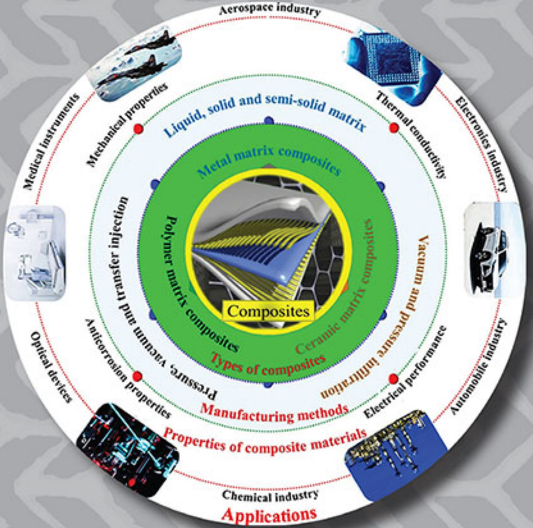
Ceramics and Composites

Preparation and Applications



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Introduction

Currently, the characteristics of ceramic materials are not enough to meet the current industrial, economic, and environmental needs. Therefore, the high demand for materials with tailored properties, which combine the advantages of many basic components, has led to the sustained growth of composite materials. The engine for the increased interest in such materials is mainly the achievement of superior characteristics to conventional materials. However, the obtaining technologies are often constrained by the complexity of the processes and the high costs associated with them.

The book contains a comprehensive and interdisciplinary study in the field of ceramic and composites materials. Therefore, the advantages and disadvantages of their obtaining methods will be presented, while conventional applications, up to cutting-edge research and developments in this fields will be approached (Figure 1.1).

In industrial applications, the materials used for the manufacturing of different components, represent essential phases that can influence most of the production factors (cycle time, product quality, energy consumption etc.) which will also, affect the final cost of the product. Therefore, in order to increase industrial efficiency and reduce manufacturing costs, since ancient times, the improvement of these materials has contributed to an efficient manufacturing process (assuring certain operation speeds, under controlled environmental conditions and higher durability). Accordingly, depending on the operating requirements, over time, improved ceramics and composites have been developed with specific shapes, complexity and overall characteristics.

The book is relevant for fundamental and applied research in the field of materials science and engineering since the base concept and advanced knowledge in these fields will be presented. Therefore, the book entitled “Ceramics and Composites: Obtaining and Applications” is a milestone, a practical and theoretical guide for both specialists in the field and for young researchers who want to deepen their understanding of obtaining and using ceramic materials, respectively composites.

A composite material is a combination of two or more materials with different chemical characteristics and a distinct interface between them. Accordingly, the constituent materials will retain their identity (at least at the microscopic level) in the composite, but through their combination, a material is formed with properties and characteristics superior to those of each constituent [1,2]. One of these constituents will represent the highest content and it forms a continuous phase that is known as matrix. The other important constituent is a reinforcement in the form of fibers or particles, which is generally added to the matrix to improve or modify its properties. The reinforcing element forms a discontinuous phase which is evenly dispersed throughout the matrix. Depending on the adhesion between the components, the surface of the reinforcing element can be chemically treated or coated with a very thin layer to improve the bond between the reinforcing elements and the matrix, as well as to protect its surface against chemical attack, moisture, or adverse chemical reactions that may occur between the matrix and reinforcement element at high temperatures.

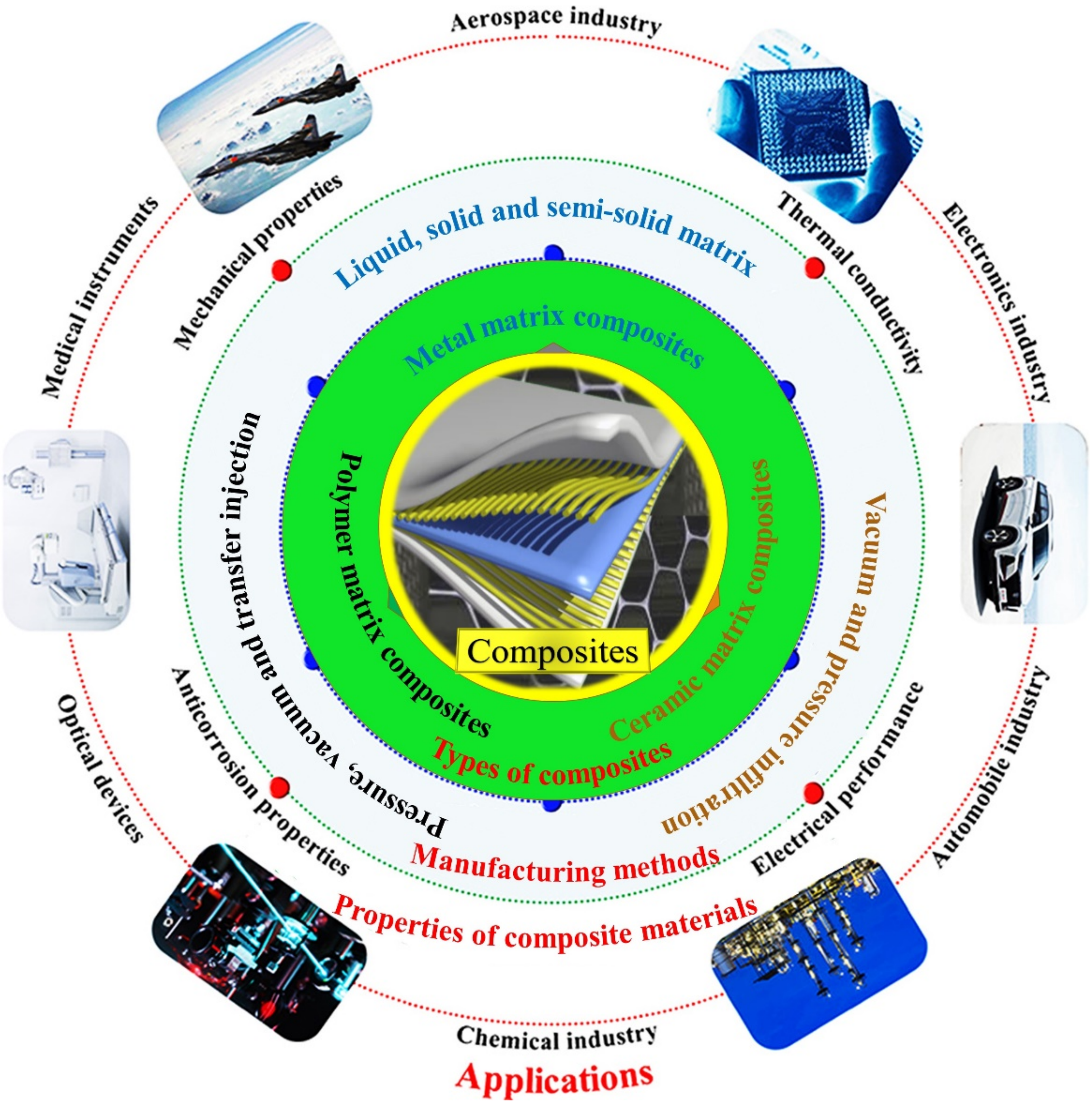


Figure 1. Graphical abstract of the book (adapted from [3]).

A composite material is made up of the distribution of a substance called reinforcement in another substance called a matrix. The reinforcement is the strong and rigid phase, while the matrix is ductile. The properties of the reinforcement and the matrix are complementary through a specific mechanism for transferring the maximum load from the matrix to the reinforcement.

The design and manufacture of composite materials were necessary because natural materials with all their advantageous properties have an essential defect: fragility. If the matrix is brittle (e.g., borosilicate glass), the reinforcement with silicon carbide fibers produces an intense increase in the elongation capacity until rupture, as a result of stopping the growth of cracks in the ceramic material.

The mechanical strength and rigidity of a composite material depend primarily on the nature of the reinforcing material, while the electrical and thermal conductivity is determined by the conductivity of the matrix [4].

The diversity of the composite materials makes a complete classification difficult. However, the accepted classification criteria take into account the following aspects: the nature of the matrix material, the type of reinforcement, the nature of the reinforcement material; the type of matrix-reinforcement joint; reinforcement geometry (configuration); the technological process used, the place of use or application of the composite material. This structure of composite material is characteristic of many natural products, which explains their special mechanical strength. It is enough to mention wood, bamboo, bones, muscles etc. [5–7].

It is also known that metal alloys have superior properties to those of the constituent components. The same can be sustained about ceramic or composite materials.

Taking into account these aspects, the first classification of composite materials can be made according to their origin [8]. Thus, they can be:

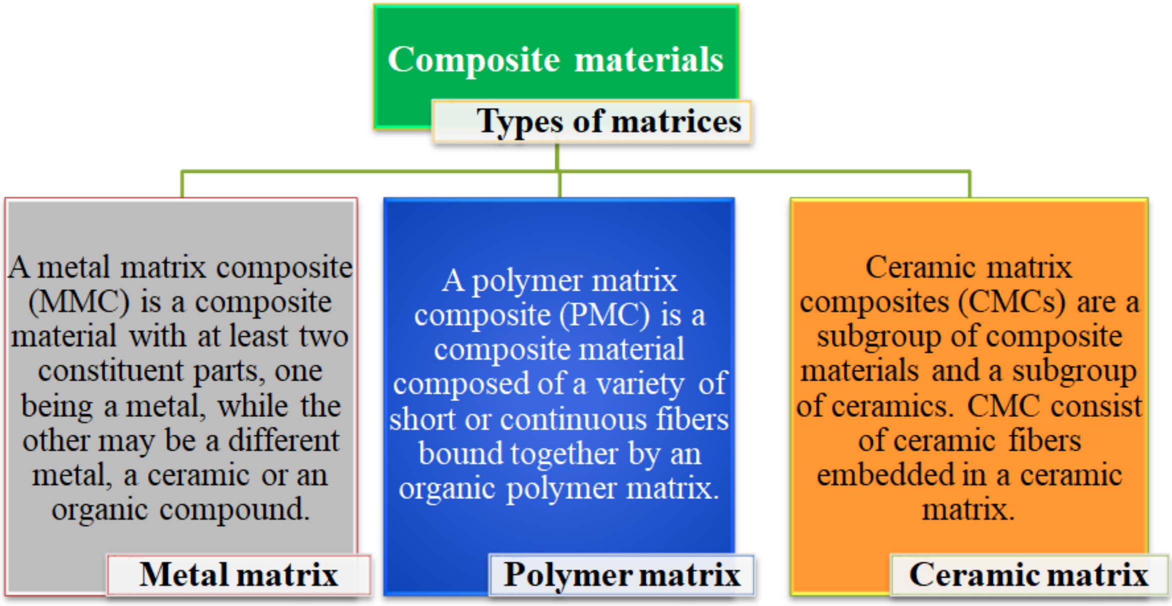
- Natural composite materials, such as wood, bones, bamboo, moss etc.
- Synthetic composite materials, such as metallic, ceramic and polymeric.

According to the nature of the matrix, composite materials are classified as in Figure 2.

When choosing the material for the matrix, the criterion of good plasticity is taken into account, but depending on the use of the composite material, the working temperature is also taken into account.

For working temperatures below 200 °C, polymers such as thermoplastics and thermosets are used, polyimides are used below 300 °C, ceramics are used below 1700 °C and amorphous carbon is used below 2500 °C. In the case of metals and alloys, the temperature depends on the type of material [9,10].

Generally, in the case of composite materials, the matrix can be metallic, polymeric, or ceramic. Depending on the matrix used, composite materials can be metal matrix composites (MMC), polymer matrix composites (PMC), or ceramic matrix composites (CMC).



A metal matrix composite (MMC) is a composite material with at least two constituent parts, one being a metal, while the other may be a different metal, a ceramic or an organic compound.

A polymer matrix composite (PMC) is a composite material composed of a variety of short or continuous fibers bound together by an organic polymer matrix.

Ceramic matrix composites (CMCs) are a subgroup of composite materials and a subgroup of ceramics. CMC consist of ceramic fibers embedded in a ceramic matrix.

Figure 2. Composites materials classification depending on the type of matrices [11].

The MMC materials consist of long or short (whiskers) fibers or particles dispersed in a metallic matrix. Considering this, the natural mixes, the alloys, can't be classified as composites, since the constituents can be classified as fibers or particles. The alloys can be considered a mix of matrices without reinforcing elements. For example, in the case of cast iron, one phase is the ferrite and the other one can be the graphite. Yet, in the case of MMC, the matrix can be an alloy, while the reinforcing element can be a different alloy or a different material (ceramic) [12]. When an MMC is designed, the matrix is chosen depending on its mechanical, tribological and corrosion properties, because, one of its main objectives is to embed and protect the reinforcing elements against the environmental factors that can affect them (oxidizing atmospheres, different corrosive products etc.). Commonly, Al, Ni, Cu, Ti, Mg, Zn, Sn, Fe, or Ag-based alloys are used as matrices, Al, Ti, and Mg being the most used. Al matrices are mainly used due to their low density and high elasticity/plasticity. Ti matrices are used due to their refractoriness, high mechanical properties and good corrosion resistance. Mg are usually used for MMC through the liquid phase manufacturing method, due to its lightweight and biocompatibility [13,14].

The PMC are generally used due to their low obtaining temperature. Basically, they can be reinforced to improve their mechanical properties or modified to obtain smart materials (additives can be introduced in the matrix to change the behavior of the polymer when exposed to specific factors, such as light, temperature etc.). The thermorigide PMC consists of a polymeric matrix with rigid 3D molecular structure reinforced with different elements. Also, the second class of PMC materials are those based on thermoplastic resins which possess lower dimensional stability, but due to the fact that they can be easily machined, they are widely spread in industrial applications.

Given the matrix-reinforcement torque, the composite materials can be in the metal-metal, metal-ceramic, metal-polymer, ceramic-polymer, ceramic-ceramic or polymer-polymer system. Accordingly, concrete and reinforced concrete can be considered composite materials formed of cement-gravel or cement-gravel-iron; also, plywood with alternative layers of wood veneer can be considered a composite material in layers.

The CMC composites are mostly used in high-temperature applications. Technical ceramic is, usually, used as a matrix material, due to its high mechanical properties, low density (2 to 3 times lower than steel) and high hardness (which can increase with the increase of temperature).

Considering the components of composite materials, it can be stated that, these materials include a discontinuous phase (the reinforcing elements) a continuous phase (the matrix) and different types of additives [11]. Accordingly, their classification depending on the type of addition is as follows:

- Composites reinforced with particles;
- Composites reinforced with scales;
- Composites reinforced with fibers.

Further, they can be customized depending on the available raw materials or intended applications. Therefore, the particles or scales can have different sizes or shapes, while the reinforcing fibers can have different lengths, thicknesses and, most important, orientation (Figure 3).

These characteristics are of high importance, since, the final properties of the material will strongly depend of it. Accordingly, the orientation and distribution of the reinforcing elements will result in a homogeneous or heterogeneous material, related to their properties. In other words, the obtained material can exhibit higher properties in one loading direction than another or at a different point. Therefore, if the product must exhibit high tensile strength in one direction, there is no point to introduce woven fibers that will increase the tensile strength in all directions (depending on the woven type). However, if the material must exhibit good tensile strength in all directions, a special woven must be chosen.

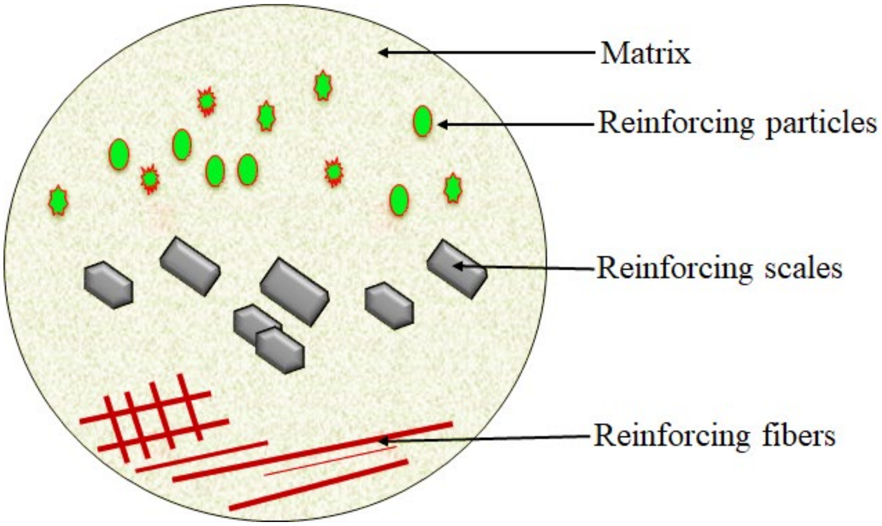


Figure 3. The main components of composite material [15].

Moreover, if one type of reinforcing element doesn't fulfill the requirements. A mix of particles and fibers can be used, while if only one matrix isn't enough, the composites can include a mix of matrices. This class is known as hybrid composites.

From the point of view of the reinforcement configuration or the reinforcement geometry, the composite materials may comprise the following configurations according to Figure 4 reinforcement from particles or short fibers distributed in the mass of the matrix (Figure 4a and b), layered reinforcement resulting from the superposition of several layers of parallel fibers (Figure 4c); multilayer reinforcement with biaxially or triaxially woven fibers in each layer resulting in a layered composite in the end (Figure 4d); three-dimensional reinforcement made by weaving or crossing fibers (Figure 4e and f).

The composite materials manufacturing techniques are vast, especially, because of the composite's versatility and a high number of components. The criterion on which the manufacturing is selected are related to the production rate (unique, series production, mass production etc.), manufacturing costs, quality standard, required characteristics, intended application (for example, the quality criterion for a composite used in the manufacture of laptop case are different than those used for the manufacture of the hood of a car or even different safety components) etc.

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Manual layout, conventional, pressure, vacuum or autoclave casting, sputtering, vacuum injection molding, or vacuum-assisted resin transfer molding are only a few of the manufacturing techniques used to obtain products based on composite materials.

During the beginning of the composites age, an ingenious combination between plastic and fiberglass as reinforcing elements was tested. This combination was considered, since plastic has excellent flexibility and versatility, while fiberglass is durable. At the same time, both have specific disadvantages, fiberglass is fragile, while plastics exhibit low mechanical strength. Therefore, by bringing these two together it is expected to obtain a material that combines the advantages of each component. Yet, the result was outstanding, since the new material also possesses new properties. Gradually, materials science engineers have realized that by combining different components, the resulting material will exhibit better properties than the sum of those of each component, when tested separately. Such an evolution may be possible due to the synergy between the reinforcing fibers and the matrix. Therefore, their combination reveals new possibilities and generates innovations.

