoxy polymer resin added with 3% synthetic oil tested for fatigue (RU 3%) and tribo-fatigue in a polymer-polymer coupling (RU3-5cpp), as well as RTT samples.

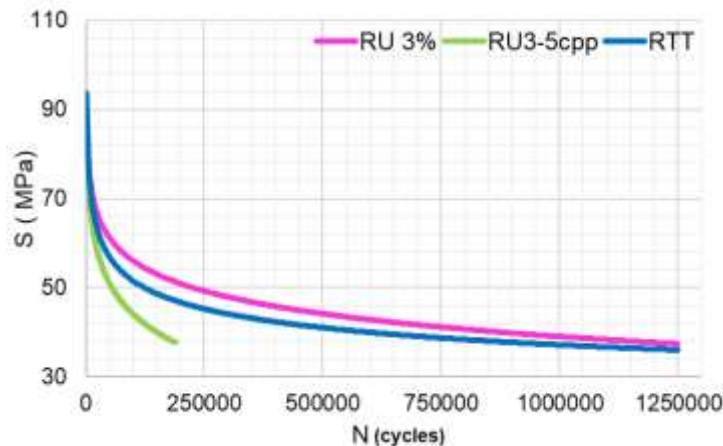


Figure 3. 12. Comparison of S-N curves for RU 3% and RU3-5cpp samples.

Figure 3.13 shows for comparison the S-N curves of the RS-5cpp, RA2.5-5cpp and RU3-5cpp samples.

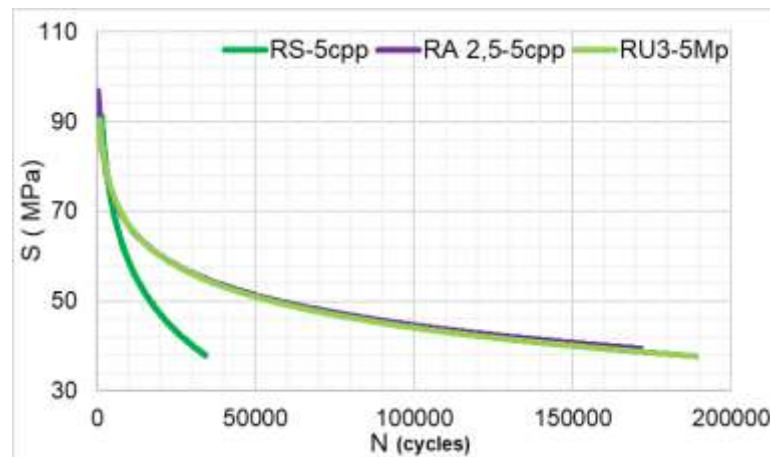


Figure 3. 13. Comparison between the S-N curves of the RS-5cpp, RA2.5-5cpp and RU3-5cpp samples.

Analysing the graph in Figure 3.13, it is observed that, following the tribo-fatigue test, using sliding friction in a polymer-polymer coupling, with the counterpressure force of 5N, the samples made of epoxy resin Epyphen RE 4020 with hardener DE 4020 to which a three-stage heat treatment (RS-5cpp) was applied, have a very short lifespan, compared to the RA2,5-5cpp and RU3-5cpp samples, which have almost identical fatigue curves, those from followed by the longest lifespan.

3.2.2. Polymer-metal couplings

Non-additive materials

A sample of RTT specimens were subjected to fatigue wear tests in a polymer-metal coupling, in which the normal strength of the metal counterpart was 5N, hereinafter referred to as RS-5cpm samples. In these tests, the ambient temperature conditions of 25°C (± 1°C) were observed and five tests were performed for each angle of inclination of the movable chuck, from 8 to 20 degrees.

The average of the results obtained from the tests performed on the RS-5cpm samples are presented in Table 3.8.

Table 3. 8. Average of the results obtained after performing the test on the RS-5cpm samples.

Angle of inclination (°)	RS-5cpm (cycles)	σ_{max} (MPa)
8	59000	35,8
10	21100	47,7
11	18600	57,3
12	17100	59,7
13	15500	63,2
14	12200	65,6
15	9800	69,2
16	6600	74,1
17	4800	77,6
18	2900	78,8
19	2400	87,2
20	1400	90,7

Figure 3.14 shows the SN curves of the material made of epoxy resin EPIPHEN RE 4020 with hardener DE 4020, which was subjected to a heat treatment in three stages and which was subjected to fatigue tests (RTT), then to cumulative stress, tribo-fatigue in a polymer-polymer coupling (RS5-Mp) and at cumulative stresses, tribo-fatigue in a polymer-metal coupling (RS-5cpm).