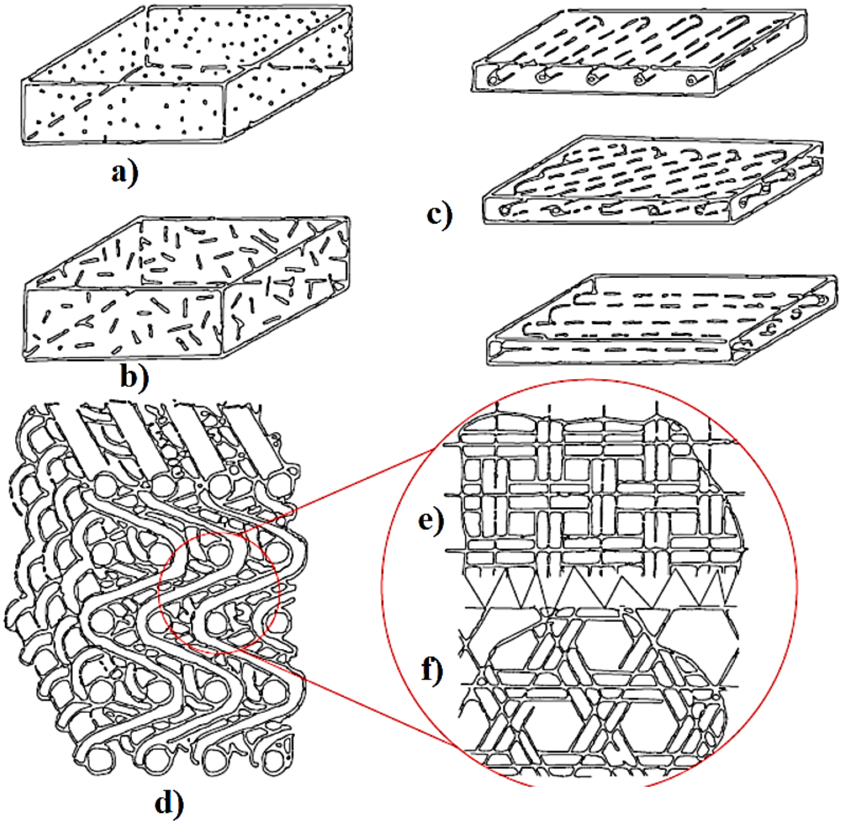

Figure 4. Distribution of reinforcing elements in the matrices: a) particles; b) short fibers; c) long fibers; d) 3D geometry of woven fibers; e) and f) 2D geometry of woven fibers [16].

Another, famous example of successful replacement of conventional materials with composites is that of changing the chrome-steel bumpers with composite bumpers, from ‘50 cars. Besides, their high corrosion resistance and science-related advantages, the reason for changing the conventional material with a composite was the low weight of plastics, as well as the advantages related to their mechanical properties, when they were adequately reinforced. Similar replacements were made to several elements from cars. In the case of bumpers, the replacement of the material acted as a driving force that generates a complex dynamic process of change. Accordingly, the introduction of plastics as a substitute for chrome steel did not immediately involve a reduction in costs as expected, as this change involved major financial investments for research and development, testing, trials and new equipment. Finally, innovation costs have been amortized, in large part, because plastics have opened up new avenues for change. Unlike metals, both reinforced or nonreinforced plastic composites can be cast and shaped during the hardening process of the resin. Since, in the conventional manufacturing method of different metal parts from a car, metal fabrication and part machining/modeling were two successive/different operations, in the case of this type of composites, both operations can be done in the same stage. The material can be cast and molded in the form of the final part through a single-stage process.

Considering these advantages, the car designers were able to redesign the bumpers according to the current style of the cars. Moreover, by reducing the number of operations, the manufacturing process becomes much more time-efficient. Also, the bumpers could be now curved and shaped along the housing line, which contributes to the integration of this protective element in the body of the car. Accordingly, instead of a separate part that is made independently and then welded on the machine, the composite shield becomes now a second protective layer wrapped around the car body.

In terms of engineering development, such synergistic effects were achieved only due to the strong interdisciplinary cooperation between chemists, physicists, designers and materials science engineers. Therefore, the innovation principle "establish a set of functions, find the properties that fulfill the functions, design a material that combines those properties" was the one that boost the development of composites. However, another important aspect that contributed to their evolution, was the advantages offered by the introduction of computes in the control of their manufacturing technologies, especially, because the computes simulation software’s offered enormous support in fitting possible components to develop tailored properties.

Therefore, such examples of synergy have led to a new paradigm for composite technologies. Since 1970, composite materials have been defined by the association of the matrix with reinforced fibers, in 1980, the synergy effect becomes part of the standard definition. For example, Philippe Cognard, author of the French textbook for materials engineers in training, wrote: "A composite material is a material whose set of constituent elements generates a synergistic effect within the properties of these elements. This two- or three-dimensional assembly consists of two or more basic elements, which can have all kinds of shapes: matrices, fibers, particles, plates, sheets etc., which allow obtaining an elastic material, whose elements are strong and durable attached. However, few composites meet this ideal definition. Such synergistic effects are neither common nor predictable [17]. Figure 5 shows the importance of composite materials over the years.

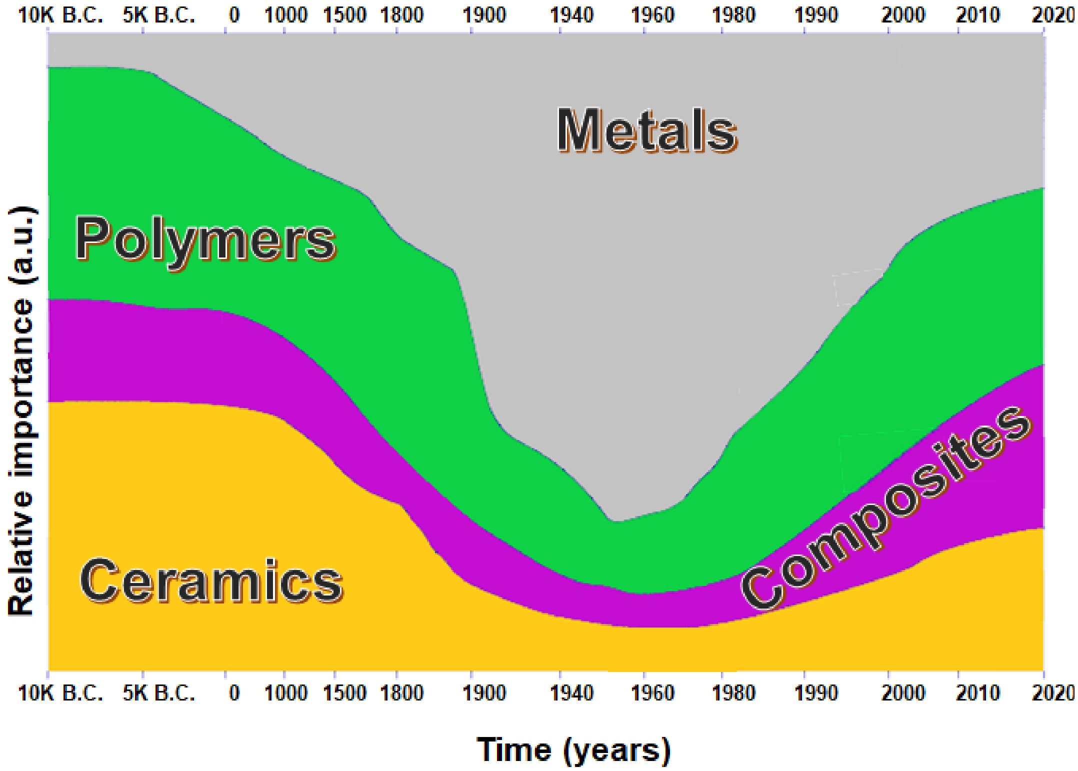

Figure 5. The relative importance of metallic, polymeric, composite and ceramic materials over time [18].

The high interest in developing these "materials of second-generation", compared with the conventional one, can be also explained through the need of the nowadays society to introduce different harmful particles in materials that encapsulates them and take advantage of their characteristics. Therefore, the challenges related to composite material manufacturing are overcome daily, however, an answer to one question starts two more questions, and so on.

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Chapter 1

Composites Matrices and Reinforcements

The composite materials have been designed to replace the traditional ferrous and non-ferrous materials, which exhibit different types of shortcomings in terms of performance, production and processing processes, geometric complexities, fields of use and manufacturing costs.

Composite materials are materials with anisotropic properties, consisting of several components, whose organization and development allow the use of the best characteristics of the components. The composite materials consist of a matrix (plastic, ceramic or metal) and reinforcing elements (reinforcements), which are arranged in the matrix in different proportions and orientations. The reinforcement gives the composite material a high resistance and represents the main element for taking over the load, while the matrix has the role of connecting the material and transferring the external load to the reinforcing elements [1]. Depending on the type of matrix, the composites can be classified as metal matrix composites, polymer matrix composites and ceramic matrix composites [2].

1.1. Metal matrix composites

The metallic matrix composites have been developed due to the necessity of developing composites that withstand higher operating temperatures than those with the polymeric matrix. The alloys exhibit good properties that promote their use in the manufacturing of specific parts, such as good thermal and electrical conductivity, good mechanical characteristics, high machinability, low porosity etc. However, their main drawback is their high specific weight (since they have a density in the range of 1.74 to 7.0 g/cm^3), accordingly, the manufacturing of this type of composites is challenging. The main metal elements used in composites manufacturing are aluminum, magnesium, iron, titanium, copper, and their alloys, which can be used in solid, liquid, or vapor states, depending on the manufacturing technology [3–5].

Considering the main reason of developing this type of composites, i.e., high-temperature resistance, the reinforcing elements are usually ceramic or metallic since those made of polymers don’t present compatibility for high-temperature applications. Moreover, the reinforcing elements can be introduced as particles and long or short filaments with different orientations (unidirectional, bidirectional, woven, or randomly distributed). Therefore, in the manufacturing of products that withstand temperatures lower than 450 °C, aluminum and its alloys are the ideal candidate for metal matrix manufacturing, because they have good fluidity and thermal conductivity, low density and price, and are easy to machining. To improve its disadvantage, i.e., refractory, the aluminum can be alloyed with titanium to create structures suitable for applications that exceed 800 °C [1,6–8].

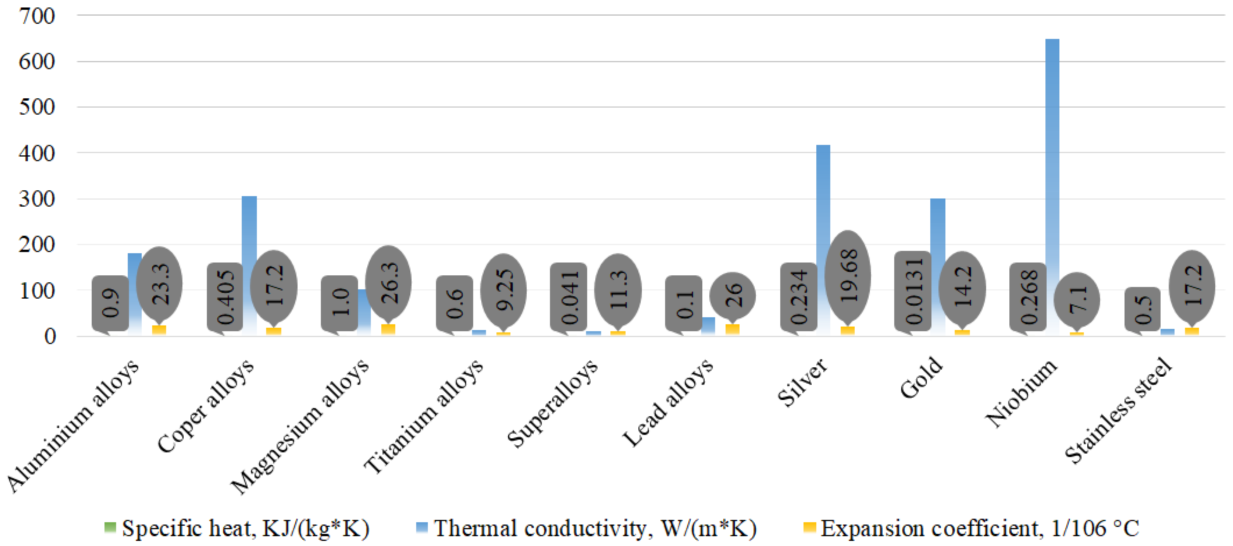

Figure 1.1. The thermophysical properties of the main alloys and elements used for metal matrices.

However, in the past few years, titanium and its alloys have become of great interest for the manufacturing of MMC, because their structure exhibit two phases α that resist up to 882 °C and β phase that appears above 882 °C. The titanium alloys have a good tensile strength to density ratio, especially when Al, V and Cr are used as alloying elements, also, they exhibit good ductility and linear expansion coefficient lower than that of iron, which boosts its use in high-temperature applications [9,10].

Even, they possess higher properties than conventional materials, the industrial development of MMC was hampered by their high manufacturing costs and economical aspects related to the manufacturing technologies and equipment.

The properties of MMC, as for any type of composites, are strongly influenced by the compatibility between the matrix and the reinforcing elements. Because the interface zone has different properties compared to the matrix or the reinforcements, and the performance of the final material depending on its characteristics, in some cases, this zone can be considered the third element (the matrix, the reinforcing elements and the interface zone). For example, the conventional obtaining method of metal matrix composites involves the introduction of the reinforcing elements, in the solid-state, in the molten matrix, i.e., liquid state. The reinforcing elements will remain in the solid-state over the entire process, i.e., until the matrix will solidify. Therefore, the bonding strength between the matrix and the reinforcing elements will be poor, because they exhibit different thermal behavior. In the case of high-temperature applications, there will be no synergy between the properties the fibers will act as defects, reducing the characteristics of the matrix. In order to avoid this issue, the fibers can be treated or coated with different functional layers to improve the matrix adhesion [11,12].

Considering that any additional step, introduced in the manufacturing process, by coating the reinforcing elements, the production cost of the final material will increase. However, for the manufacturing of MMC reinforced with metal particles, the compatibility between the matrix and them can be improved by alloying the matrix with lithium, which will create active compounds which will activate the surface of the particles, improving the adhesion. Also, the reinforcements can be overheated and coated with copper or nickel films [13–15].

Another problem, related to the compatibility between the constituents, is the high reactivity of the reinforcements in the matrix, in some cases. Therefore, these undesired chemical reactions will result in the consumption of one of the constituents, therefore, the conversion of the MMC into a conventional alloy. However, this process can be reduced or eliminated by protecting one constituent or by the introduction of corrosion inhibitors. The method is usually used for the coating of boron or carbon fibers when they are used in reactive matrices [16].

According to the information presented, it can be stated that the matrix material has a complex role, not just to gather the reinforcing elements into a compact body, because its microstructure and properties will significantly influence the performances of the final material. This aspect is more important in the case of MMC since, despite the form of the reinforcing elements, particles, or filaments, the incorporation of the components can result in the changing of the matrix, at the microstructural level [17].

Usually, the matrix purity degree is influenced, because the presence of the reinforcing elements will influence the micro and macro-segregation, and the grains size. These changes will influence the characteristics of the matrix since the properties of metal material are highly dependent on the microstructure, especially grain size. Therefore, this aspect can also be considered when designing the composite with tailored properties. These advantages were observed by the scientists, since during the ’30 machining tools with tungsten carbide particles embedded in cobalt matrix were industrially manufactured. This composite was developed to combine the high hardness of the particles and the good mechanical properties of the matrix. During the ’60, a new type of MMC was developed to combine the properties of tungsten, carbon and boron fibers with nickel, aluminum, or titanium matrices. Accordingly, composites with aluminum matrix reinforced with boron fibers have been used to manufacture the spaceships [18–20].

Another discovery that boosts the development of MMC, was the introduction of intermetallic compounds which can result in specific compositions, with is related to the possibility of establishing clear ratios between the chemical elements of the composition.

On the other hand, an important step in the development of this category of composites is the use of intermetallic compounds - alloys from at least two chemical elements that have the property that the energy of the different species of atoms is higher than between the same species atoms [21].

These compounds are intermediate phases, which may form in fixed compositions and correspond to well-established reports between the chemical elements (cases where they are called compounds) or may extend over a wider range of compositions (so it will appoint variable composition compounds) [22].

It is characterized by its crystalline structures, distinct from those of the constituents, which means that they will have specific physical and chemical properties. They are usually remarkable in their oxidation resistance qualities and high temperatures, thermal stability, corrosion resistance in very aggressive environments, mechanical strength, etc.

Also, the category of intermetallic compounds includes shape memory alloys, whose industrial use in new and emerging technologies is increasing. Moreover, an important aspect is that these compounds do not contain rare metals, so they are relatively inexpensive.

In principle, composite materials are manufactured (including ceramic matrix) with particles from titanium and molybdenum intermetallic compounds. Relatively recently appeared composites with matrix based on intermetallic. These require special manufacturing techniques such as reactive or isostatic pressing at high temperatures, or vacuum pressing.

It can be appreciated that the expansion of industrial applications of metal composites is slow but safe, especially in the conditions of the appearance of some fiber (silicon or alumina carbide, for example) less expensive and with better compatibility with the metals used as a matrix. In addition, progress in developing technologies such as powder metallurgy, liquid forging, or isostatic pressing was recorded [23,24].

Today, two main types of metal matrix composites can be distinguished, depending on their field of use, as follows:

a) for large-scale applications, including transport, entertainment, and sports sectors, materials that can be obtained with not too high expenses, such as the aluminum alloys reinforced with silicon or alumina carbide particles;

b) for high-performance applications, such as the aerospace industry, the price of materials has much less importance, and the maximum interest was shown to light alloys (such as titanium), reinforced with unidirectional continuous fibers.

Simultaneously with the progress made in the production and use of metallic matrix composites, concerns were widened for the analytical and experimental study of their properties, which is materialized by the quantitative and qualitative increase in the literature in the field. Note that this increase occurs after decades in which polymeric composites have held the supremacy in researchers' concerns [11].

Currently, aluminum alloy composites containing alumina fibers, which have a much lower density than steel, but comparable or superior stiffness properties.

Moreover, it should be pointed out that there are some important advantages of metal matrix composites, compared to those based on plastic matrix [25,26]. The following are the main advantages:

- dimensional stability, due to low coefficient dilatation;
- control of material properties by applying heat treatment;
- small porosity;
- insensitivity to moisture and radiation;
- electrical and thermal conductivity;
- machining by cutting or by plastic deformation;
- ignition resistance, but also at low temperatures;
- the possibility of making the composite material "in situ" (e.g., by directed solidification of the eutectic alloys) [17,27].

Of course, weaknesses of metal composites can also be found. These concerns, for example, are the mediocre values of their ductility properties, or sensitivities to structural defects and cracks.

However, the technical conditions required for the development of metal matrix composites of which optimized properties, by adjusting the composition, structure and/or conditions of elaboration, in accordance with the field of applications a designer with the needs contemplated by a particular user.

Metal composites are increasingly used in cutting-edge applications [28,29], such as:

- to make components that operate at high temperatures, in motors, turbo-reactors and rockets, composites with nickel and cobalt matrix, carbide fiber, or intermetallic compounds (TAC, NIC, WC, Al_2O_3) are indicated.
- wear-resistant car parts and high temperatures, under the conditions of a smaller weight of the steel parts, are manufactured from the aluminum alloys of ceramic fibers of SIC or $A1_2O_3$.
- for turbine parts, which must have high refractivity (i.e., to withstand well at 1200-1300 °C), oxidation and corrosion resistance, resistance to thermal shocks, reliability, and structural stability at large thermal gradients, are used composites obtained by the directed solidification of alloys based on Ni, SB, CR and Al.
- for drilling tools in hard or high depths, composites were made by introducing diamond granules and metal carbides into Sn-Ni alloys.
- for aerospace constructions, aluminum alloys with carbon or boron fibers are widely used due to the advantageous ratio between mechanical properties and specific weight.
- the solidified eutectic composites (InSb, NiSb) having super-conductibility or magnetic properties are widely used in electronics for non-contact switches, variable control resistors, transducers etc.
- from magnetic powders (from waste or reversed magnets) incorporated into a low melting alloy, solidified under the influence of a magnetic field, permanent magnets are obtained which are easily deformed and machined by cutting.
- Also, composites have been developed, such as armed aluminum alloys with ceramic particles, for example in the manufacture of carcasses for airplane control. These materials have the modifiable heat expansion coefficient depending on the composite recipe, high thermal conductivity, as well as good mechanical properties, in the conditions of a small density.