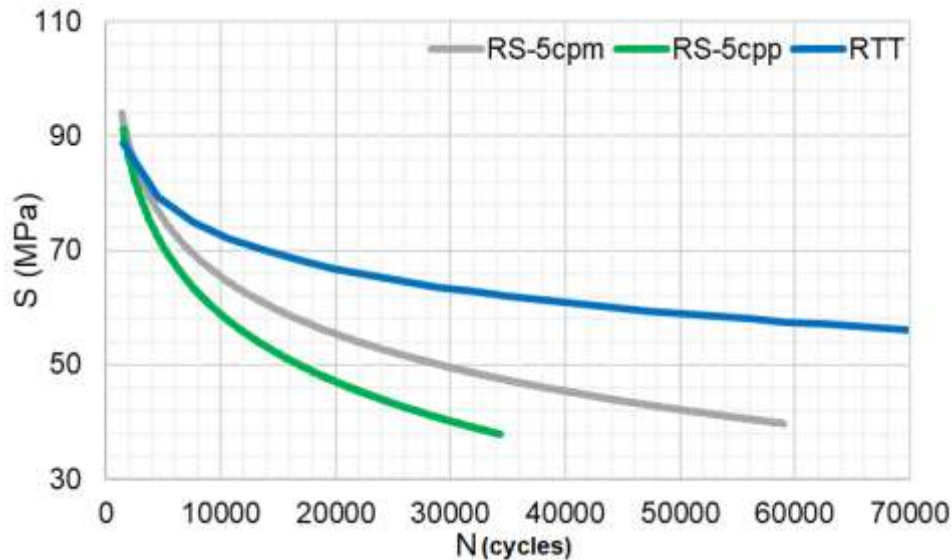


Figure 3. 14. Comparison between RTT, RS-5cpm and RS5-Mp samples

Additive materials with 2.5% aramid powder

A sample of the test tubes made of epoxy resin EPIPHEN RE 4020 with hardener DE 4020, subjected to three-stage heat treatment and added with 2.5% aramid powder, were subjected to wear and tear tests in a polymer-metal coupling, thus referred to as RA2.5-5cpm samples.

The average results of the tribo-fatigue tests applied to the RA2.5-5cpm samples are presented in table 3.9.

Table 3. 9. Average of the results obtained for the RA2.5-5cpm samples, following the tribo-fatigue tests in a polymer-metal coupling.

Angle of inclination (°)	RA2,5-5cpm (cycles)	σ_{max} (MPa)
8	51600	35,8
10	42500	47,7
11	37800	57,3
12	15200	59,7
13	12300	63,2
14	10700	65,6
15	7200	69,2
16	5600	74,1
17	4600	77,6
18	3200	78,8
19	2700	87,2
20	1900	90,7

Figure 3.15 shows the S-N fatigue curve obtained after wear fatigue tests, in a polymer-metal coupling, for RA2.5-5cpm samples, as well as the S-N curve obtained for RTT

samples. For a better representation in order to compare the two curves, the longitudinal axis was limited to 80,000 cycles.

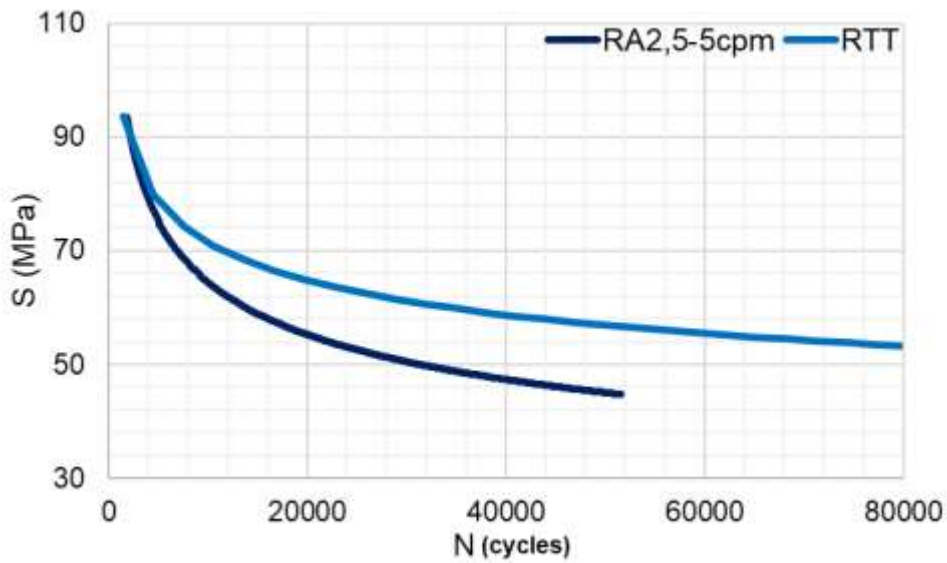


Figure 3. 15. S-N curves for RA2.5-5cpm samples and RTT samples.

To compare the results of fatigue and cumulative tribo-fatigue tests for composite material consisting of EPIPHEN RE 4020 epoxy polymeric resin with hardener DE 4020, added with 2.5% aramid powder subjected to heat treatment In three steps, the three SN curves were superimposed as a graph in Figure 3.16, where the longitudinal axis was limited to 200,000 cycles.

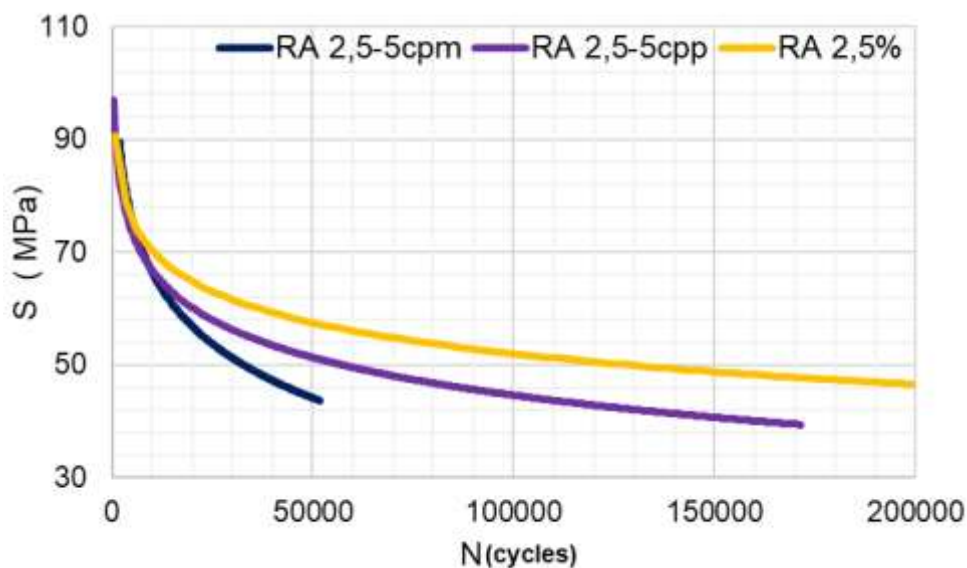


Figure 3. 16. Comparison between S-N curves of RA 2.5%, RA2.5% -5Np and RA2.5-5cpm samples

Analysing the graph in figure 3.16, it can be seen that the composite material RA 2.5%, in the case of a polymer-metal coupling (RA2.5-5cpm), has a very low performance in terms of fatigue combined with wear, the causes being heat accumulation in the contact area and a slight dissipation of it in the whole mass of the material or the retention of the temperature, without yielding it to the metal counterpart.

Materials added with 3% synthetic oil

Part of the samples made of epoxy resin EPIPHEN RE 4020, with hardener DE 4020, subjected to heat treatment in three stages and added with 3% synthetic oil, were subjected to tests of accumulated fatigue with wear, in a polymer-metal coupling (hereinafter referred to as RU3-5cpm samples), to observe how, if or in what percentage, the wear influences the fatigue of the respective material.

The average results obtained from the wear and tear processes performed on the RU3-5cpm samples are presented in Table 3.10.

Table 3. 10. Average of the results obtained for the RU3-5cpm samples, following the tribo-fatigue tests.

Angle of inclination (°)	RU3-5cpm (cycles)	σ_{max} (MPa)
8	71000	35,8
10	54500	47,7
11	34200	57,3
12	23000	59,7
13	16900	63,2
14	14800	65,6
15	11600	69,2
16	9700	74,1
17	6100	77,6
18	4500	78,8
19	3700	87,2
20	2500	90,7

Figure 3.17 shows the S-N curves of the RU3-5cpm samples obtained from the wear fatigue tests, in a polymer-metal coupling, and of the RTT samples. The longitudinal axis was limited to 80,000 cycles, for a better view, in order to compare the two fatigue curves.

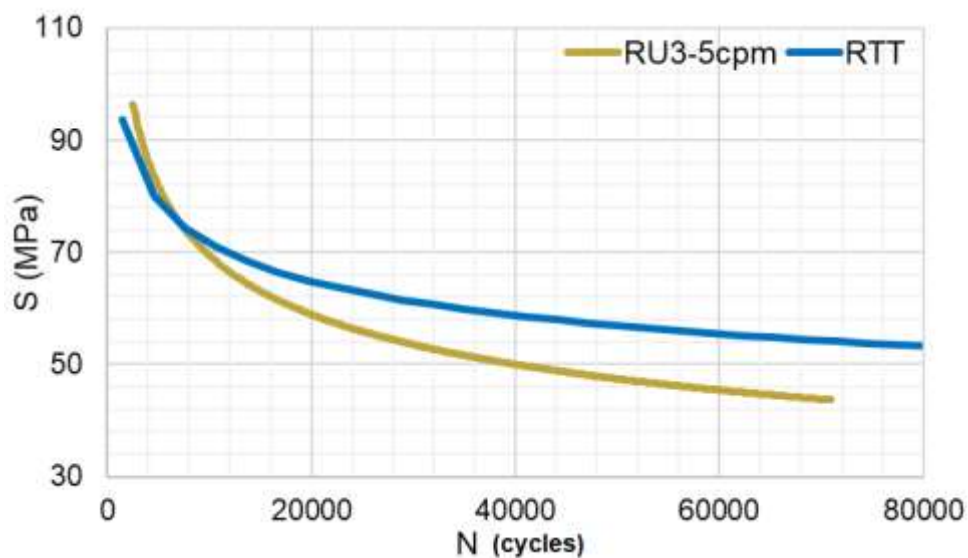


Figure 3. 17. S-N curve for RU3-5cpm samples.

Figure 3.18 is a graph showing the SN fatigue curves of the composite material consisting of Epiphen RE 4020 epoxy polymeric resin, with DE 4020 hardener, heat treated in three stages, added with synthetic oil in a mass proportion of 3% and which was subjected to

fatigue stress (RU 3%), for cumulative tribo-fatigue in a polymer-polymer coupling (RU3-5cpp), as well as for cumulative tribo-fatigue in a polymer-metal coupling (RU3-5cpm). The longitudinal axis was limited to 200,000 cycles, for a better visualization of the results.

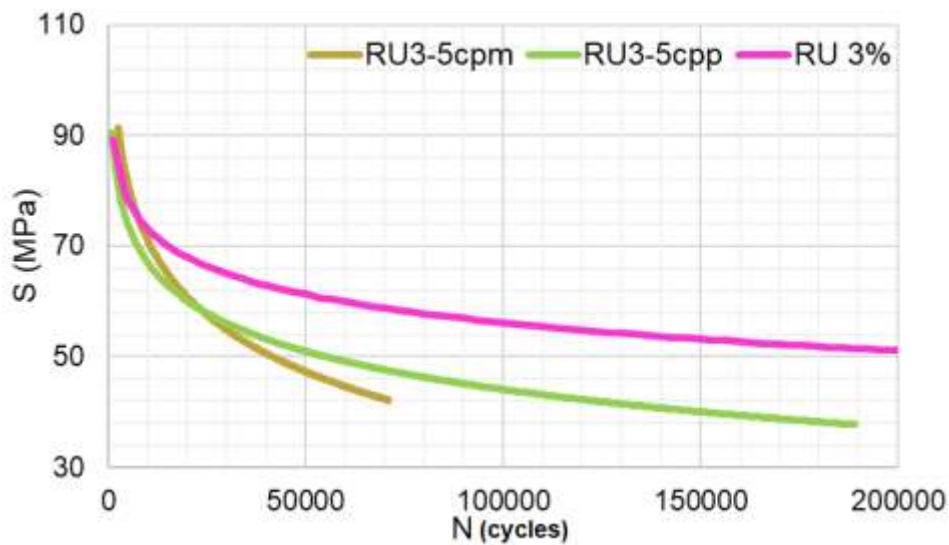


Figure 3. 18. Comparison between S-N fatigue curves of RU 3%, RU3-5cpm and RU3-5cpp samples.