Metal composites are a distinct category of materials, composites with organic matrix, these materials can be used at higher temperatures, ignition resistant and at the action of organic solvents, and the face of ceramic composites has better tenacity and can be processed easier [2].

The most famous metallic composites have matrix based on Al, Mg, Ni or ferrous alloys and superalloys, the dispersed phase (in the form of fibers or particles) being made up of graphite, B/W, oxides (Al_2O_3, TiO_4, ZrO_2, Al_2O_3-SiO_2), nitrides (Si_3N_4, ALN) or carbides (SIC, ICT) [17].

In multiple applications in which special tribological properties are required, it is recommended to use metal composites with particles. These composites are easier to obtain, compared to those with fibers, and are therefore cheaper, which is why they have gained a great spread [11,30].

Metallic composites are achieved to the fullest extent by casting or powder metallurgy (as metal powders are used by Co, Ni, Cr, W, Mo, Ti, and as ceramic powder material of $A1_2O_3$, MgO, carbides, nitrides, borides etc.).

The criteria underlying the choice of complementary phase are related to its properties and geometric shape: density, elasticity module, breaking resistance, melting point, thermal dimensional stability, fiber or particle size, physical and chemical compatibility with the material the matrix. A factor of such importance is the cost of production, which is reflected in the final price of the composite material.

1.2. Polymer matrix composites

The properties of the composite material are totally different and clearly higher than those of the individual components [31].

The basic characteristic of polymer composites is the very high strength/weight ratio (after this characteristic, composite materials outperform the best steels).

According to the nature of the components participating in the formation of polymeric composite materials, they are classified in [32,33]:

- polymer composites with porous fillings;
- macroscopic composites;
- thermoplastics: polyphenylene sulfide (PPS); polysulfide (PS); poly (ether-ether) ketone (PEEK); polyether ketone (PEKX polyphenylene sulfone (PPS); polyethersulfone (PES); polyiodide (PAI);
- thermoactivated: polyimide (PI); polystyrene pyridine (PSTP);
- elastomers – silicones.

The use of one or other of the polymers mentioned is dictated by the field of use of the composites obtained on the basis of them.

Unsaturated polyesters are macromolecular compounds obtained by polycondensation of diols with saturated or unsaturated diacids. The unsalted acids that make up these resins give the product a certain degree of unsaturation. These double bonds allow the crosslinking of the resin with the help of polymerization monomers. Due to this, unsaturated polyester resins have the advantage that they can be used in very thick layers, in a single application, which greatly reduces labor and time required for application and avoids solvent losses, its role being taken by the monomer itself [34].

Synthesis of unsaturated polyesters. Standard polyester resins are obtained by polycondensation of maleic anhydride with Salic anhydride and 1,2-propylene glycol. Polycondensation takes place in the melt, and the product obtained is dissolved in a convenient monomer, preferably styrene.

The process takes place in two stages. The first consists of the opening of anhydrous cycles, a strongly exothermic process, which causes the temperature of the reaction mass to rise from 80 °C to 150-170 °C.

The retransmitted of this process can be reduced by replacing some of the Mayan anhydrides with fumaric acid. in this case, the reaction mass is heated to only 100 UC to initiate the reaction.

The second stage consists of the postherniation of the semi-esters and takes place at much higher temperatures (165 - 225 °C). The cooling process takes place at equilibrium and to move it to the right it is necessary to remove water from the system. This can be accomplished by vacuum distillation or azeotropic distillation using toluene as a starting agent, the latter process having great practical maturity [35].

In order to avoid thermal destruction processes, the reaction is conducted in an inert atmosphere, using nitrogen or carbon dioxide for this purpose.

The overall reaction for obtaining unsaturated polyesters is as follows:

The process is aimed at periodically determining the viscosity and acidity index. During the synthesis process, a series of side reactions occur.

The unsaturated polyester obtained in the end has an average molecular weight between 1200 and 3000 and an acidity index between 15 and 50 mg KOH/g.

The strengthening of unsaturated polyesters takes place by copolymerizing the double bonds present in macromolecules with vinyl monomers, acrylics etc. For this purpose, the resin is delivered as a monomer solution. The copolymerization process takes place through a radical mechanism. In order to prevent gelling during storage, inhibitors (hydroquinone, P-benzoquinone, substituted phenols etc.) are introduced in an amount of 5n-100 ppm. The curing process takes place in the presence of initiators or redox systems. Depending on the nature of the initiator used, curing takes place hot or at room temperature [36].

The hardening process comprises several stages: initialization (when the stabilizer is consumed); gelling, hardening and setting, or post-baking (reactions in the solid-state of the resin).

The monomers used in the hardening process of the resins must meet the following conditions: after the hardening of the resin to give homogeneous and transparent films; to copolymerize with the double bonds of the resin and to homopolymerize (the homopolymer causes the appearance of blurs); have a low evaporation rate; to dissolve the resin well; boiling and ignition points to be as high as possible; copolymerization should take place under acceptable conditions [37,38].

The properties of unsaturated polyester resins depend on their molecular mass, degree of unsaturation and chemical composition. Increasing the molecular weight improves the main mechanical properties, increases the hardness, temperature resistance and takes chemical agents.

The degree of unsaturation of the resin is regulated by the saturated acid to saturated acid ratio and determines the crosslinking density and. thereby the hardness of the resin and the resistance to temperature. For standard polyester resins, this ratio may vary.

The chemical composition of the resin is the decisive factor that influences its properties. This allows by varying the type of unsaturated acid. of saturated acid or polyol to obtain a very wide range of properties, depending on the purpose pursued. Thus, the use of aromatic diacids leads to the increased melting point, increased hardness, improved resistance to temperature and chemical agents. The nature of glycol also influences the hardness, mechanical properties, chemical resistance, melting points and flame resistance of the resin. The nature and concentration of the monomer influence the properties of the hardened product [39].

According to previously mentioned characteristics, various types of unsaturated polyester resins are obtained in practice.

For each type of resin, it is important to know the properties of pure resin diluted in monomer, the properties of unreinforced crosslinked resin and the properties of crosslinked resin reinforced with various agents.

The properties of dilute pure resins differ from each other by the chemical composition of the unsaturated polyester, by the nature of the monomer used for dilution, by dilution, by the nature of the inhibitor etc.

Analyzing the possibilities of synthesis and properties of polyester resins, they show that they have the following distinct characteristics [40]:

- easy obtaining and accessibility of raw materials;
- rapid hardening, without elimination of by-products;
- good dimensional stability;
- good mechanical characteristics of the reinforced shape;
- multiple coloring possibilities;
- transparency;

- good chemical resistance to the action of non-oxidizing acids and alcohols in the use of polyesters must take into account the following limits;
- high shrinkage during formation (which can be diminished by loading, reinforcing, or adding thermoplastic adjuvants.);
- poor flame behavior of standard resins (improved by the use of halogenated diacids or diols and by the introduction of specific additives);
- low resistance to alkalis and hot water.

The main areas of use of unsaturated polyesters are:

Constructions: flat or corrugated slabs, translucent or opaque tiles, used for roofs or facade elements, sanitary units, fireplaces, street furniture, telephone booths, concrete formwork, funerary monuments, construction site huts, prefabricated signage panels etc. [41].

Tanks, cisterns, pipes: wine tanks and presses, liquid fertilizer tanks, silos (for powdery materials, cereals etc.), marine containers, sewage treatment plant pipes, septic tanks, collectors, ventilation pipes, gas washing towers, chemical transport pipes [42].

Electrical and electronic equipment, cabinets, boxes, enclosures, insulators, cable fireplaces, antennas, insulating booths, support for printed circuits.

Transport, car body parts, bumpers for cars, cabs for trucks and tractors, utility bodies (isothermal trucks, refrigerators), bodies for sports cars [43].

Nautical and sports industry: sports boats, accessories (buoys, pontoons, frames, etc.), cable cars, fishing rods [44].

1.3. Ceramic materials

The development of CMC materials was conditioned by the emergence of high-temperature-resistant fibers, such as SiC fibers because the fibers that melted at low temperatures were destroyed during the sintering process of ceramic materials. An important step was the discovery of SiC fibers.

Brittle ceramic materials need a reinforcing component to reduce the area under the stress-strain curve which is directly proportional to the hardness of the material. In ceramic materials, the reinforcing fibers act as bridges against cracks, creating a compressive force at the edges of the crack, thus preventing its development [45,46]. Also, the tubular reinforcing elements will absorb some of the damaging energy, i.e., slowing the propagation of the cracks, by "pulling" the matrix together. In order to intensify this “pulling” process, the fibers are covered with different types of layers that improve the adhesion of the matrix to the surface of the fibers. The deposition method varies depending on the nature of the fiber [47,48].

Aluminum oxide (Al_2O_3) is the most widely used ceramic material for the construction of artificial prostheses. This is a fragile material that cannot be used to construct hip implants without reinforcing elements. By adding SiC fibers to the aluminum oxide matrix, the resilience of the implant increases by up to 50%. SiC-reinforced aluminum oxide is also used in the construction of high-durability wood and metal cutting tools. The presence of graphite fibers in a carbon matrix leads to the emergence of another important class of ceramic matrix composite, known as, carbon-carbon composites (CCM). CCM exhibit excellent heat resistance, wear and shock resistance. These specific characteristics boost their introduction in aircraft manufacturing, especially in the production of braking system components.

1.3.1. Ceramic composite materials

Ceramic-composite materials (MCMC) are generally formed of ceramic fibers in a ceramic matrix, as can be seen in Figure 1.2. Ceramic matrix composite materials are designed to improve the main disadvantage of monolithic ceramic material, fragility. They are called reverse composites because the tensile strength of the matrix is lower than the tensile strength of the fibers, which is the opposite of most polymer matrix composites or metal matrix composites. Therefore, it is the matrix that yields first. To prevent premature failure of the reinforcing fibers when microcracks appear in the matrix, the connection between the fibers and the matrix must be checked during processing. Composite materials with the ceramic matrix are hard materials and will have a low strength if the connection between the fibers and the matrix is too weak or too strong, in this case, a fiber coating treatment will be done, called the interface. The manufacture of ceramic matrix composite materials requires specific processing techniques. It is preferable to form the matrix and the interface around the fibers during the transition from the gas phase to the liquid phase (or a combination of both), from gaseous or liquid precursors [49,50].

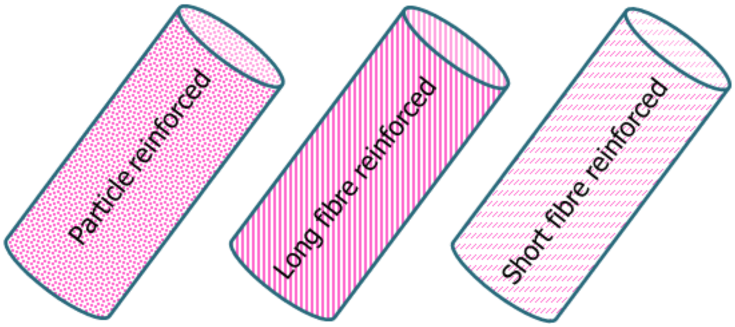

Figure 1.2. Types of composite materials according to the shape of the reinforcing fibers.

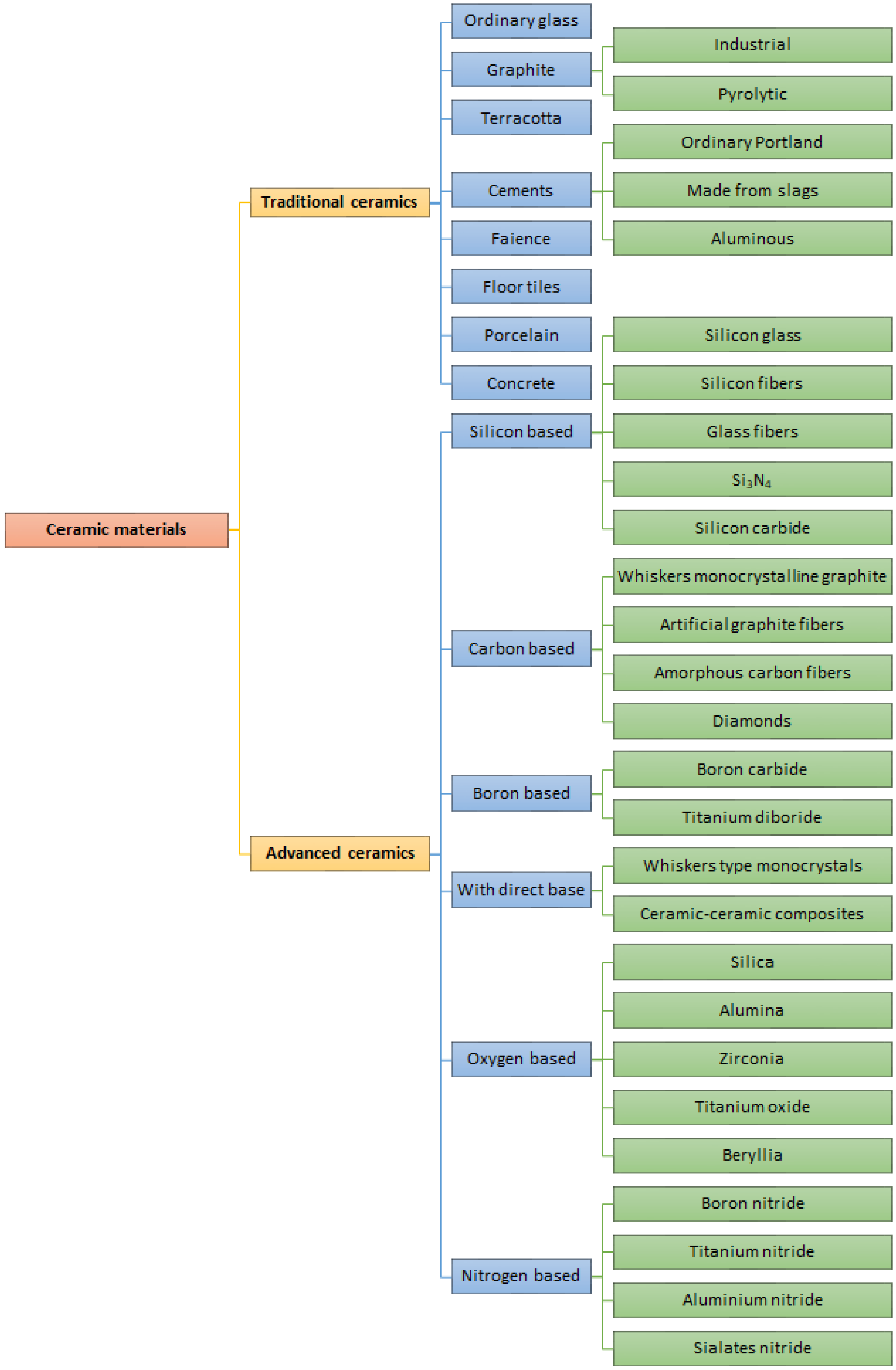

Figure 1.3. Ceramic materials classification by the composition and the nature of the structure.

The classification of ceramic materials is difficult due to the very wide field regarding both the composition, structure and properties, as well as the manufacturing technologies of the components and their destinations. If the nature of the material structure is accepted as a classification criterion, then ceramics can be: traditional ceramics and technical ceramics

Traditional pottery, as shown by the Greek word Keramik's (burnt clay) is the transformation of clay raw materials into pottery and bricks by burning them. Currently, in addition to traditional ceramic materials, there are also advanced technical ceramics consisting of oxides (natural and synthetic), carbides, nitrides, boron, technical carbon etc., or technical ceramics resulting from the reunion of two or more chemical compounds.

The classification of materials according to the nature of the structure and composition is shown in Figure 1.3.

Ceramic matrix composites are, usually, used in environments with severe working conditions, such as those that will expose the material to high temperatures, mechanical stress and corrosive agents, at the same time (flue gases systems). Commonly, for this type of application, the composites that include a non-oxide ceramic matrix are ideal, because they possess high refractoriness and don’t react with the flue gases. Currently, the components of this system are manufactured from silicon carbide (SiC)/SiC composites, carbon (C)/C composites, or C/SiC composites, where, the first component represents the material used for the manufacture of the reinforcing elements [5].

The properties of ceramic materials are [51]:

- Good chemical stability;
- High wear resistance;
- High mechanical strength;
- Good hardness;
- High melting point (refractoriness);
- Low density.

Therefore, the development of the ceramic composite was mainly stimulated by the high-temperature resistance superior to other types of materials. As can be seen from Figure 1.4. the ceramics are superior to both polymers and metals.

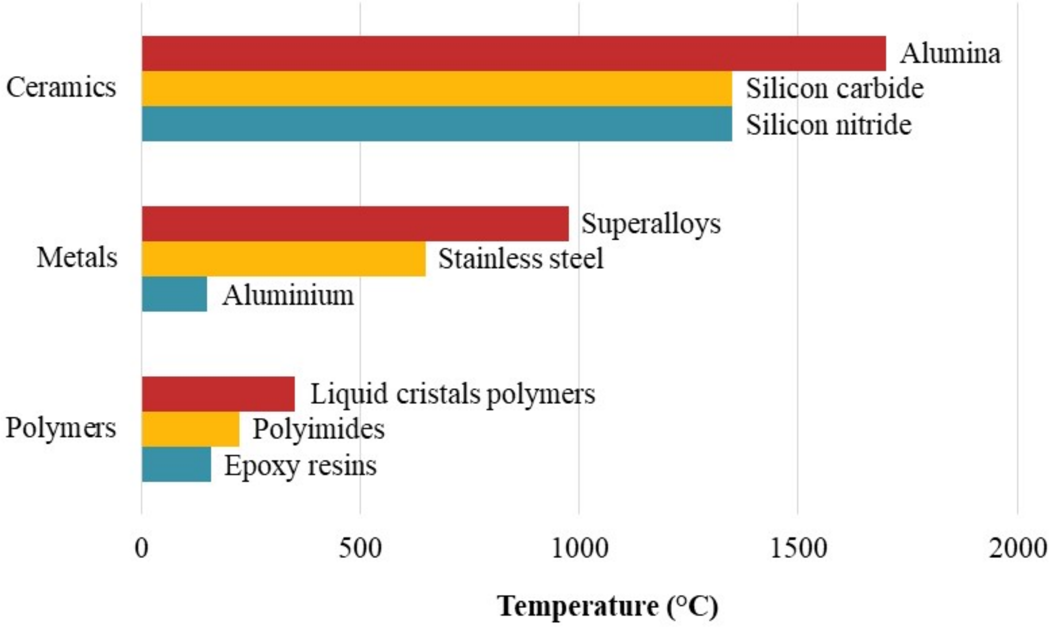

Figure 1.4. Maximum temperatures of use of different material structures [52,53].

Also, besides their refractoriness, ceramic materials are used in industry due to their low density and low coefficient of thermal expansion (Figure 1.5).

In addition to the favorable characteristics, ceramic materials also have some unfavorable characteristics listed below [54,55]:

- Complicated and quite expensive manufacturing process.
- Low crack resistance (Brittle behavior, consequently, low tensile strength)
- Low thermal shock resistance (Advanced ceramic materials have low tensile and impact resistance. Unlike metals, they have low plasticity and are prone to breakage or cracking from thermal or mechanical shock.)