Analysing the graph in figure 3.18, it is observed that the composite material made of epoxy polymeric resin Epiphen RE 4020 with hardener DE 4020, heat treated in three stages and added with synthetic oil in a mass proportion of 3%, has a very low performance on tribo-fatigue, in the case of a polymer-metal friction coupling.

From the point of view of the fatigue resistance combined with the wear caused by sliding friction in a polymer-metal coupling, where the pressing force of the counterpart was 5N, the polymeric material with the best characteristics is the one added with synthetic oil 3%, followed by the one added with aramid powders in proportion of 2.5%, and the last one is the non-added one. These aspects are visible in the graph shown in Figure 3.19.

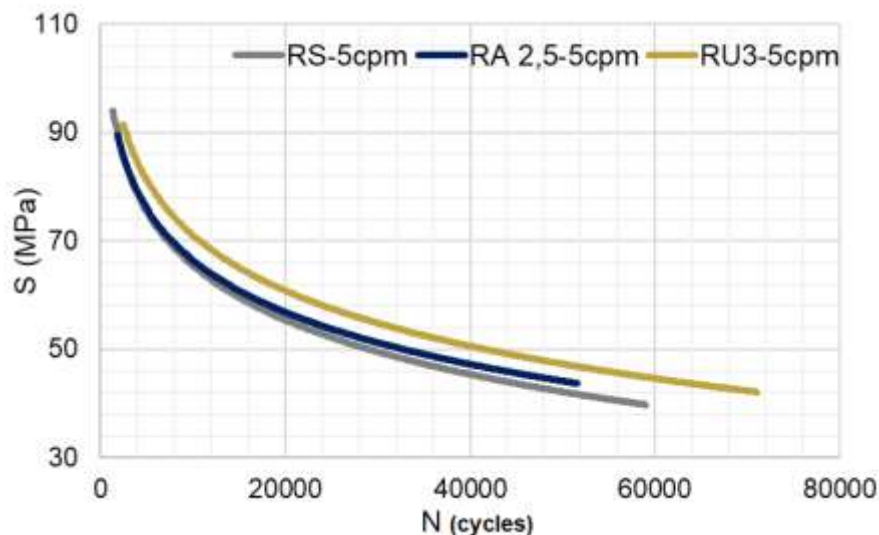


Figure 3. 19. Comparison between RS-5cpm, RA2.5% -cpm and RU3-5cpm.

3.3. Partial conclusions

Following the analysis of the results obtained after performing the fatigue tests cumulated with those of wear, about those mentioned above we can conclude that:

- Samples that have been made of Epyphen RE 4020 epoxy resin, with DE 4020 hardener (RTT), have a very low resistance to cumulative tribo-fatigue.
- Wear occurs faster in the case of RTT samples, which generates the thinning of the area subject to the two stresses, having as a negative effect the increase and accumulation of temperature in that area.
- From a thermal point of view, the 2.5% RA samples are more resistant than the RTT samples, but due to the poor heat dispersion in the whole mass of the samples, it accumulates in the contact area, leading to an overheating of this area, which leads to a decrease in mechanical strength.
- RA2.5% samples have an increased wear resistance, compared to RTT and RU 3%.
- Samples with synthetic oil have a faster heat dissipation, which leads to maintaining a low temperature in the contact area.
- In the case of polymer-polymer couplings, the most suitable materials to use are the additive ones, because they have a much longer lifespan and a tribo-fatigue resistance than the non-additive ones.
- In the case of polymer-metal couplings, materials added with synthetic oil, in proportion of 3%, are the most suitable to use, compared to other materials.
- Analysing the temperature graphs, it is observed that the samples added with synthetic oil, in proportion of 3% (RU 3%), have a very low breaking temperature, followed by those added with aramid (RA2.5%) and the last ones, not added (RTT). Considering that temperature has a very important role in determining the properties of epoxy polymeric materials, when used in friction couples, the most suitable are those added with synthetic.

Chapter 4.

Modelling tribo-fatigue processes using neural networks

4.1. Neural networks

Neural networks are parallel computing systems that work analogously to biological brains: a structure consisting of elementary computing units (artificial neurons), interconnected by synapse-like connections, characterized by specific sizes - weights, structure connected to the outside through input ports, respectively exit. Artificial neurons are computational elements that process the information received with a specific transfer function and pass it on.

The mode of operation of a neural network consists in changing the value of the proportions so that at known input data, we obtain known values at the output (training or learning stage) and the delivery of unknown values for known input data (prediction stage). It should be noted that a neural network provides output data with a certain error whose value is known and accepted by the user. Although, compared to algorithmic computing systems, this error is a disadvantage, it is compensated by the fact that no mathematical relations are required to describe the processed data.

For problems involving prediction or optimization the recommended structure is the type "Feed-forward" [34],[35]. In this representation, figure 4.1, [36], artificial neurons are arranged in several layers, two of which have specific functions: the input layer, the neurons in this layer serving as network inputs and the output layer whose function is to deliver data processed by the network. These two layers are the only ones available for user interaction, the other layers (called hidden layers) cannot be accessed by the user.

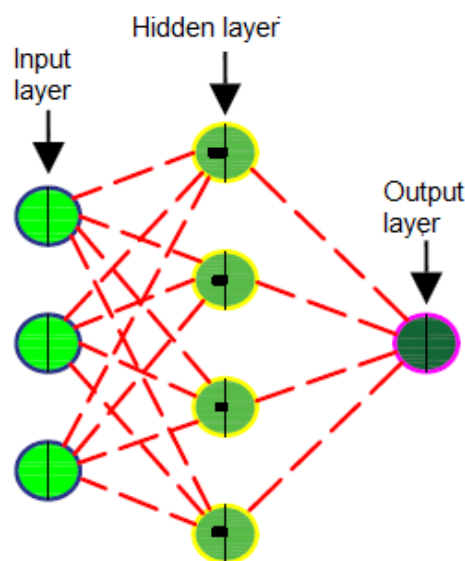


Figure 4. 1 Structured neural network „Feed-forward”

Neural networks are widely used in modelling, simulation and analysis of processes in which polymeric materials are involved [37], [38], [39], [40].

4.2. Basics of modelling with neural networks

Building a model based on neural networks involves using specialized software and going through several work steps: [34],[35]

- Identification of known input-output data pairs
- Choosing and optimizing the network architecture
- Network training
- Network validation

A neural model can be used for several purposes: analysis of importance and sensitivity, prediction and optimization.

The neural model can be used to set input values corresponding to extreme output values: minimum or maximum.

4.3. Modelling tribo-fatigue processes

4.3.1. Identification of known input-output data pairs

The purpose of the presented neural modelling is the analysis of experimental data obtained from tribo-fatigue tests performed on the studied materials. For this purpose, it is necessary to identify the appropriate input-output parameters and to establish the limits of their values, table 4.1.

Table 4. 1 Parameters and limit values

The input parameters	Limit values		Output parameters	Limit values	
	Minimum	Maximum		Minimum	Maximum
Angle	8°	20°	Number of cycles	645	1250000
Loading	0 N	5 N			
Aramidă	0	1			
Ulei	0	1			
Polymer	0	1			
Metal	0	1			

The output parameter was chosen as the number of cycles until the samples broke.

4.3.2. Choosing and optimizing the network architecture

In this stage, the aim is to identify an optimal neural network architecture for the proposed problem. For this purpose, the initial data pairs are introduced in a dedicated software package (Pythia), able to determine the optimal structure of the neural network related to a specific problem using a genetic algorithm.

4.3.3. Network training and validation

In this stage, the known input-output data pairs are presented to the network, in order to configure the internal weights so that the network models the studied phenomenon. The

condition for completing the network entrainment is: the correlation coefficient $R > 0.999$, recommended value in case of modelling polymeric materials [41]. Figure 4.2 shows the graph of the evolution of errors during the learning stage, the imposed condition being met after 611000 cycles.

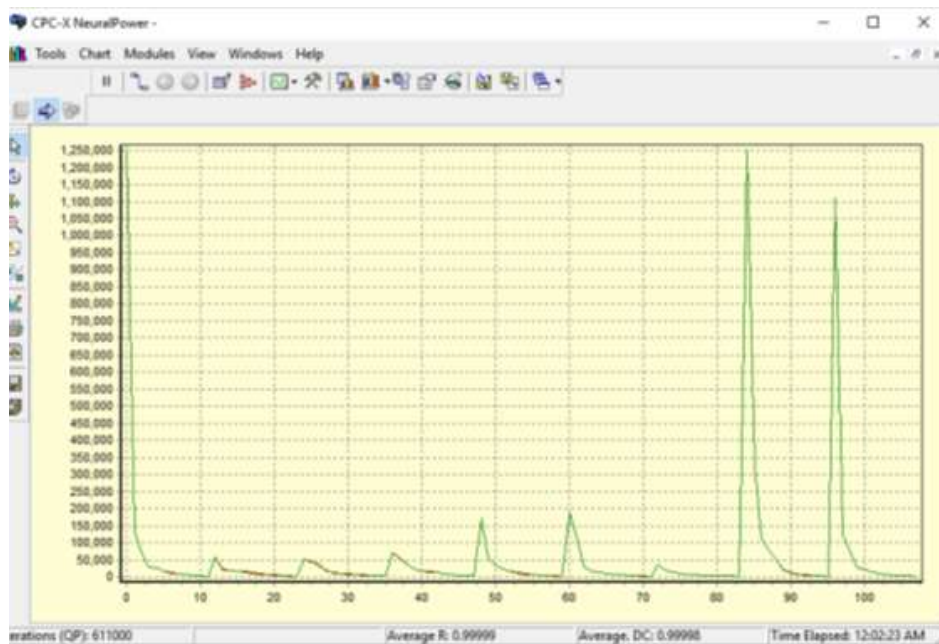


Figure 4. 2 Evolution of the error during the training period

The validation step consists in presenting to the network some data pairs that were not used in training, for each type of test, the step being considered completed when an error of less than 8% is obtained [101]. The result of the validation is presented in table 4.2

Table 4. 2 Validation result

Number of cycles	No. real cycles	No. network cycles	Error [%]
Without wear (pure resin) RTT	1250000	1250332	-0.026
Without wear (synthetic oil additive 3%) RU3%	1250000	1249997	0.00024
Without wear (aramid additive 2.5%) RA2.5%	1100000	1110000	0.9
Whit wear polymer-polymer	34300	34298	0.005
Whit wear polymer-metal	58000	58992	-1.71
With wear (synthetic oil additive, polymer- polymer coupling)	189000	188998	0.001
With wear (synthetic additive oil, polymer metal coupling)	71000	71100	-0.14
With wear (aramid additive, polymer-polymer coupling)	171400	171393	0.004
With wear (aramid additive, polymer-metal coupling)	51600	51490	0.21
Absolute average error			0.332

4.3.4. Analysis of importance and sensitivity

One of the benefits offered by the neural model is the presentation of hierarchies of input parameters, ordered by the degree of influence and sensitivity on the output, figure 4.3.