There are three constituents in ceramic matrix composites: a fiber, a matrix and, in general, an interface produced by covering the fibers. The type of material, the volume concentration and the arrangement of the constituent elements depend on a number of considerations, including the functionality of the part to be manufactured, the operating conditions and its cost. Tables 2.1 and 2.2 show the oxide, non-oxide matrices and typical reinforcements used [56].

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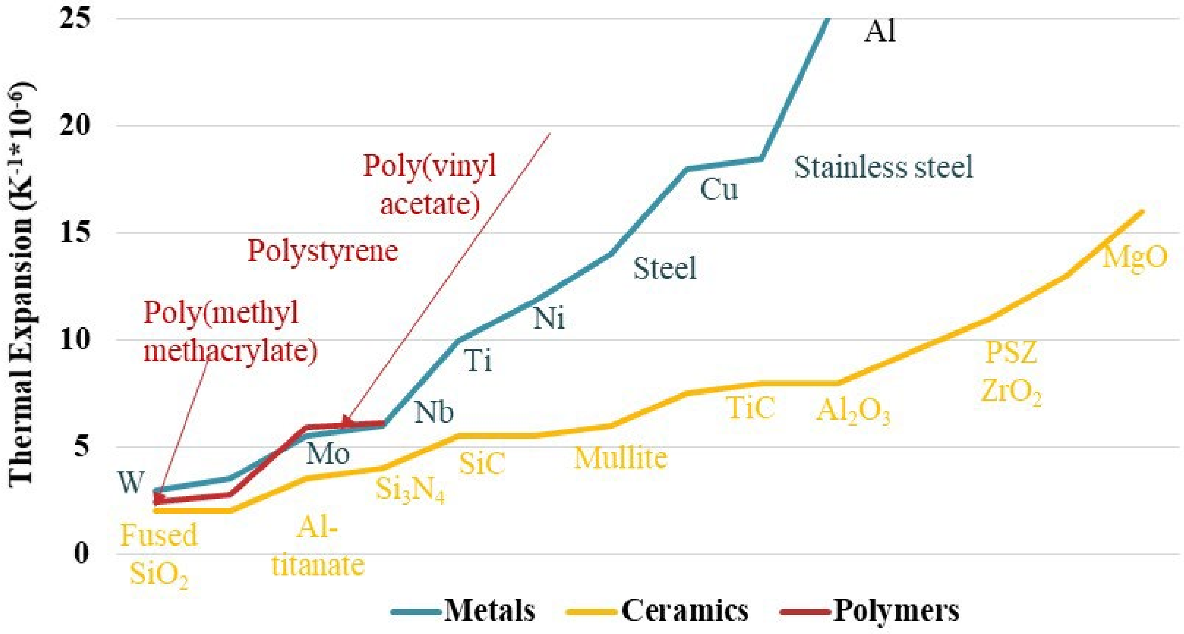

Figure 1.5. The thermal expansion coefficient of common materials.

Table 1.1. Tensile strength of common materials [6].

Material	**Type**	**K_{Ic}, MPa*$m^{1/2}$**
Polyethylene	**Polymers**	1-2
Nylon		3
Epoxy resin, polyester		0.5
Pure metals (coper, aluminum)	**Metals**	100-300
Aluminum alloys		20-50
Titanium alloys		50-100
Hypoeutectoid steel		50
Cast iron		4-10
Glass with SiC	**Ceramics**	0.5-1
Magnesium oxide		3
Alumina		3
Silicon carbide		2-4
Silicon nitride		3-5

Table 1.2. Non-oxidic matrices, other than carbon and typical reinforcements [17].

Matrix	Particle	Powder	Filaments	Fibers
SiC		SiC	B_4C	C, SiC
Si_3N_4	SiC, TiC, TiN, TiB_2	Si_3N_4	Si_3N_4	SiC, Al_2O_3, C
$MoSi_2$	ZrO_2, TiB_2, Nb, SiC, Si_3N4	Al_2O_3	SiC	SiC, Al_2O_3
B_4C	B_4C	SiC	SiC	SiC, C
BN (hexagonal)	B_4C			
AlN			SiC	
TiB_2	B_4C, BN			
TiN				
TiC				

Table 1.3. Oxide matrices and typical reinforcements used [7].

Matrix	Particles	Powders	Filaments	Fibers
Alumina, Al_2O_3	ZrO_2, SiC, TiC, TiN, TiB_2, ZrB_2, metals	SiC	SiC, B_4C	SiC, Al_2O_3, $3Al_2O_3$, $2SiO_2$
TZP*	Al_2O_3	Al_2O_3	Al_2O_3	
PSZ**	Al_2O_3	SiC		
Mullite, $3Al_2O_3$-$2SiO_2$	Al_2O_3, ZrO_2	Al_2O_3	SiC	SiC, Al_2O_3
Yttrium- Aluminum (YAG)			Al_2O_3	Al_2O_3
Magnesium aluminate, MgO, Al_2O_3	Al_2O_3			
Beta- alumina	ZrO_2			

**TZP- zirconia tetragonal polycrystals, contains a metastable tetragonal ZrO_2 in a large proportion*
***PSZ- partially stabilized zirconia with an insufficient stabilizer to form a cubic phase material*

Currently, both oxide and non-oxide fibers are used in different industrial applications. For example, in the manufacturing of insulation materials, the alumina core fibers, which belong to the refractory oxide fibers category, are used, mainly due to their high resistance to oxidizing atmospheres. However, due to granular growth at high temperatures, they exhibit lower creep resistance and tensile strength. Accordingly, in industrial applications, usually, mixtures of aluminum oxide fibers and silicon oxide fibers are used, due to their working temperature range between the limit of 1000-1100 °C. In some cases, non-oxide fibers, such as carbon and SiC-based fibers can be included. Nevertheless, the oxide fibers show a different behavior, instead of losing their strength and other properties, the oxide fibers have a lower density, therefore, they exhibit high mechanical strength and good creep resistance even at high temperatures. For example, up 1500 °C - 2000 °C the carbon fibers show an increase of breaking strength with the increase of temperature. Accordingly, SiC fibers exhibit their highest resistance in the 1400 °C - 1500 °C temperature range. Also, the peak of the highest value for conductivity or insulation (depending on their composition and microstructure) is presented in the same temperature range. Still, the main problem of the oxide fiber remains, above 500 °C (for most of them) and 800 °C (for SiC) they will react with oxygen [57,58].

Considering the design of fiber-reinforced composites, it must be stated that, the properties of the final material highly depend on the fiber's arrangement in relation to the type of load and its direction. They are rarely unidirectional, most often multidirectional. Those oriented in several directions are often found in composite materials with ceramic matrix, the raw material is a preform made of fibers, i.e., a self-contained porous body made of continuous or short fibers that have approximately the shape and the sizes of the manufactured part [59].

The interface plays a key role in ceramic matrix composites, although it represents a very low percentage of the volume of the material. This is a thin layer (<1 μm) deposited on the surface of the fibers or formed during the manufacturing process exhibit three important functions: (i) crack deflection (acts as barriers in cracks propagation, (ii) load transfer (anchors the fibers to the matrix, which makes them act as a single body) and diffusion barrier (in those composites where the matrix reacts with the surface of the fibers). According to different studies [55], the best materials that can be used as interface are those with a layered crystal structure (such as hydrocarbons (PyC) or hexagonal boron nitride) or those possessing a layered microstructure (such as multilayer hydrocarbon-SiC (PyC – SiC) n, where n = 5-20). Moreover, the material performance will be also depending on the fiber's arrangement. Figure 1.7 shows the most suitable alignment of the fibers to increase the crack deflection, accordingly, the layers should be oriented parallel to the surface of the fibers.

Non-oxide matrices generally comprise carbon, silicon nitride and silicon carbide. Depending on their microstructure, the carbon matrices offer a variety of properties. The hydrocarbon can be isotropic, non-graphitizing and insulating on the one hand, or anisotropic (laminar), graphitizing by heat treatment at temperatures in the range of 2500 °C – 3000 °C and strongly conductive (the so-called hard-rolling used in C/C braking systems), on the other hand. The use of carbon in composites manufacturing was sustained by its lightweight (1.8-2.1 g/cm^3), biocompatibility (the composites type C/C have been successfully used for prostheses manufacturing) and refractory (>3500 °C). Compared to carbon, SiC is denser (3.2 g/cm^3) a less refractory (>2400 °C) [60]. This is extremely harsh but more interesting is the fact that it reacts with oxygen, creating a surface protective layer of silica, this being the main reason of using SiC fibers at temperatures up to 1500 °C in oxidizing atmospheres. However, in atmospheres with low oxygen content, the reaction produces SiO which doesn't provide enough protection. Silicon carbide matrices can be used in mixtures of SiC with Si and C or in SiC-SiC. When free Si is introduced in the matrix, the working temperature limit decreases to a maximum of 1400 °C, while in the case of free C in the matrix, the working limit remains unchanged. Compared to SiC, silicon nitrate matrices exhibit lower conductivity and thermal stability, yet, the other properties (mechanical, chemical etc.) are similar [61].

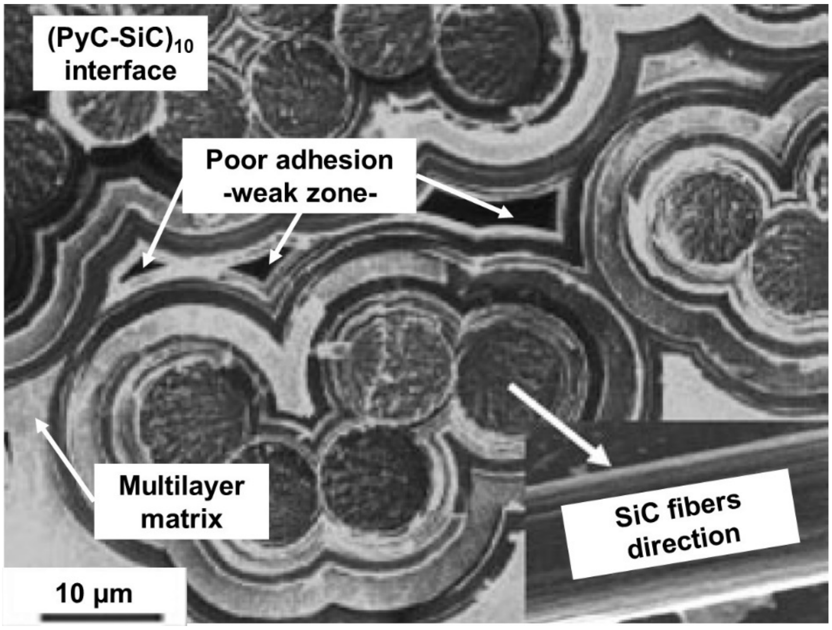

Figure 1.7. Multilayer interface with "self-healing" matrix in a (PyC-SiC)$_{10}$ preform [55].

When choosing a particular fiber-interface-matrix system, several aspects must be taken into consideration. First, the constituents must be compatible. Accordingly, the thermal expansion coefficient of the fibers and the matrix must exhibit close values. Also, the coefficient of radial thermal expansion of the fibers must be considered, because the matrix "catches" the fibers during cooling from the processing temperature to the ambient temperature, resulting in a strong bond between the fibers and the matrix and a brittle behavior [53]. On the other hand, during cooling, the fibers and the matrix can detach from each other. Also, in terms of chemical compatibility, the matrix and the fibers should exhibit similar properties. Reactions may affect the initial strength of the fibers and/or alter the connection between the fibers and the matrix [62]. For example, SiC fibers react with silicon-based glass-ceramic matrices with a suitable interface, such as the hexagonal boron nitride dual interface and SiC, to be used. To fulfill all these requirements, the matrix and the fibers usually have relatively close compositions in most of the ceramic composites.

Ceramic matrix composite materials must be compatible with the atmosphere in which they will operate. Usually, these working atmospheres are oxidizing, due to the presence of air or combustible gases. Therefore, the non-oxidizing materials will be ideal. However, there are two things to solve this problem: firstly, the use of different types of protective layers or oxidizing inhibitors; and, secondly, designing self-healing interfaces and matrices. For example, to decrease the oxidation rate of carbon matrices, phosphorus and boron components can be introduced as inhibitors. Multilayer coatings that contain SiC or B (glass formers) can also be used to reduce the diffusion of O in C/C composites. The concept of a “self-healing” multilayer interface boosts the development of advanced SiC matrix composites because they provide a significant increase in durability in oxidizing atmospheres at high temperatures [63,64].

The possibility to introduce other types of fibers, not ceramic, is significantly reduced by the low refractoriness. The polymeric fibers withstand temperatures lower than 500 °C, while conventional glass fibers exhibit softening or melting below 700 °C, therefore, they also cannot be used for the ceramic composites reinforcing [65].

The fiber-reinforced ceramic composites exhibit the following characteristics [28,66]:

- continuous fibers (woven, unidirectional, bidirectional etc.);
- good creep resistance;
- small diameter, <20 mm (depending on application);
- high tensile strength in the air or other hostile environments;
- high modulus of elasticity.

The main objective in the design of CMC is the development of an energy dissipation mechanism. Accordingly, the presence of particles, filaments, or continuous fibers increases the mechanical strength properties in three mechanisms:

- the deflection mechanism of cracks propagation;
- energy absorption, due to the phenomenon of fibers "pulling";
- cracks bridging.

Generally, the CMC includes different types of glass, ceramic-glass mixtures, or ceramics such as C, SiC, Si_3N_4, aluminides, or oxides.

The percentage ratio between the reinforcing element and the matrix is usually subunit or equal to 1 for ceramic matrix composites. This ratio between a polymer and a metal matrix is usually between 10 and 100. Ceramic matrix composites have a low ratio, which means that the reinforcing element and the matrix have approximately the same load-bearing capacity.

Advantages of fiber-reinforced ceramic composite materials [67–69]:

- high mechanical strength and strength-density ratio;
- high resistance to temperatures (refractory);
- low density;
- high stiffness-density ratio;
- high resistance to thermal shock and impact;
- high wear resistance;
- high creep resistance;
- controllable conductivity and thermal expansion;
- high hardness and good resistance to erosion.

1.3.2. Basic ceramic materials

Table 1.4. Types of basic ceramic materials [9].

Base material	Chemical formula	Type	Application
Barium titanate	$BaTiO_3$	Oxide materials	Functional materials
Lead zirconate titanates	$Pb(Zr,Ti)O_3$		
Ferrite	$M^{2+}O{\cdot}Fe_2O_3$		
Alumina	Al_2O_3		Structural materials
Forsterites	$2MgO{\cdot}SiO_2$		
Zirconia	ZrO_2		
Zirconia silicate	$ZrO_2{\cdot}SiO_2$		
Mullite	$3Al_2O_3{\cdot}2SiO_2$		
Steatite	$MgO{\cdot}SiO_2$		
Cordierite	$2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$		
Aluminum nitride	AlN	Non-oxide materials	Functional materials
Silicon nitride	Si_3N_4		Structural materials
Silicon carbide	SiC		

1.3.2.1. Barium titanate

Barium titanate ($BaTiO_3$) is a white-transparent inorganic compound with large crystals. $BaTiO_3$ possesses ferroelectric characteristics, with piezoelectric properties and a photorefractive effect. In solid-state it can be obtained in five phases: rhombohedral> orthorhombic> tetragonal> cubic> hexagonal, from low to high temperatures. Except, the cubic phase, all the phases exhibit a ferroelectric effect. The cubic phase at high temperature consists of octahedral centers of TiO_6 that define a cube with Ti at the tops and Ti-O-Ti groups on the sides. In the cubic phase, Ba^{2+} is located in the center of the cube with a nominal coordination number 12 [70]. The phase with low symmetry is steady at low temperatures, due to the exit of the element Ba^{2+} from the central position [70].

$BaTiO_3$ is a dielectric ceramic material used in capacitors. Barium titanate ceramics with a perovskite structure have values of dielectric constant up to 7000 F/m, but other ceramics, such as titanium oxide have values between 20 and 70. At temperatures above 130 °C values of up to 15000, while the best-known ceramic and polymeric materials have less than 10 [71].

Due to its piezoelectric properties, $BaTiO_3$ is mainly used for the manufacture of microphones or other transducers. At room temperature, its spontaneous polarization reaches 0.15-0.26 C/m^2, with a Curie temperature range between 120 °C and 130 °C. The differences are given by the growth technique, high purity of the crystals will result in high spontaneous polarization values [72].

Barium titanate has been replaced as a piezoelectric material by lead zirconate titanate in most applications. However, it has been used in thermistors and self-regulating heating systems due to the positive temperature coefficient. $BaTiO_3$ crystals are also used in nonlinear optics. In the case of photorefractive applications, $BaTiO_3$ can be doped with iron to obtain corresponding characteristics.

Barium titanate is used for certain cooling sensors due to its good pyroelectric and ferroelectric properties. Also, for the energy storage systems, used in the manufacture of electric vehicles, $BaTiO_3$ with high purity shows suitable properties [73].

1.3.2.2. Lead zirconate titanate

Lead zirconate titanate, $Pb\ [Zr_xTi_{1-x}]\ O_3$, where x has values between 0 and 1, is an inorganic intermetallic compound, also called PZT, with the piezoelectric effect that show suitable characteristics for the electro-ceramic field. Also, PZT is an insoluble white solid substance [74].

One of the most studied chemical compositions of lead zirconate titanate is $PbZr_{0.52}Ti_{0.48}O_3$. PZT can be used in the manufacture of sensors for thermographic cameras. They are used both in the thin layer and as a bulk structure. Moreover, PZT is used in the manufacture of ultrasonic transducers, actuators, and other sensors, as well as FRAM processors and capacitors. It is also used in the manufacture of ceramic resonators for reference synchronization in electronic circuits. PZLT can, also, be used for goggles to protect pilots' eyes from very bright light, such as those produced by nuclear explosions that could cause burns or blindness. PLZT lenses can become opaque in less than 150 microseconds [75].

From a commercial point of view, PZT isn't used in the pure state, it is usually doped with elements that create vacancies after interacting with metals or oxygen, which facilitates the movement of atoms. In general, when the elements react with oxygen, they will result in a hard material, while when they will react with metals, the final materials will be softer. Also, the doping element will influence the piezoelectric constant, as it is correlated with the polarization or the electric field which is generated by the mechanical stress or an electric field. In general, soft PZT exhibit a higher piezoelectric constant, but there are higher material losses due to internal friction. In the hard PZT, the movement of atoms is constrained by impurities thus reducing material losses, but the piezoelectric constant is low [76].

1.3.2.3. Ferrite

Ferrite (Fe_2O_3) is a ferromagnetic electric insulator, which can be used in ceramic materials in combination with other metallic elements. Depending on their demagnetization resistance and magneto-coercion, Fe_2O_3 can be divided into two categories. For example, in the manufacture of magnets for refrigerators, speakers, or electric motors, the hard ferrite is usually used due to its high coercivity. However, when low coercivity is required, such as in the manufacture of cores for transformers or inductors, soft ferrites are ideal [77].

Also, ferrite cores can be used in the manufacture of electronic indicators, transformers, and electromagnets due to the very small losses of Eddy currents [78]. They can usually be seen as a node on the computer cable that helps prevent high-frequency current from flowing in and out of the equipment.

Computer memory storage units were made of hard ferrite. These were assembled from cells in basic memory. Ferrite powders (iron III) are used to cover the surface of magnetic recording tapes. Also, this type of particles can be used in the manufacture of radar-absorbing materials or as a covering material for "Stealth" aircraft and for the insulation of rooms where electromagnetic compatibility measurements are made [79].