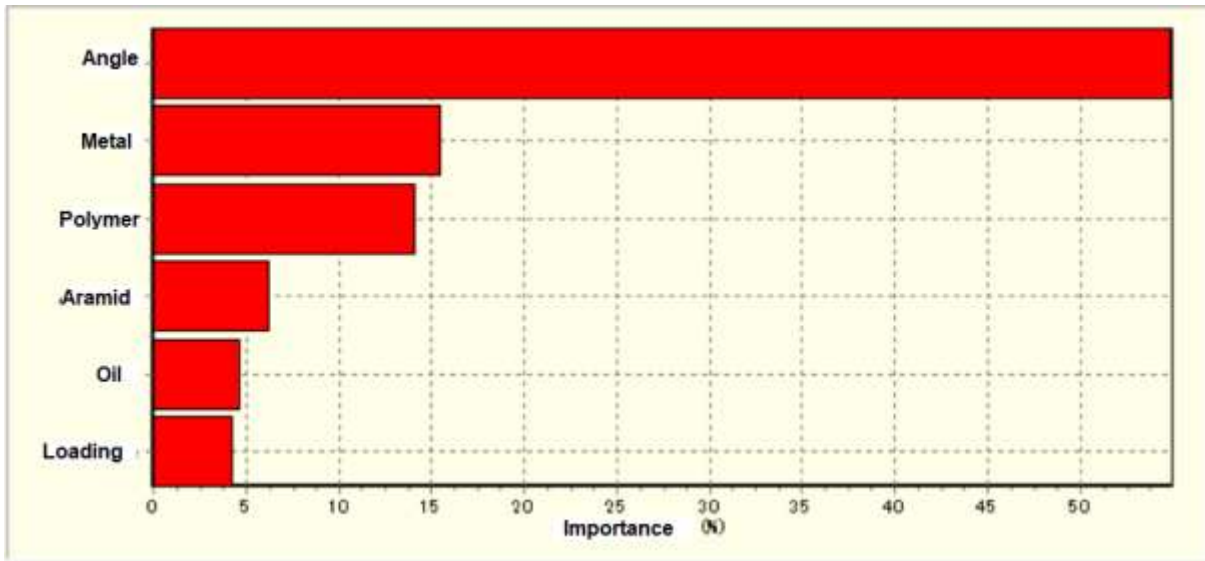


Figure 4. 3. Hierarchies of importance.

It is observed in figure 4.3 that the maximum influence on the tribo-fatigue resistance of the tested materials has the bending angle. From this it can be concluded that the fatigue stress - induced by the bending angle, is the main stress that leads to destruction by this type of compound stress

The influence of the additives used is on the penultimate position, proving that their influence is minimal.

4.3.5. Prediction

The proposed neural model can also be used to predict the number of fatigue cycles that the tested material withstands, provided that the values of the input parameters are combined in other variants than those used to acquire the initial data, the only condition being that these values between the minimum-maximum limits used in the training stage.

The predicted values of the number of fatigue cycles can be used to schedule maintenance operations, so as to eliminate the possibility of unexpected damage.

4.3.6. Optimization

The optimization procedure offered by the model based on neural networks consists in determining the values of the input parameters corresponding to extreme values (minimum or maximum) of the output or to some values imposed by the user.

Table 4.3 shows the maximum values obtained in the optimization stage for fatigue and tribo-fatigue stresses in polymer-polymer and polymer-metal friction couples, respectively, as well as for a desired value of the number of cycles.

Table 4. 3 Optimized values for the number of cycles

Number of cycles	Input values
Fatigue whitout wear	Angle= 8°
Optimized value = 1278676	Loadind = false
Measured value = 1268254	Aramid = 0
Error = -0.821	Oil = 0.9
	Polymer = false

	Metal = false
	Angle = 9.2°
Wear polymer-polymer coupling	Loading = 0
Optimized value = 480697	Aramid = 0
Measured value = 471960	Oil = 1.8
Error = -0.1851	Polymer = true
	Metal = false
	Angle = 8°
Wear polymer-metal coupling	Loading = 0
Optimized value = 390730	Aramid = 0
Measured value = 392105	Oil = 3
Error = 0.350	Polymer = false
	Metal = true
	Angle = 10.4°
Imposed value = 52689	Loading = 3.5
Measured value = 52996	Aramid = 0
Error = 0.579	Oil = 0
	Polymer = true
	Metal = false
Absolute average error optimized values	0.483 %

Following additional tests, with the input values obtained after optimization, an average value of the absolute error of 0.483% was obtained, which validates the applicability of the proposed neural model.

4.4. Partial conclusions

Following the above, a number of conclusions can be highlighted:

- Modelling with the help of neural networks is particularly useful in the case of processes involving polymeric materials, this type of material having a nonlinear behaviour
- In the case of tribo-fatigue stresses, the use of a model based on neural networks is particularly appropriate, offering multiple possibilities such as:
 - analysis of the importance of input parameters on the output: this analysis identifies the parameters with maximum influence (positive or negative) on the number of cycles
 - fatigue resistance prediction - in the form of the number of cycles: allows to determine the value of the number of cycles for a combination of input values different from that obtained by experimental measurements
 - optimization of the output values, in the sense of obtaining maximum or minimum values: this procedure can identify the extreme values of the number of cycles, including the assumption that certain input parameters have prescribed values

Chapter 5.

General conclusions, personal contributions and future research directions

5.1. General conclusions

In this doctoral thesis, a series of tests were performed to determine the lifespan of thermosetting polymeric materials and composite materials, made of epoxy resin, added with various additives. These materials were also subjected to simultaneous tribo-fatigue stresses to determine whether wear has a major influence on the life of the tested materials.