Due to its superparamagnetic properties, ferrite is used in the construction of magnetic instruments for pickups.

1.3.2.4. Aluminum oxide

Aluminum oxide (Al_2O_3) is commonly called alumina or alkoxide, alkoxide depending on the chemical formula or applications. In its natural form, it is in the crystalline phase, polymorphic α-Al2O3 as a corundum mineral, from which precious stones, rubies, or sapphires are formed. Al2O3 is mainly used for the production of aluminum, as an abrasive material, due to its hardness, or as a refractory material, due to its high melting point [80].

Due to the fact that alumina is a well-balanced material, it is used in a wide range of applications, including the manufacture of wear-resistant parts, materials for industrial furnaces working at high temperatures, and various electronic components [81]:

- alumina is the best-known ceramic material due to its chemical and physical stability;
- thermal properties: resistance to high temperatures and high thermal conductivity;
- mechanical properties: high strength and hardness;
- other properties: the good electrical insulator, good corrosion resistance and biocompatibility.

More than 90% of the amount of alumina produced worldwide is used to produce aluminum. This is because converting bauxite to alumina naturally is the first step in obtaining aluminum. However, the interest in industrial applications of alumina as an independent material wasn't decreased. Currently, over 4 million tons of alumina are used as raw materials for different applications (excluding alumina used for the production of aluminum). The variety of applications of this material are related to its acid resistance, refractoriness and purity, as well as the possibility to mold it in complex shapes [82].

The most important industrial applications of alumina are in the field of ceramic materials manufacturing. Alumina is used as an insulating material due to its high thermal shock and dielectric properties. However, when high-frequency currents are present, high sintering temperatures and pure alumina are required. For example, in the manufacture of TV tubes, satellite transmitters, microwave generators, or powerful lasers [83].

In the electronics industry, alumina is widely used in the electronics industry in the manufacture of passive components, such as resistors, capacitors, or substrates for hybrid circuits or as materials for frequency resonators and capacitors [84].

Alumina is used to make slabs that secure fuel lines and flue pipes to power plants or coal from the inside to protect high-risk areas. They are not suitable for areas that require impact strength because they are fragile.

In the manufacture of ceramic tools, alumina is used due to its high hardness. Therefore, it came as the ideal candidate in the manufacture of cutting or abrasive tools. These types of ceramics are used in the manufacture of extrusion dies or grinding nozzles and for various parts of mining machinery (those applications that require high wear resistance). They are also used to manufacture different types of blades and high-temperature gaskets (such as those used at thermal engines) [85].

In the military industry, alumina is used due to its shock-resistant properties which makes it suitable for the manufacture of protective shields for tanks and helicopters, as well as bulletproof vests, and in the aeronautical field, they are used to protect hydraulic parts.

Another important use of alumina is in the manufacture of refractory materials. Refractors must be resistant to thermal shock, chemical attack, vitreous dissolution, compression, cracking, erosion, and heat transfer. These requirements required the use of a larger amount of alumina in refractory materials. As a result, a quarter of the amount of alumina, except the one used for aluminum obtaining, is used in the production of refractory materials [83].

In medical applications, alumina components were developed due to the fact that it is inert in ordinary chemical reagents. Also, because it can be polished up to a very smooth surface, alumina can be used as a protective layer for joining medical implants. Moreover, porous alumina can be used for the manufacture of dental implants (porous surface increases the hydroxyapatite growing rate and integration) and "spacer between bones". It is also used in the manufacture of medical instruments, such as tubes and other devices [86].

Enameling is the coating of the surfaces of insulators and other ceramic materials, such as tiles. Al_2O_3 contributes to the adhesion between metal and ceramic, opacity, corrosion and scratch resistance, important properties of enameling. Enamel is also used in the manufacture of decorative objects.

Alumina was also developed in chemical industry applications since the filters manufactured of this material are chemically inert and can be very small particles due to its fine porosity. Moreover, this property also promotes it as an ideal candidate for the obtaining of tanks used to store chemical agents or for the transport of chemically aggressive substances [87].

1.3.2.5. Forsterite

Forsterite (Mg_2SiO_4) is part of the series of solid olivine solutions, rich in magnesium. It is an isomorphic material, rich in iron, fayalite. Forsterite crystallizes in the orthorhombic system being associated with metamorphic and volcanic rocks [88].

There are two known polymorphic phases of forsterite: wadsleyite (orthorhombic) and ringwoodite (isometric). Both are found mainly in meteorites.

It is an excellent microwave insulator at high temperatures and is ideal for the manufacture of electron tubes and circuit substrates. Due to the high coefficient of thermal expansion, it easily creates bonds with metals and glass. Due to its high mechanical properties, forsterite is used as a biomaterial for implants [89].

The electrical properties also make it an ideal candidate for electronic substrates.

1.3.2.6. Zirconium dioxide

Zirconium dioxide (ZrO_2) or zirconia, is a white oxide, usually meet as baddeleyite, with has a cubic crystalline structure. Zirconia can be doped and synthesized into different colors that are used as gemstones or diamond substitutes [90].

Zirconia has excellent mechanical properties with superior tenacity and breaking strength to alumina. It is used in milling machines, sliding parts and cutting blades [91].

This is a good thermal insulator with a thermal conductivity 90% lower than other ceramic materials.

The main use of zirconium dioxide is in the production of ceramic materials but it also can be used in the obtaining of protective layers (pigments in paintings) or insulating, abrasive, or enamel material. Moreover, zirconia was successfully introduced in the manufacture of cell membranes or oxygen sensors, especially, because it allows the free movement of oxygen ions throw its crystalline structure. In electrochromic devices, ZrO is used as a solid electrolyte.

Due to its low thermal conductivity, zirconia becomes ideal as a protective thermal layer, in jet or diesel engines to allow them to operate at high temperatures. From a thermodynamic point of view, the higher the operating temperature of the engine, the higher its efficiency. Another use due to its low thermal conductivity is as an insulator for ceramic fibers used in ovens. Its hardness makes zirconia suitable for ceramic knives that remain sharper than their equivalent in steel [92].

Other applications of zirconia are in the manufacture of crowns and bridges for dental implants, which will further be covered with porcelain shells.

Zirconia has been proposed for the electrolysis of carbon monoxide with oxygen to produce fuel that can be used for transportation of the surface of Mars.

1.3.2.7. Zirconium silicate

Zirconium silicate, ZrO_2 – SiO_2 composite powder can be prepared by a wet chemical method using zirconium chloride and burnt silicon oxide as raw materials and subsequently sintered by hot pressing to obtain this type of ceramic material [93].

The composite of zirconium dioxide and silicon dioxide comprises advanced physicochemical properties, such as high thermal and chemical stability, high mechanical strength. Due to these advanced properties, it is considered for use in applications of catalytic processes, highly permissive insulated layers, high tear-resistant glass lenses and optical coatings [94].

Zirconium silicate is used in the manufacture of refractories for applications where high corrosion resistance to alkaline materials is required. It is also used in the production of ceramics, enamels. In emails, it is used as an opacifier [95,96]. It can also be present in different types of cement. Another use is in the manufacture of grinding balls. The thin layers of zirconium silicate and hafnium silicate produced by chemical vapor deposition can be used as dielectric materials of the higher category k, replacing silicon dioxide in semiconductors.

This material is also used in medical applications. For example, ZS-9 is a zirconium silicate that has been specially designed for potassium ions that can capture other ions throughout the gastrointestinal tract [97].

1.3.2.8. Mullite

Mullite is a rare silicate mineral of post-clay genesis. It can have two stoichiometric forms $3Al_2O_3$ $2SiO_2$ or $2Al_2O_3$ SiO_2. Unusually, the mulch is not balanced in terms of the cations present. As a result, there are three distorted places of the position of the aluminum in the lattice: two tetrahedral and one octahedral [98].

In terms of suitable characteristics that support this material for industrial applications, mullite has [99]:

- excellent resistance to heat and thermal shock, particularly good creep resistance;
- coefficient of thermal expansion is approximately equal to that of a silicon semiconductor chip;
- excellent thermal stability;
- resistance to chemical attack;
- good oxidation resistance in the furnace atmosphere;
- high abrasion resistance;
- good electrical resistance;
- can be used at temperatures up to 1800 °C in air and 1600 °C in a vacuum.

Most of the product is used in the manufacture of refractory materials that are mainly used in the glass and steel industry [100].

Due to the high properties of refractoriness, creep resistance and thermal shock resistance, the mold is ideal for applications in the steel industry. The main use of molding products is in the manufacture of bricks used in regenerative air preheaters. Many materials used in the steel industry have large quantities of milling in them. Steel poles, spears, reheating furnaces and sliding gates are some examples based on molding with different alumina content. The use of monolithic forms and prefabricated products increases with the decrease in the use of this type of material in bricks.

The glass industry uses refractory materials based on molding on the blocks of burners, supports, bricks as well as on the upper structure of the enclosures in which the glass is melted, as well as on the chamber in which it is drawn. Mullite is used in the glass industry due to the following properties: resistance to thermal shock, chemical attack, heat and creep [101].

Mold-based products do not introduce impurities, in large quantities, into molten glass. This is especially important in the production of glass panels, where Al_2O_3 contamination is not desired.

The next major user of molding is the ceramic industry, most of the furniture elements in the kiln, such as kiln tiles or supporting frames are made of this promising material [102].

The aluminum and petrochemical industries also use molding variants for applications where chemical attack resistance, thermal shock resistance and temperature resistance in load are required.

New molding variants that have control over mechanical and physical properties offer new opportunities for the use of this material.

The good mechanical properties at high temperatures of the high purity mold make it the ideal material for turbine engine components.

Mullite is a prime candidate for the manufacture of windows resistant to infrared transits. Other applications include electronic substrates and protective coatings [103].

1.3.2.9. Steatite

Steatite (also known as soapstone or talc) is a type of metamorphic rock. It is mostly made of magnesium-rich talc minerals. It is produced by dynamo-thermal metamorphism and metasomatism, which takes place in the contact areas of tectonic plates, rock change by heat and pressure with fluid inflow, but without melting [104].

Steatite ceramics are cheap biaxial porcelains with a nominal chemical composition $(MgO)_3$ $(SiO_2)_4$ [35]. In terms of mass, in terms of quantity, steatite consists of about 67% silicon dioxide and 33% magnesium oxide, but may also contain small amounts of other oxides, such as CaO or Al_2O_3. Steatite is mainly used for its dielectric and thermal insulation properties in applications such as tile, bushings, washers and pigments [36].

Steatite possesses both superior electrical and mechanical properties, as well as excellent mechanical machinability.

Today, steatite is most commonly used in architectural applications such as interior surfaces and countertops. There is only one active soapstone mine in North America. The mine is located in central Virginia and is operated by Alberene Soapstone. All architectural soapstone extracted from Brazil, India and Finland is imported into the USA [105].

Welders and processors use steatite as a marker due to its high thermal resistance, which remains visible when heated. Steatite has also been used for many years by tailors, carpenters and other craftsmen as an auxiliary tool for marking points, as its traces are visible and not permanent.

Steatite can be used in the manufacture of molds for casting soft metal objects such as tin or silver. This soft stone is easy to carve and does not degrade on heating. The soapstone surface can be easily removed from the finished object [104].

1.3.2.10. Cordierite

Cordierite (mineral) is a cyclosilicate of aluminum, iron and magnesium. Cordierite is an extremely light material with a specific density of 2.65 g/cm^3, low thermal expansion and high mechanical strength properties [106].

Catalytic converters made of ceramic contain a large proportion of synthetic cordierite. The manufacturing process focuses on the use of the low coefficient of axial thermal expansion of the cordierite. This prevents thermal shock that can lead to cracking when used in the catalytic converter [107].

1.3.2.11. Aluminum nitride

Aluminum nitride (AlN) is a semiconductor material used in ultraviolet optoelectronics, an electrical insulator with excellent thermal conductivity, and also ideal for applications where rapid heat dissipation is required. In addition, due to the coefficient of thermal expansion close to that of silicon and excellent plasma resistance it is used in the manufacture of components in the equipment used for semiconductor processing [108].

Due to the piezoelectric properties of aluminum nitride, it is deposited on silicon wafers and is used in the manufacture of surface acoustic wave sensors. Another application of this is the RF filters used in mobile phones, also called thin-layer acoustic resonators [109].

Aluminum nitride is used in the construction of ultrasonic microprocessors for piezoelectric transducers, which emit and receive an ultrasound that is used to identify airborne particles at distances of up to 1 m.

Metallization methods allow the use of AlN in applications similar to those of alumina or beryllium oxide. AlN can be used as quasi-one-dimensional inorganic nanotubes, which are isoelectric with carbon nanotubes, they have been suggested for use as toxic gas sensors [110].

Aluminum nitride is used in the manufacture of resonators, gyroscopes and microphones due to its piezoelectric properties [108]. It is used in optoelectronics, in the manufacture of dielectric layers from optical storage units, electronic substrates, high thermal conductivity chip holders, in military applications and in the manufacture of crucibles for melting gallium arsenide and steel.

1.3.2.12. Silicon filler

Silicon nitride (Si_3N_4) is chemically inert (can be diluted only with hot H_2SO_4), high melting point, white compound with high hardness. From all the silicon nitrides, Si_3N_4 is the most used form, especially, due to its high thermodynamical stability [84].

- silicon nitride has high breaking strength at high temperatures.

It is used in high temperature resistant structural components of car engines, gas turbines .

- silicon nitride has a low coefficient of thermal expansion and an extremely high resistance to thermal shock.

In general, the main problem with the application of silicon nitride is cost. With its decrease, the demand for production also increased.

In the automotive industry, sintered silicon nitride is mainly used in the manufacture of engine parts. In diesel engines, it is used to build spark plugs that help start engines faster, the combustion chamber to reduce emissions, increase starting speed and reduce noise, and turbochargers to reduce engine delay and emissions. In the case of spark-ignition engines, silicon nitride is used to protect the tilting arm against wear. For turbochargers to reduce inertia and reduce engine delay, and for exhaust control valves to increase acceleration. Approximately 300k sintered silicon nitride turbochargers are manufactured annually [111,112].

Silicon nitride has been used for a long time in high-temperature applications, because of its resistance to severe thermal operating conditions, such as hydrogen-oxygen combustion chambers. However, their main field of industrial application is in the manufacture of abrasive cutting tools. These products were mainly developed due to the high wear resistance, thermal stability and hardness. Also, they are recommended for the machining of cast iron parts, because the previously mentioned characteristics make the silicon nitride tools suitable for hard steel and nickel-based allows cutting at speeds up to 25 times faster than those used with carbide or tungsten tools. The use of Si_3N_4-based cutting tools had significant effects on the production of processing tools. For example, machining gray cast iron surfaces with silicon nitride inserts doubled the cutting speed and increased tool durability by up to 6 times by reducing the cost of processing materials by up to 50% compared to tungsten carbide tools [113,114].

1.3.2.13. Silicon carbide

Silicon carbide (SiC) is a moissanite mineral, industrially used for cutting tools and abrasive materials. SiC particles can be sintered into hard ceramic parts, especially, those that have to withstand high strength and temperatures, such as clutches or braking system components [84]. In electronic applications, SiC is used in the manufacture of radar detectors, light-emitting diodes, or semiconductor electronics that operate at high voltages or temperatures. Because in the natural state it is very rare, different methods have been developed to obtain synthetic moissanite, for example, large crystals can be obtained through the Lely method [115].

- silicon carbide maintains its resistance even at temperatures up to 1400 °C;
- the notable characteristics of this material are extremely high thermal conductivity and electrical semi-conductivity.

Carbon-carbon silicon composites are used in the manufacture of different braking system components, due to their refractoriness. In this type of composite, Si reacts with graphite resulting in SiC reinforced with C fibers. These combinations are used for the manufacture of braking discs suitable for fast racing cars. Also, since tailored porosity and characteristics can be obtained, SiC was the ideal candidate for car exhaustive system particle filters and oil additives [116,117].

1.4. Reinforcement materials

The incorporation of reinforcing materials in a matrix aims to improve its mechanical properties, especially the specific rigidity and strength. The matrix fulfills several functions, among which [118]:

- provides support and alignment for fibers under load;
- stabilizes the fibers against loops and avoids shearing between the fibers.

Although some fiber-reinforced reinforcing materials are also used with a metal or ceramic matrix, it is currently unanimously accepted that the polymer matrix offers the best combination of composite characteristics when there are no temperature restrictions.

The reinforcement of the polymers is made with fibers used as such or in the form of fabrics and braids and with macroscopic and microscopic particles in the form of spheres, flakes, etc.

When choosing the reinforcing material are taken into account: its properties and requirements imposed by the field of use, forming technology and its influence on the physical and mechanical characteristics of the composite, adhesion to the matrix, economic appearance and coefficient of linear expansion to prevent stresses internal composite [41,119].

The reinforcing fibers increase the resistance to various mechanical and thermal stresses, the rigidity of the composite and reduce the creep and moisture absorption.

Particle-shaped reinforcing materials, especially spherical, reduce shrinkage formation and improve the flow characteristics of the resin

Polymer reinforcement is done with continuous (or long) fibers, discontinuous (or short) fibers and monocrystalline filaments (whiskers) [120]. The continuous fibers give the composite material the maximum achievement of the mechanical characteristics. Short fibers give inferior mechanical properties to continuous fibers due to their unfavorable length-to-diameter ratio but have certain processing advantages and the possibility of pneumatic orientation. In the last 5-10 years there has been a permanent concern to increase the efficiency of reinforcement. so that the properties of the short fiber composite are close to those of the continuous fiber-reinforced composite, with at least 90% modulus and 50% tensile strength [121].

The efficiency of the reinforcement with discontinuous fibers depends on several factors: the alignment in the direction of the main force, the uniform distribution of the fibers, the length of reinforcements and the adhesion strength between the fibers and the matrix.

The performance of the fibers depends largely on the propagation of the load from the matrix, through the interface, to the fiber. If the fibers are too short, less than a critical length, then the strength of the fiber cannot be fully realized.

In order to achieve maximum reinforcement efficiency, the fiber length must be longer than the critical length, and the interfacial connection is strong.

The reinforcement of polymers with whiskers ensures the composite material resistance to different mechanical stresses and much higher modules compared to fibers of the same chemical composition. This is explained by the very high length/diameter ratio and by the perfection of the crystalline structure, which minimizes the influence of defects on mechanical strength [122,123].

Quartz, aluminum and calcium carbide fibers are used to obtain high-temperature-resistant composite materials in combination with ceramic matrices and, for certain applications, with heat-resistant polymer matrices. In order to obtain composite materials used in ablative and resistant structures, intended for aerospace structures, quartz and carbon fibers incorporated in phenolic, epoxy matrices are used in particular. polyimide etc. [124]

The reinforcement of the polymers can be carried out with a single type of fiber or by combining two or more different types of fibers, in a common matrix. Therefore, a hybrid composite material is obtained [125].

The manufacture of composites aims to [126]:

- improving the mechanical properties by combining the best characteristics of individual fibers;
- modification of a certain disadvantageous mechanical characteristic, produced by the predominant fibers in the matrix;
- achieving an advantageous performance/cost ratio.

Except for boron and silicon carbide fibers, which are obtained in the form of a single filament of relatively large diameter, the other fibers are obtained in the form of bundles of continuous and fine filaments, which can be used as such, cut to lengths. standardized or processed by textile operations into nonwovens or 2D and 3D structures, unimpregnated or pre-impregnated fabrics and braids [122,127].

The bundles of continuous filaments were coded according to some details of the manufacturing process in tow, roving and yam. The first form, tow corresponds to the bundle of separate and untwisted filaments. Roving is a set of long filaments, parallel to each other, joined together and untwisted .

1.4.1. Fiberglass

Glass has been developed, initially, in industrial applications, as electrical insulation at high temperatures. The combination of glass fibers with polyester resins and, later, with epoxy resins was the first step in the manufacture of composite materials with superior mechanical properties. The composite material with epoxy matrix and fiberglass was used in 1943 to execute the fuselage of a fighter jet [128].

In the years following the war, the manufacture of fiberglass composites experienced a rate of continuous growth. Therefore, methods of obtaining and treating have been developed. Accordingly, the conventional manufacturing methods of fiberglass, that consist of mixing sand with limestone, boric acid and fondants were optimized and automatized by the designing of industrial installation with high production capacity and great control on the manufacturing parameters [129].

Fiberglass is manufactured by three important processes [130]:

- dispersing a stream of molten glass, in the form of fibers, by centrifugation;
- dispersion by blowing with compressed air or hot gases;
- Thracian fibers through dies.

The latter process is usually applied to obtain continuous glass fibers [131].

The glass is melted in a valve-type furnace, provided at the bottom with one or more die plates which normally have 102 or 104 holes. The glass filaments coming out of the holes are quickly cooled by convection and radiation and then clustered with and acquaint. The formed fiber is wound on a drum that rotates at high speed, then is dried. Immediately after obtaining, the glass filaments are subjected to temporary and permanent finishing treatments.

Glass filaments transformed into roving, yam and matte are subjected to a permanent plastic treatment, by treatment with the finishing agent, and containing a film-forming polymer, a lubricant, an antistatic additive and a coupling agent, usually an organo-silane bifunctional. The polymer (polyvinyl alcohol, polyvinyl acetate, epoxy resin, etc.) joins the glass filaments and forms a film that protects them from abrasion destruction. The lubricant, often a vegetable oil, reduces the high coefficient of friction of the fibers wear [132].

The coupling agent ensures the compatibility between the fiber and the matrix. The antistatic additive prevents and reduces surface electrostatic charge.

Glass fibers intended for processing are subjected to a temporary textile treatment, which protects them during textile operations of weaving or weaving, from abrasion wear and which ensures the formation of fabrics without lint and wrinkles. Usually, textile treatments use aqueous solutions of starch, polyvinyl alcohol, or polyvinyl acetate. The protective film is removed by washing or heating before the fabric is used for reinforcement. The fabric has then retreated with the coupling agent [133].

The diameter of the glass filaments is controlled by viscosity and melting temperature. The typical diameter of the glass filaments varies between 0.8 and 19 films. The standardized length of the cut glass fibers is 3.6, 12. 25 or 50 films.

There are several types of glass, differentiated by the oxide composition, which give them different physical and chemical properties.

Glass A is the most common type, being used in the manufacture of jars and for windows. It resists well to acids, but due to the high content of alkaline oxides (14%) it is sensitive to the action of water, and the dielectric characteristics are disadvantaged [134].

E borosilicate glass with low alkalinities combines moisture stability with good electrical insulating properties and high mechanical strength. E-glass fibers are most commonly used to reinforce polymers and to make electrical insulation. However, E glass is susceptible to degradation in strongly alkaline and acidic environments.

ECR glass combines the good electrical characteristics of E glass with the high chemical resistance, especially to acids, of C glass.

The S and R bottles, the American and European versions of a bottle based on aluminum and magnesium silicate, respectively, have high mechanical resistance and excellent moisture behavior. S, S-2 and R glass fibers are used to reinforce polymers for the aerospace and aerospace industries and some military applications. S fiberglass is advantageous when high performance is required, and S-2 fiber, for an advantageous moderate cost/performance ratio [135].

Other types of special products have been manufactured: high modulus M. glass (110-140 GPa), not marketed due to its high toxicity beryllium oxide content, lead oxide glass for protection against ionizing radiation and glass on lithium oxide base, transparent to X-rays [136].

All varieties of glass have a macromolecular structure, with disordered spatial networks. wherein the structural unit is [SiO_4] with the tetrahedral arrangement. In the network, the tetrahedra are joined by vertices, by Si-O bridges.

Although different types of chemical bonds are found in silicate bottles, the covalent bond is predominant, which determines the high values of mechanical strength.

1.4.1.1. Mechanical properties

Some properties of fiberglass, such as tensile strength, modulus of elasticity and chemical resistance are measured directly on an individual filament or bundles of filaments. Other properties, such as density, elastic characteristics, thermal dissipation coefficient and refractive index are usually measured on solid glass specimens (block) [137].

The mechanical strength of glass fibers is higher than that of block glass. It increases with decreasing diameter and fiber length. The values measured on the bundle of fibers (roving) are usually 20-40% lower than those determined on the individual filaments, due to surface defects, introduced during their formation.

In the absence of structural imperfections or deformation mechanisms, the tensile strength of glass fibers is very high (3.45 GPa for E-type glass fibers and 4.6 GPa for S-glass fibers). However, due to the continuity of the silicate network and the elastic nature of the material, cracks propagate easily and the resistance of the composite is sensitive to stress concentrators such as solid particle inclusions or surface cracks [138].

The mechanical properties of glass fibers worsen with increasing temperature. The tensile strength of glass fiber decreases rapidly at temperatures above 250 °C. In this respect, the S and R glass fibers have a more advantageous behavior, the temperature decrease of the resistance being reduced to higher temperatures.

Moisture negatively affects the mechanical strength of solid glass and fiber. The measurement of the resistance of an individual glass filament at the temperature of liquid nitrogen, at which the influence of humidity is minimized, indicates an increase of 50-100% compared to the value measured at room temperature, m air, with a relative humidity of 50%. This is explained by the micro-cracks usually existing on the surface of the glass, which cannot be completely removed in the manufacturing process. Micro-cracks are stress concentration points. When a critical voltage is reached, under constant load and in a humid environment, the microcracks propagate rapidly and lead to rupture. Thus, the fiber breaks after a while under load, and the breaking time is shorter at a higher stress level. The phenomenon is known as static fatigue [73].

1.4.1.2. Chemical properties

All types of glass are subject to a greater or lesser extent to the action of water, including atmospheric humidity. The attack begins by absorbing water at the glass surface followed by ion exchange by diffusion and then by hydrolysis of silicates, which causes the formation of superficial Si-OH groups. If there are also aluminum or boron atoms in the silicate network of the glass, Al-OH and B-OH groups also appear on its surface. The monovalent cations, Na^+ and K^+ easily replace the H^- ions in the Si-OH groups on the glass surface. Ion exchange reactions can change the reactivity of the surface [139].

The amount of water adsorbed on the surface of the bottle depends on its chemical composition. Bottles that do not contain alkaline oxides are more resistant to the action of water than other types.

Fiberglass, especially grades D, E and S, have good electrical insulating properties. Electrical characteristics are affected by temperature and humidity. The relative permittivity, the tangent of the loss angle and the breakthrough voltage depend on the frequency. The penetration stress also depends on the time of application of the voltage and in the case of glass fiber composites, it also depends on the direction of the fibers in

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the laminate and the inhomogeneity of the polymer matrix. The thermal conductivity of the glass depending on the oxide composition varies from 0.55 W/mK, a value corresponding to lead silicate glass (80% lead oxide and 20% silicon dioxide), up to 1.4 W/mK, corresponding to pure silica glass. The E and C bottles have thermal conductivities of 1.3 W/mK and 1.1 V.7 mJC, respectively [140].

Glass fibers E have good behavior at different types of radiation: X and P produce almost no effect, radiation and neutron fluxes cause a decrease of 5-10% of tensile strength, a decrease of less than 1% in density and a slight discoloration of the fibers. Boron-free S and S-2 bottles have excellent radiation performance. However, semi-finished products made of glass fibers are sensitive to radiation, due to the organic binder and the organic finishing agents. Most organic materials are slightly degraded by ionizing radiation [141].

In summary, all the materials that have been presented as being suitable for the manufacturing of composites matrix can, also, be used for the fibers obtaining. Therefore, the above-described properties and characteristics can be considered when evaluating that specific type of reinforcement.

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Chapter 2

Composites Manufacturing and Applications

2.1. Composite manufacturing methods

Following the industrial developing and growing interest in the properties of these class of materials, the designing and manufacturing methods have been significantly improved and extended to a broad range. The main factor, was, clearly, the economic aspects, considering that the obtaining methods of composites are more expensive than those of conventional materials (polymeric, ceramic or metal). Accordingly, the casting methods were pushed to the top.

There are multiple techniques which can be used for the manufacture of MMC, basically, they can be classified depending on the state of the matrix, accordingly, there are four categories: liquid matrix, semi-solid matrix, solid matrix, matrix with fine liquid particles.

These four categories include different customized parameters as can be seen in Figure 2.1.

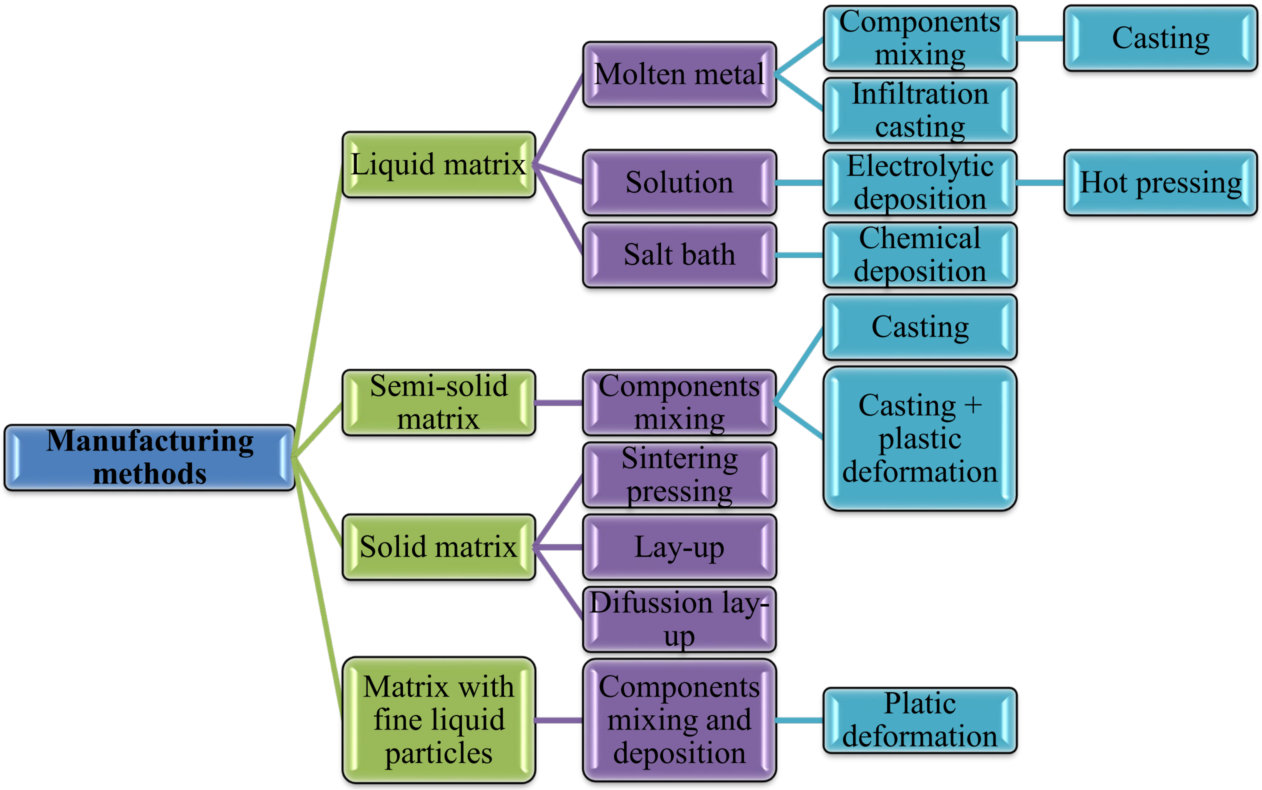


Figure 2.1. Classification of MMC manufacturing methods [1].

Depending on the molding type, the composites manufacturing techniques can be classified as follows [2]:

a) open molding: hand lay-up; spray up and filament winding.

b) closed molding: vacuum bag molding; vacuum infusion; resin transfer molding; compression molding; pultrusion; RRIM; centrifugal casting and continuous lamination.

c) cast polymer molding: gel coated stone molding; stone surface molding and engineered stone molding.

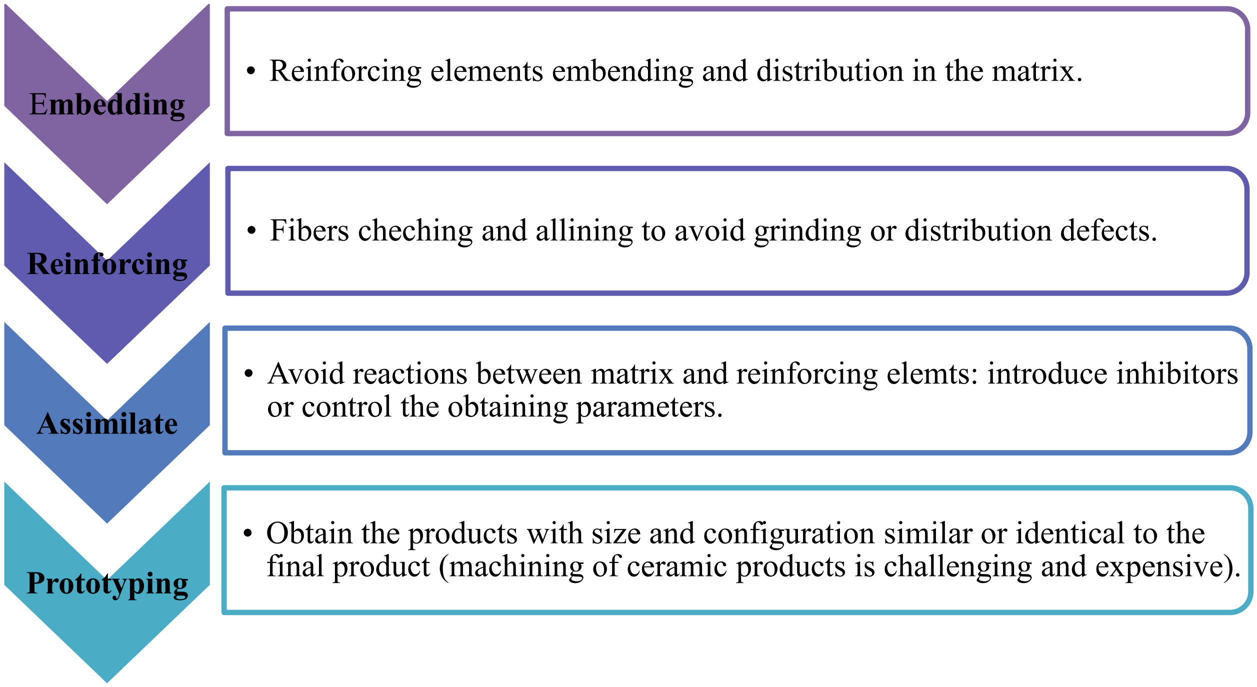
d) additive manufacturing.

In the case of PMC, the manufacturing techniques have been customized depending on the required dimensional and surface qualities. However, even these types of composites can be produced using different techniques or installations. All of them follow ten important steps. During the first step the mixture (the resins and the reinforcing elements) are pulverized or deposited by other methods on the surface of the matrix. Secondly, the deposited layer is compacted by vacuum (autoclave) or different objects. During the third step, in the case of long fiber reinforcement, the impregnated fibers are positioned according to the desired configuration. In the following stage, the mixture is poured in a metallic matrix, under pressure, to assure the obtaining of parts with complex geometries. This method can be replaced by gravitational pouring or injection (the mixture is introduced in the matrix using a piston). However, if these methods aren't suitable for the desired product, in the case of circular parts (tubes, gaskets etc.) the centrifugal infiltration comes handy. Also, for sandwich structures, the lamination technique can be used. While for the products which must possess high compactness and low deviations, the extrusion and stamping are the most suitable methods. The molds used for PMC manufacturing are usually metallic or ceramic. As metals, steel and cast iron are the most used, however, those made of aluminium or coper alloys can be introduced in industrial applications. Accordingly, considering the economic aspects, the molds are usually manufactured through the sintering method. Therefore, the metallic powder is mixed with a binder and poured in a form. Then, the mixture is heated, pressed, while the binder excess is removed. Thirdly, the compact part (mold) is removed and maintained in specific conditions for drying. Following drying, the part is exposed to high temperature for sintering. The sintering stage is followed by the machining, to assure the sizes and desired surface quality. Then the mold can be successfully used for the manufacturing or thousands of PMC composites.

The manufacturing of CMC is much more versatile, because the mixture obtaining usually occurs at room temperature. However, this topic is also challenging, as the compositions involving long fibers are hard to obtain at the desired characteristics, while the main issue of those with short fibers is related with the homogeneity of the mixture. Therefore, this type of composites, usually, exhibit properties anisotropy (the properties are different in different sections of the sample).

In the case of the aluminum alloy matrix, it is reinforced with abrasive type ceramic powders or short alumina fibers. Their manufacture is performed by infiltrating the reinforcement with the liquid matrix, in vacuum or by mixing. in the case of the zinc alloy matrix, it is reinforced with abrasive silicon carbide powders, short aluminum fibers, etc.

The technological method of vacuum infiltration is performed in an installation in which the zinc alloy is melted in an electric furnace with crucible located in a closed enclosure in relation to the external environment. The reinforcement elements, either in the form of particles or preformed from fibers, is inserted into an electrically heated mold. When inert chamber is sent under inert gas under pressure and in the mold a vacuum is created with the pump, the alloy moves through the connecting tube, enters the mold and infiltrates into the armature, which absorbs spontaneously. The absorption process depends on the degree of watering of the reinforcement, the liquid alloy and is ensured by the pressure exerted on the liquid zinc alloy.



Embedding
- Reinforcing elements embending and distribution in the matrix.

Reinforcing
- Fibers cheching and allining to avoid grinding or distribution defects.

Assimilate
- Avoid reactions between matrix and reinforcing elemts: introduce inhibitors or control the obtaining parameters.

Prototyping
- Obtain the products with size and configuration similar or identical to the final product (machining of ceramic products is challenging and expensive).

Figure 2.2. Manufacturing steps of CMC [3].

The infiltration method allows a homogeneous infiltration of silicon carbide powders or preforms of short aluminum fibers.

The mechanical mixing method followed by die casting is used when the reinforcement is a silicon carbide powder which is introduced into the liquid zinc alloy [4].

The mixing takes place in an electric oven with the crucible under the action of a mechanical stirrer with propeller. The furnace is located in an enclosure in which an inert gas is introduced under pressure, in which case the liquid zinc alloy moves through the connecting tube in the mold.

The liquid matrix composite fills the cavity of the mold after which, by cooling, it solidifies, resulting in the desired product. In order to stimulate the wetting process, various additional solutions are used by energetic mechanical agitation accompanied by the reinforcement of the alloy in the semi-solid phase or by the variation of the solid-liquid surface tension coefficient with the fiber coating or the modification of the matrix composition [5,6].