Following these tests, as well as the documentation required in order to carry them out, the following conclusions can be drawn:

- Thermosetting polymeric materials, as well as composites with thermosetting polymeric matrix, have experienced a sharp development lately, as a result they are used in all industrial fields, especially in the wind, naval, automotive, electronics, etc. industries.
- For the production of composites, the most used thermosetting polymeric materials are epoxy resins, due to their special properties, such as adhesion and high compatibility to a diverse range of materials.
- The study of the behaviour of polymeric materials, when subjected to various demands, has been and is the concern of researchers in the field because, knowing these aspects, their properties can be improved (mechanical, tribological, thermal, etc.).
- The specialized literature offers numerous results of researches and experiments performed on thermosetting polymeric materials, for the determination of mechanical properties, these being obtained by tribological tests or bending, stretching and compression. With regard to fatigue and wear of thermosetting polymeric materials, as well as thermosetting polymeric composites, there are far fewer studies investigating these issues and, in any case, they have been carried out separately.
- In this doctoral thesis we studied the influence of wear on the fatigue of thermosetting polymeric materials or, better said, if or in what way the life of thermosetting polymeric materials is influenced (simple or composite, added with various additives or polymerized using various methods), when subjected to simultaneous tribo-fatigue stresses
- The fatigue test stand was greatly improved after the first tests, so that the subsequent results were conclusive and, with their help, the Wöhler Curve for the test materials could be determined.
- The device with which the wear by sliding friction was achieved, attached to the fatigue test stand, made it possible to perform the tests at stresses caused by tribo-fatigue.
- The thermal imaging camera and the camera had a very important role in evaluating and interpreting the results,
- Following the preliminary fatigue tests, a series of parameters could be established such as: the optimal polymerization scheme; the permissible quantities of additives and the time at which they must be introduced into the matrix; the benchmark for subsequent results,

- The standard in this study is represented by the results of the heat-treated samples, to which the three-stage heat treatment scheme was applied. Data from fatigue tests show that:
 - the service life of the samples is influenced both by the heat treatment scheme applied and by the degree of inclination of the movable chuck (the degree of bending of the samples);
 - analysing the data comparatively, it can be seen that the heat-treated samples have a longer service life of more than 92% than those naturally cured, if the movable chuck is inclined at an angle of 8 degrees,
 - their lifespan has a tendency of logarithmic decrease, following the increase of the inclination degree of the mobile chuck. Thus, for the maximum degree of inclination at which the tests were performed (20 degrees), the heat-treated samples withstand a higher number of cycles by about 56%, than those that were naturally polymerized;
 - analysing the averages of the life of the two types of samples compared, it is observed that the heat-treated specimens have a longer service life of approximately 87%,
 - comparing the averages of the results of the samples used as standard and those added with aramid powder in the proportion of 2,5%, it is observed that the latter have a fatigue life of less than about 11%;
 - With regard to the comparison of the average of the results between the standard and the samples with 3% synthetic oil, it is observed that the samples proved to be more resistant than the standard by approximately 19%,
 - therefore, in the case of fatigue tests, the best results were obtained with 3% synthetic oil additive samples;
- Comparing the averages of the standard results with those of the samples that were subjected to cumulative tribo-fatigue stresses within the polymer-metal friction couples, it is observed that:
 - the standard has a lifespan 89% longer than simple samples, which were heat treated in three stages,
 - compared to samples added with 2.5% aramid powder, ethanol resists to fatigue, more by 87%,
 - in the case of samples with 3% synthetic oil, the average of the results shows that the standard is more resistant by 83%,
 - In conclusion, in the case of cumulative tribo-fatigue stresses in polymer-metal friction couplings, the results show that the polymeric material added with 3% synthetic oil has higher characteristics than those added with 2.5% aramid powders, and compared to the simple ones,
- In the case of samples which have been subjected to cumulative tribo-fatigue stresses within the polymer-polymer friction couples, the averages of the data obtained and compared with the standard show that:
 - simple samples, which have been heat-treated in three stages, have a lower fatigue resistance of about 93% compared to the standard;
 - test samples with 2.5% aramid powder added to the standard are approximately 79% less resistant to fatigue;
 - test samples with 3% synthetic oil have a lower fatigue resistance than the standard, by about 75%;
 - In conclusion, in the case of cumulative tribo-fatigue stresses in polymer-polymer friction couples, the results show that of the three materials tested, as in the case of a polymer-metal friction couple, the best performance is given to the additive material with 3% oil
- The use of programs with neural networks is very useful for the prediction and optimization of some parameters of polymeric materials, because they have a nonlinear behaviour.
- In these studies, the Neural Power program validated the data obtained experimentally, confirming that the greatest importance, both in case of fatigue and tribo-

fatigue, is the bending angle of the test piece and the scheme of heat treatment applied, and the less important being the additives used (in this case).

- The experimental results showed that the polymeric material added with synthetic oil in a mass proportion of 3%, has improved characteristics both for fatigue and cumulative stress-fatigue. Data were validated through artificial intelligence, namely neural networks (computer application Neural Power).
- An optimization was performed with the help of neural networks, on the polymeric materials studied and introduced for analysis. Based on the results obtained, stand tests were performed, and the experimental results confirmed that the prediction was good, because the absolute average error of the optimized values was 0.483%.

5.2. Personal contributions

Personal contributions in determining the synergistic effect of wear and fatigue processes on the degradation of polymeric materials are:

- Documentation in order to establish the level of innovation in the field of thermoset polymeric materials, as well as the studies performed on the fatigue, wear and tribo-fatigue of these materials;
- Obtaining composite materials, having epoxy matrix added with aramid powders or synthetic oil;
- Determining the optimal way to mix epoxy resin with additives;
- Analysis of the dispersion of additives in the epoxy resin;
- Design of a protocol for testing thermosetting polymeric materials for fatigue and tribo-fatigue;
- Construction and improvement of a stand that allows the performance of fatigue tests;
- Development of a friction device that can be attached to the stand and with the help of which tribo-fatigue tests can be performed;
- Designing a methodology for monitoring the temperature of the specimens;
- Designing a methodology for capturing images, in order to follow the evolution of wear on the tested materials;
- Microscopic analysis of ruptures resulting from tests;
- Determining the type of destruction of the tested materials, by observing the characteristics of the destruction caused by their submission to variable stresses or caused by reaching the temperature T_g ;
- Obtaining experimental data, with the help of which it was possible to determine the fatigue curves of the studied materials;
- Use of neural networks to build and validate models that can simulate and optimize the amounts of additives or how they are mixed;
- Carrying out simulations, with the data provided by neural networks, in order to create new materials, as well as determining their lifespan;
- Determination of the influence of wear on the fatigue strength of thermosetting polymeric materials.
- The influence of various additives on wear reduction.