The methods and technological procedures for making finished products from composite materials are chosen depending on: the nature of the material of the matrix and the reinforcement; material properties; reinforcement geometry, requirements required for each product in practice [7].

Given that the most widely used matrix is that of polymers, the technological methods and processes applied in this case are representative.

The succession of technological operations knows common elements, but also particularities from one case to another.

Thus, in the case of the polymer matrix, the following technologies can be applied to make the composite material separately or simultaneously with the realization of the finished product:

The manual forming technology by spraying includes the operations preparation of the mold surface; alternative application of layers of polymer and reinforcement material or shredded reinforcement mixture, matrix polymer, catalyst and accelerator, heating polymerization; extraction of the product from the mold; finishing the product.

The elastic membrane forming technology comprises the operations: (i) preparation of the mold; (ii) pre-impregnating the reinforcement with the matrix polymer, (iii) placing the pre-impregnated reinforcement in the mold (several superimposed layers) or applying the polymer, (iv) pressing 0.05 - 0.35 MPa by means of the elastic membrane under the action of pressurized air; (v) final hardening heat treatment; (vi) extraction of the part from the mold; (vii) finishing and final control of the product [8].

The winding forming technology comprises the following four operations: (i) execution of the product support model; (ii) distribution, by continuous winding on the support model of the reinforcement (thread, tape, fabric) usually impregnated with the appropriate resin; (iii) treatment by polymerization; (iv) finishing and control processing [9].

The injection molding technology includes four steps: (i) preparation of the mold, placement of the reinforcement in the lower half-mold; (ii) closing the mold; (iii) injecting the resin - catalyst - accelerator mixture; (iv) final strengthening treatment [10].

The injection methods/technologies can be divided in three categories, as shown in Figure 2.3. For each method, the technological parameters, such as time, temperature, pressure and injection velocity are different, depending on the constituents of the composite.

Die-forming technology comprises the following operations: (i) preparation of the mold, placement of the composite material in the mold; (ii) punching at 4-30 MPa, depending on the type and volume of the material; (iii) polymerization with heating; (iv) evacuation of the part; (v) finishing and final control [11].

The extrusion forming technology starts with the preparation of the supply chain, followed by loading with the composite mixture (matrix, reinforcement, adjuvants) in the heated mixing chamber. Then, the plasticization of the mixture step occurs, followed by continuous pressing with screws, thermal treatment and cutting lengthwise. Then, the finishing and final heating control occurs, followed by extraction of the product from the mold and finishing of the product [12].

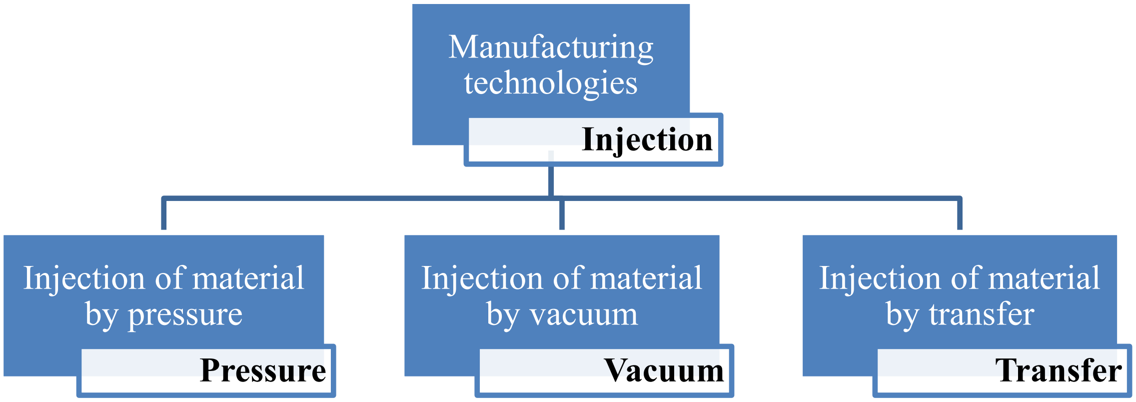


Figure 2.3. Classification of injection technologies.

2.1.1. Pre-impregnation manufacturing

The fiber reinforced composites manufacturing by pre-impregnation (pre-preg) method can be used as parts with controlled fiber to matrix ratio. Also, this manufacturing method is used in sandwich structure obtaining, generally, for the manufacturing of composites with parts that develop high bending strength at low weight. [13]

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Moreover, this method is mainly suitable for the manufacturing of prototypes and low series products, since it can be easily realized manually. Therefore, where parts with complex geometry that must be cast-in-place can be developed through this method.

However, due to the high industrial interest offered by these materials, especially, where high resistance panels with low thermal conductivity is requires or different types of structure that must offer anisotropic characteristics, automatic methods for sandwich composites have been developed. Figure 2.4 shows a schematic representation of industrial manufacturing of these types of composites. As can be seen, the lower layer is stretched out between multiple rolls, over it an adhesion addition is poured, such as a resin, followed by the reinforcement or middle layer applying. If the reinforcements are applied as a continuous layer, then the cutter is removed, if not, then the cutter is used to cut the middle layer in small pieces, depending on the desired characteristics. Over the reinforcement, a new layer is applied (upper layer). The upper layer, usually, has the same characteristics with the lower layer. Further, the composite is pressed between multiple rolls to eliminate the resin excess and air bubbles.

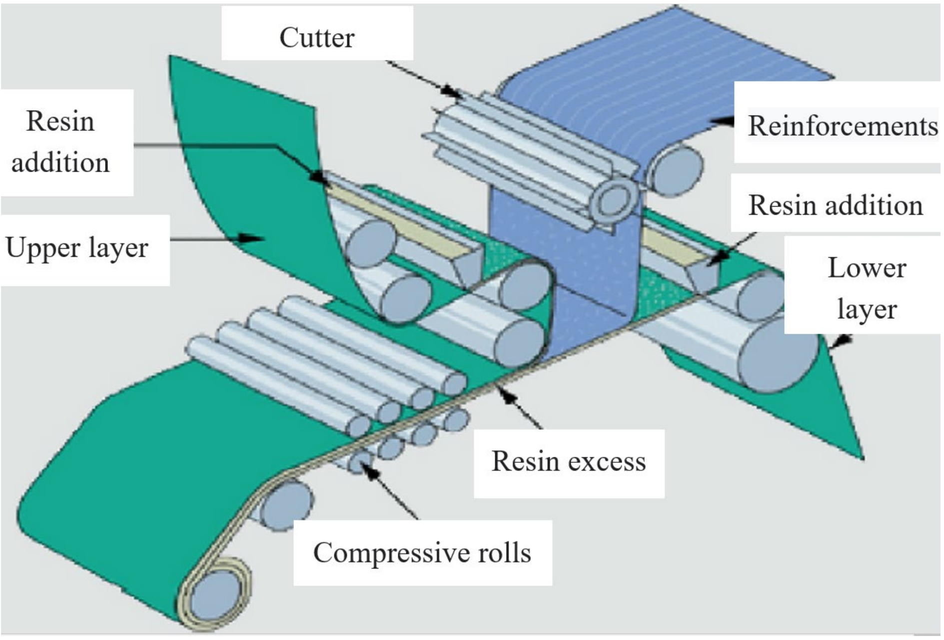

Figure 2.4. Schematic representation of automatized methods for sandwich composites manufacturing [14].

The biggest advantage of this method is related with high its high versatility and productivity as the number of layers can be increased and the type of materials used are only limited by their elasticity.

2.1.2. Compressive pouring

The compressive pouring is one of the main methods that can be used for the manufacturing of mass production parts. This method consists of pushing the mixtures of fibers and matrix into a matrix (Figure 2.5). Usually, the matrix is preheated, in order to avoid thermal shocks or too fast setting.

This method has also high productivity, however, the costs of the final products are significantly influenced by the costs of the matrix. Therefore, from economic point of view, it can only be considered for mass production.

However, to avoid the costs associated with the conventional method, a simplified equipment has been developed. Figure 2.6. shoes the schematic representation of this simplified method. As can be seen, over the die (a part with the size and geometry of the final product) the reinforcements are sputtered after going through the chopper, which assure the desired length of this components. The resin matrix is sputtered through two feeders, one being positioned above the reinforcement particles and the other one below them. Accordingly, when the particles go from the chopper to the die, they will be fully covered in resin. Further, the compactness of the product assured by the pressing role, this component will assure a constant layer and the elimination of air bubbles [9,15].

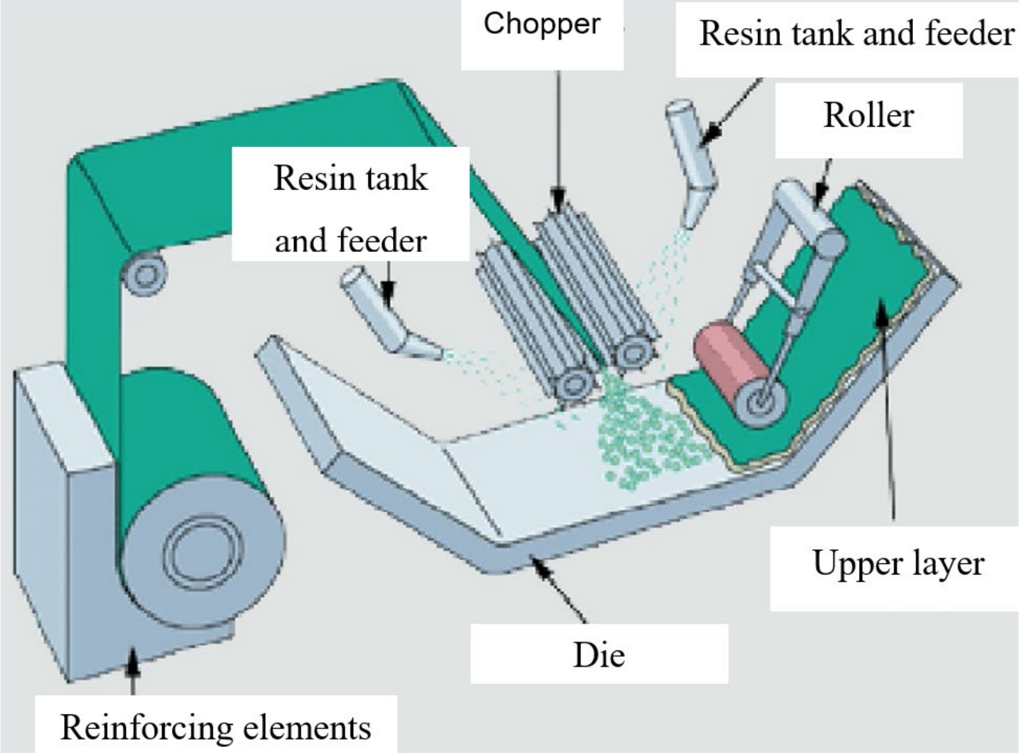

Figure 2.5. Schematic representation of composites manufacturing through compressive method [14].

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2.1.3. Vacuum bagging manufacturing method

It is achieved by inserting the resin and reinforcing it on an open mold with the help of a vacuum pressure. This pressure acts on the layers by pushing the resin, ensuring a fast and complete impregnation of the industrial fabrics. As can be seen from Figure 2.7 the composite is positioned on a porous layer, which can be easily peeled after composite setting and assure a clear passing of the resin excess to the absorbent material that will capture the unnecessary amount of resin. The resin excess is removed from the composite due to the vacuum pressure, that will squeeze the layers together through the vacuum bag, which will easily copy the form of the composite, while the composite is positioned on a component with the desired shape of the final product (Figure 2.8) [9].

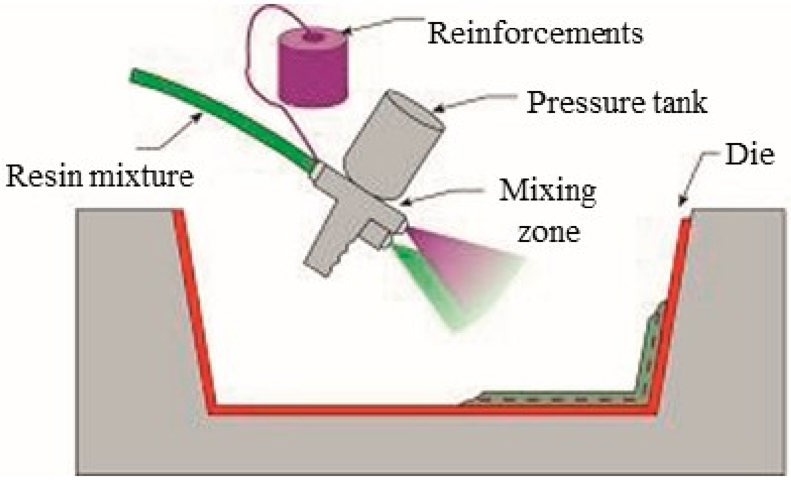

Figure 2.6. Simplified method of composites manufacturing through sputtering method (without roller) [16].

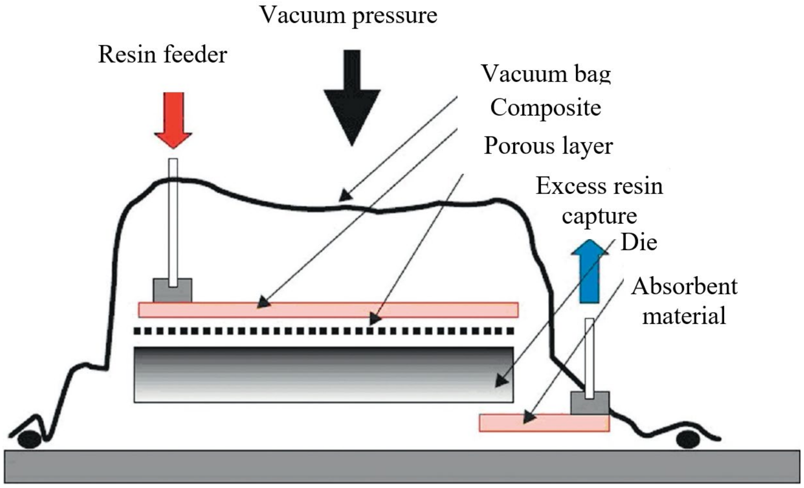

Figure 2.7. Schematic representation of composites manufacturing by vacuum infusion method [16].

The method assures the obtaining of quality products with uniform layer thickness. Yet, the process is high energy consuming and requires long setting time at high temperatures (depending on the resin type, usually, temperatures close to 100 °C).

2.1.4. Resin transfer molding method

This composite manufacturing method is suitable for high to medium volume production, depending on the setting time. In this method the polymer matrix is injected, with a low-pressure pump, into the die until it is full. The obtained products can have complex configurations and quality comparable with those obtained through infusion [17].

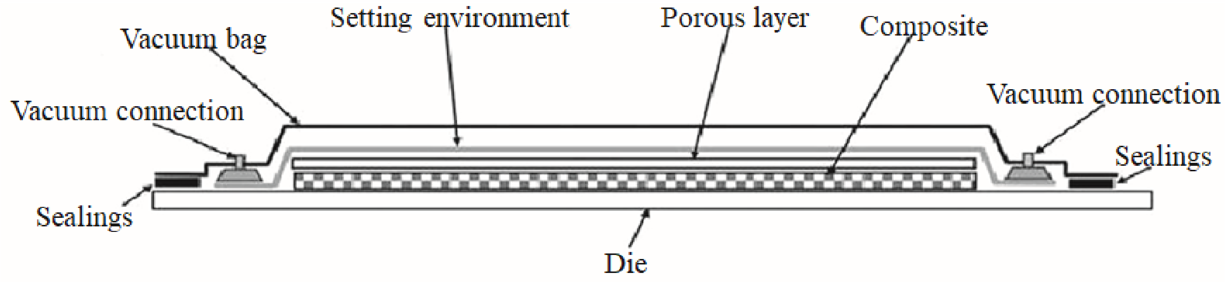

Figure 2.8. Simplified representation of vacuum infusion set-up.

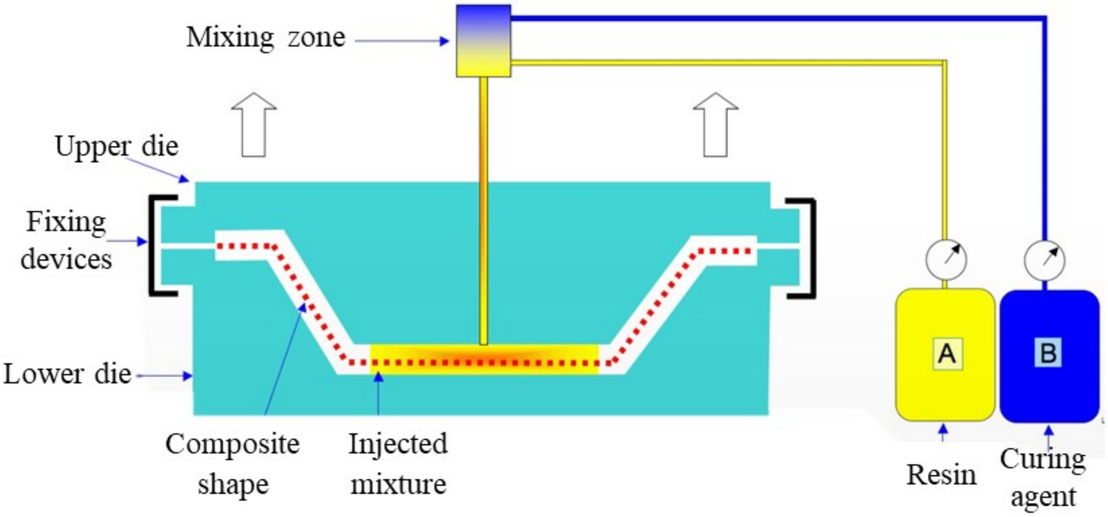

Figure 2.9. Schematic representation of composite manufacturing through resin transfer molding method [18].

As can be seen from Figure 2.9, the A and B tanks, containing the resin or polymer and the properly mixing agent are maintained under pressure, until the wright amount of each is released into the mixing zone. Further, the mixture is injected into the cavity between the upper and lower die (molds) copying the shape of the final product. The upper and lower die are heated to the established setting time. After the composite is cured, the fixing devices and upper die are removed, followed by the composite extraction.

There is also another type of compression casting, which is cold pressing casting. It is called cold press casting because it does not require heat during the process. The process is usually done at room temperature using a hydraulic or mechanical press. The final products will have two finishing surfaces where it is necessary to cut after hardening, because it has sharp edges.

2.1.5. Filament winding manufacturing method

Filament winding is the most common method of producing parts with axial symmetry. The filament winding is a conventional step used to combine 2D reinforcing with 3D reinforcing for different type of matrices. This method is mainly used for circular parts (tubes, bushings etc.) manufacturing that require fiber reinforcing. The process (Figure 2.10) consists of fibers twisting and pulling through the resin bath before winding over the open die which has the external diameter equal to the internal diameter of the final product [9].