5.3. Future directions of research

Based on the documentation, the experiments performed, as well as the personal experiences accumulated, for future research I propose:

- Investigation of the behaviour of thermosetting polymeric materials or composites based on epoxy resins, when they are subjected to cumulative tribo-fatigue stress, at negative temperatures.
- Development of polymeric materials using various additives, which facilitate the dispersion or release of heat from the highly stressed area, as well as the investigation of their wear and tribo-fatigue behaviour
- Investigating the behaviour of thermosetting polymeric materials, which will be subjected to tribo-fatigue stress until the first cracks appear, reconditioning them by specific methods and continuing testing.
- Implementation of a device, on the current stand, to allow the performance of cumulative tribo-fatigue tests caused by rolling friction
- Implementation of a device that can be attached to the stand, with the help of which cumulative fatigue tests with abrasive friction or lubrication can be performed
- Realization of composite materials based on prediction and simulations performed using neural network software, using input data obtained from research conducted for the development of this thesis.

List of published works

Publications in WOS indexed journals

1. **C. Humelnicu**, S. Ciortan, V. Amortila, Artificial Neural Network-Based Analysis of the Tribological Behavior of Vegetable Oil-Diesel Fuel Mixtures, *Lubricants*. 7 (2019) 32.
2. **C. Humelnicu**, V. Amortila, E. Mereuta, Fatigue life investigation on a MAC engine piston, *MATEC Web of Conferences*, EDP Sciences, 2018, 178, 06013.
3. V. Amortila, E. Mereuta, S. Veresiu, M. Rus, **C. Humelnicu**, Positioning study of driver's hands in certain areas of the steering wheel, *MATEC Web of Conferences*, EDP Sciences, 2018, 178, 06014.

Publications in SCOPUS indexed journals

1. **C. Humelnicu**, E. Mereuta, V. Amortila, M. Gingarasu, M. Novetschi, Reduction of polymeric waste by applying heat treatments during the polymerization period, *International Multidisciplinary Scientific GeoConference Surveying Geology and Mining Ecology Management, SGEM*, 2020, 2020-August(4.1), pp. 475–482.
2. M. Gingarasu, E. Mereuta, V. Amortila, **C. Humelnicu**, M. Novetschi, The importance of vehicle steering system diagnosis in reducing environmental impact, *International Multidisciplinary Scientific GeoConference Surveying Geology and Mining Ecology Management, SGEM*, 2020, 2020-August(4.1), pp. 523–530.
3. M. Novetschi, E. Mereuta, M. Gingarasu, **C. Humelnicu**, V. Amortila, The effects of auto pollution on the population, *International Multidisciplinary Scientific GeoConference Surveying Geology and Mining Ecology Management, SGEM*, 2020, 2020-August(4.1), pp. 499–506
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6. M. Gingarasu, E. Mereuta, V. Amortila, **C. Humelnicu**, The influence of the vehicle steering system missalignment on the environment, *International Multidisciplinary Scientific GeoConference Surveying Geology and Mining Ecology Management, SGEM*, 2019, 19(4.2), pp. 295–302
7. V. Amortila, E. Mereuta, **C. Humelnicu**, M. Gingarasu, The vibration and noise pollution's impact on the driver, *International Multidisciplinary Scientific GeoConference Surveying Geology and Mining Ecology Management, SGEM*, 2019, 19(4.1), pp. 1143–1150.
8. M. Gingarasu, E. Mereuta, V. Amortila, **C. Humelnicu**, The influence of vehicle diagnosis on reducing gas emissions that affects the earth's climate system, *International Multidisciplinary Scientific GeoConference Surveying Geology and Mining Ecology Management, SGEM*, 2019, 19(4.1), pp. 1109–1115.
9. V. Amortila, E. Mereuta, **C. Humelnicu**, M. Gingarasu, N. Monica, Controversy about car pollution: The electric vehicle or the classic vehicle?, *International Multidisciplinary Scientific GeoConference Surveying Geology and Mining Ecology Management, SGEM*, 2019, 19(4.2), pp. 193–200.
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2. Mihai Gingărașu, Elena Mereuță, Valentin Amortilă, **Costel Humelnicu**, Monica Novetschi, „THE WEAR OF THE SPHERICAL JOINTS OF THE VEHICLES STEERING SYSTEMS - VIBRATIONS AND THEIR ROLE IN DIAGNOSIS”, „8th Edition Scientific Conference of Doctoral Schools SCDS-UDJG 2020”,
3. Monica Novetschi, Tarek Nazer, **Costel Humelnicu**, Mihai Gingarasu, Valentin Amortila, „LOWER LIMB ANALYSIS WHEN THE CLUTCH PEDAL IS ACTUATED”, „8th Edition Scientific Conference of Doctoral Schools SCDS-UDJG 2020”,
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Award-winning papers at international conferences

1. Mihai Gingărașu, Elena Mereuță, Valentin Amortilă, **Costel Humelnicu**, Monica Novetschi, The wear of the spherical joints of the vehicles steering systems - Vibrations and their role in diagnosis, 8th Edition Scientific Conference of Doctoral Schools SCDS-UDJG, 2020 – FIRST PRIZE,
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Other awards obtained

Premiul 2 la concursul de „Planuri de afaceri” din cadrul programului antreprenorial desfășurat în cadrul Activității 5, participant ca membru al grupului țintă al proiectului „BeAntreprenor!” din partea partenerului P2 (Universitatea „Dunărea de Jos” din Galați)

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