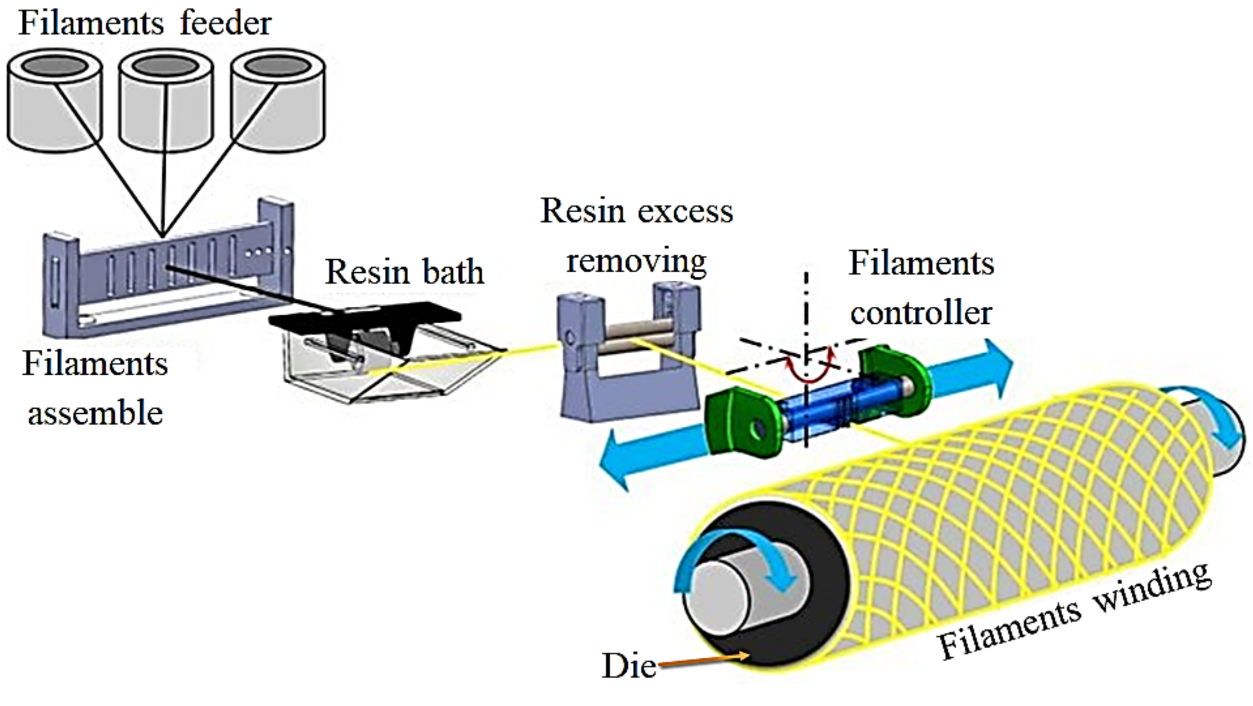

Figure 2.10. Schematic representation of filament winding manufacturing method [19].

The transverse displacement head or the filaments controller will continuously adjust the filaments position to assure a uniform distribution over the die, and to assure the desired 3D structure.

2.1.6. Centrifugal casting manufacturing method

Similar to conventional centrifugal casting of alloys, this method consists of introducing the mixture or the separate components of the composites into a continuously rotating die (Figure 2.11). Due to centrifugal force, the components will mix together depending on their specific weight and the tubular part will be cured at specific temperature, by heating the die [20].

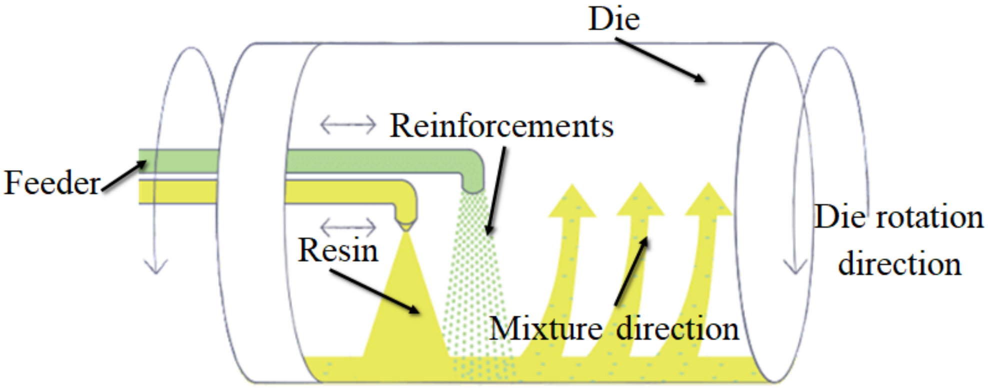

Figure 2.11. Schematic representation of composites manufacturing by centrifugal method [21].

Due to the rotation of the die, the feeders are moving from one end to another of the die, to assure a uniform longitudinal distribution of the mixture.

2.1.7. Automatic Fiber Positioning (AFP) and Automatic Laying Tape (ATL)

ATL is an advanced method of composites manufacturing consisting of compacting and heating of prepreg fibers (Figure 2.12).

The method consists in depositing the composite layer by layer while heating the contact zone and applying a specific pressure. The main advantage of this method is the degree of automatization, since all the operations can be fully automated. Therefore, the quality of the products can be assured even in the case of mass production. Moreover, the method can also be used for the manufacture of composites with woven fibers, however, the weaving complexity will significantly influence the process cycle time [23].

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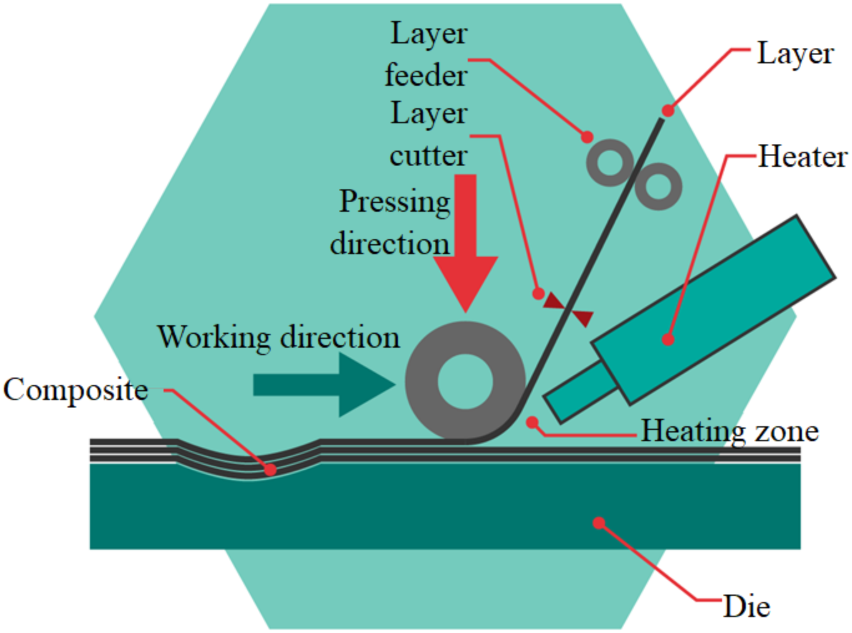

Figure 2.12. Schematic representation of ATL method [22].

2.1.8. Pultrusion manufacturing method

Pultrusion is a combination of pulling and extrusion, therefore, the method consists of pulling the prepreg fibers through a resin bath and binding them together in unidirectional or multidirectional nets. Further, the woven composite passes through a die, maintained at curing temperature, and goes to the cutting area. The resulting materials are continuous composites with tubular or bars shape (Figure 2.13).

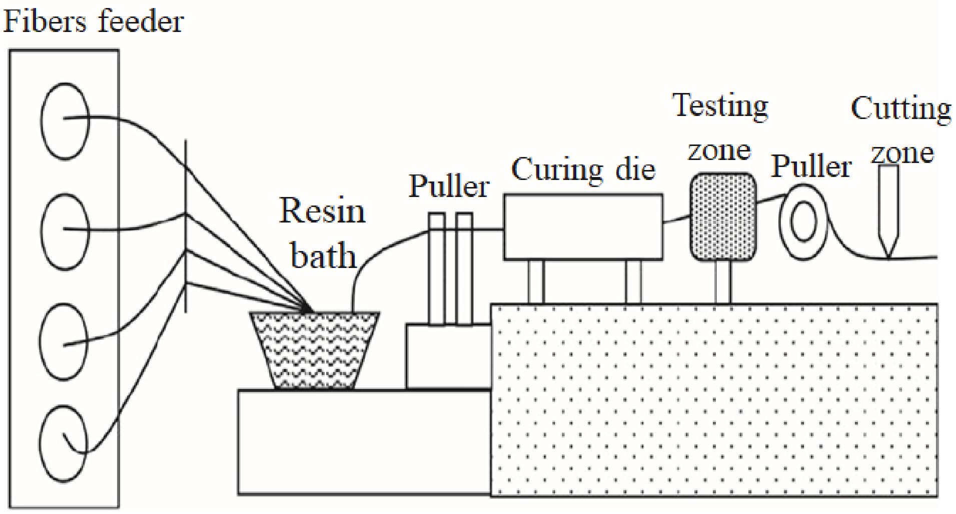

Figure 2.13. Schematic representation of pultrusion manufacturing method [24].

The main advantage of this method is the high productivity; however, it is limited to products with simple shape (panels, bars or tubes).

This section provides detailed information on composite manufacturing processes for aerospace applications. Accordingly, it can be concluded that there is a wide area of research and development in the field of composite manufacturing processes. Improving the manufacturing process produces a better quality of the composite. However, great care must be taken to choose the best manufacturing process for any type of composite, whether natural fibers or synthetic fiber composites are used. Theoretically, a process that can be used with high volume of fibers will produce composites with better strength. Moreover, it must be considered the fact that the manufacturing process affects the properties of the composite produced, as the process is also related to fiber size, fiber orientation, fiber direction and length, and also the type of matrix used during the process.

2.2. Properties of composite materials

In the general case of any material, we must be sure that the material will not break (so it has adequate mechanical strength) and that it will not deform excessively under a certain load force (so it has a certain rigidity) most important mechanical properties of composite materials.

Although they have been the focus of researchers' attention to the possibilities of applying polymeric composites, other properties that can be included in the wide range of physical and chemical properties are of great interest. Thus, in many cases, fillers are incorporated into polymers in order to modify and improve both physical and chemical properties or to reduce the cost of the final material. For example, good conductive fillers, such as carbon, are often added to perfectly insulating substances, which tend to accumulate static electrical charges, which in some cases can have disastrous consequences. A particular case of carbon-filled plastics is vinyl polychloride used to make belts in coal mine conveyors [20].

Pigments are used to improve the appearance and to reflect the sun's rays, which can cause chemical degradation of the polymer. The use of a material for a single purpose can often lead to the deterioration of other properties. Thus, compromises must be allowed regarding the nature of the filler, its concentration and its interaction with the polymer matrix. disadvantages in other directions.

Among the properties of fiz.ee. the most important are thermal, electrical, optical, magnetic and acoustic.

Among the chemical properties of particular importance is the resistance to attack by chemical agents including water and O_2. as well as chemical changes that may occur under the application of mechanical stresses or electric fields.

- The most important thermal properties are: thermal expansion, thermal conductivity, specific heat, softening and melting, thermal degradation and related phenomena.

Thermal expansion of composite materials is influenced by several factors: the nature of the composites, the possibility of microcracks, the nature of the bond to the interface, the voids in the case of expanded polymer matrices, the shape and orientation of the filler, the thermal transitions that may occur in the polymer. A thermal transition will lead to a change in the coefficient of thermal expansion, and a possible orientation inside the matrix or filler will lead to different coefficients of thermal expansion in the two orthogonal directions. By reinforcement, the coefficient of thermal expansion of the polymer matrix decreases greatly, and can even reach negative values in the case of a laminate based on phenolic resins reinforced with asbestos fibers [23].

Thermal conductivity. There is a growing interest in the effect of fillers on the thermal conductivity of polymers, both in terms of optimizing the heat introduced in the manufacturing process of thermoplastic and thermosetting polymers, and in increasing the thermal conductivity coefficients for certain applications. Polymers have low thermal conductivity compared to metals and most inorganic materials, but by incorporating metal or carbon filaments a substantial increase is obtained [1].

In general, particle size does not influence thermal conductivity. in the case of an epoxy resin filled with copper, this is true at ambient temperature, but at lower temperatures, the higher contact resistance of small particles reduces the conductivity. The specific surface area is an important factor, for example copper and aluminum metal powders, by oxidation. lead to increased conductivity.

In connection with the thermal conductivity property of composites, mention may be made of their use as insulators and supports for superconducting magnets, as well as for non-metallic vessels of complex shapes.

Thermal transitions. The two most important thermal transitions refer to the melting point and the glass transition temperature. In connection with these and dependent on them are the softening points, which represent the temperatures at which the polymer undergoes arbitrary deformations for a given charge, under specific experimental conditions. in practice, the softening point reflects the influence of the matrix.

The melting point for polyamides and polypropylene is not affected by the presence of conventional fillers, as the crystalline material will remain unchanged provided that the filler does not affect the crystalline order. However, if the filler can act as a nucleating agent for crystallization (or, in rarer cases, will an antinuclear agent), then, by changing the size of the crystallites, it can influence not only the melting behavior, but also the mechanical properties. which depend directly on morphology through a subsequent effect on the size of the spherulites [25].

The glass transition temperature is less sensitive to the presence of the filler than the thermal deformation temperature.

• Electrical properties. The most important electrical properties are: conductivity, dielectric strength and permittivity.

Due to the low level of electrical conductivity, for most composite systems it is difficult to obtain data in this regard. For this reason, the exact mechanism of conductivity in composites is not well explained, as very small changes in structure can have considerable effects on conductivity. Thus, a good conductive impurity, at several parts per billion, can theoretically increase the conductivity of the composite material by several orders of magnitude [26].

With regard to electrical permittivity, it has been established that it is influenced by both temperature and frequency. The most important effect of adding a filler is that of producing an interfacial polarization at the interface, which depends on the ratio between the permittivity's of the components. In general, mineral and metal fillers tend to increase the overall permittivity, while organic agents decrease it, but in both cases, the tendency of the water adsorbed by the filler will lead to changes in the response values.

• *Optical properties.* The use of fillers has an indirect or direct effect (in the case of pigments) on the optical properties of polymers, ranging from complete opacity to certain degrees of transparency. For polymers such as: polymethylmethacrylate, polystyrene. polycarbonates and polyvinyl butyral (which are capable of transmitting up to 98% of visible light), the use of fillers should be done very carefully because they lead to a decrease in the transmission, although for aesthetic reasons, on roofs or covers, for example, a certain percentage of emissivity may be an advantage. Light reflection in polymers can also occur due to other causes, such as: ruptures, cracks, etc. in the polymer network for composite materials, in addition to light scattering sources derived from polymers, other sources may also be involved which relate to any irregularity which the filler may introduce on the surface [27,28].

• *Magnetic properties.* Although polymers have well-defined electrical and other physical properties, they do not have an intrinsic magnetic character, but magnetic fillers such as ferrite can be added to them. For this purpose, polymers such as nylon, polyethylene, epoxy resins, polypropylene and polyisoprene are used. In this way, flexible or rigid, lightweight magnetic components are obtained, which can be used in fields such as: television, communications, storage of computer applications, the automotive industry, etc. [29]

• *Acoustic properties.* Solid polymers have a high acoustic impedance compared to air, thus being effective sound reflectors. Due to the mechanical nature of the energy at a given temperature, the polymeric component of a mixture can suffers a large mechanical loss for the frequency considered, which leads to a sound absorption. Thus, polymers that have a low glass transition temperature are good sound absorbers at room temperature. A recently discovered advantage of composite materials, compared to conventional ones is that of reinforced polymers from which water pipes can be made, in which the flow noise is much lower, the tendencies of cracking and water blows being much quieter [30].

• *Friction and wear of composite materials.* Composite materials can be used either in areas where a low coefficient is required (wear objects, gears) or in those with a high coefficient (brakes, clutches, etc.), while tires are an intermediate case, but very important. low wear characteristics are generally required, unless high wear is tolerated due to the requirement of a high coefficient of friction, in a single use application [31].

• *Effects of the environment on composite materials.* Organic matrix composites, subjected to the long action of the environment, degrade, a process known as aging. The main factors that, individually or cumulatively, lead to material degradation are: temperature, mechanical stress, chemical exposure and solar radiation. Other interventions may include: ionizing radiation, exposure to electric or magnetic fields, high vacuum, impact with meteorites or other objects, the action of microorganisms, etc. [32].

In the polymer matrices, chemical destructive reactions occur, which lead to the breaking of the bonds between the main chains or to the elimination of the substituents and to the decrease of the crosslinking density, as a result of the breaking of the transverse bridges between the chains. Simultaneously, the reinforcement materials are also destroyed to a greater or lesser extent.

The action of the environment on the composite material is manifested by the appearance of chemical changes in the microstructure of the resin, by the worsening of the physical-mechanical properties, the formation of cracks, the dimensional change, color and texture of the composite.

The degree of damage depends on the exposure time, the level of stress and the susceptibility of the composite to any of the environmental factors, which in turn depends on the type of matrix and fiber.

The response of the composite material to chemical agents depends in particular on the polymeric matrix, while the change in tensile strength and modulus depend mainly on the reinforcing material. Both depend on the strength of the interfacial link.

2.3. Applications of composite materials

Composite applications currently cover a wide range of fields, such as aeronautics, space flight, shipbuilding, electronics, radiolocation, automotive construction, industrial equipment, light industry, industrial and civil engineering, building protection against the effects of earthquakes, sports etc.

2.3.1. Aeronautical industry

The composite materials used in the aeronautical industry are of the type reinforced with fibers or filaments coated in a polymer matrix. The most common reinforcing fibers used are carbon, aramid and glass, used separately or in hybrid combinations. The polymeric materials used are generally epoxy resins, which require operating temperatures between 120 and 175 ° C [7,33].

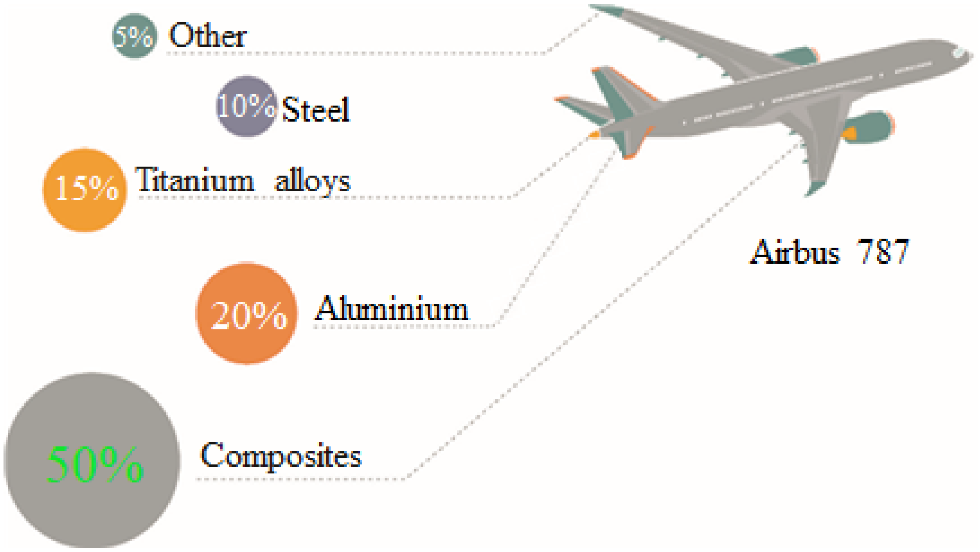

Figure 2.14. Composites application in planes manufacturing [34].

2.3.2. Applications at high temperatures

High temperature resistant polymers are used in aerospace, electrical and electrical industries, as well as in other fields that require suitable mechanical properties for high temperature operation (Figure 2.15). The vast majority of polymers used in these fields are in the form of: adhesive liners, fibers, films, foams, laminated insulation and powders.

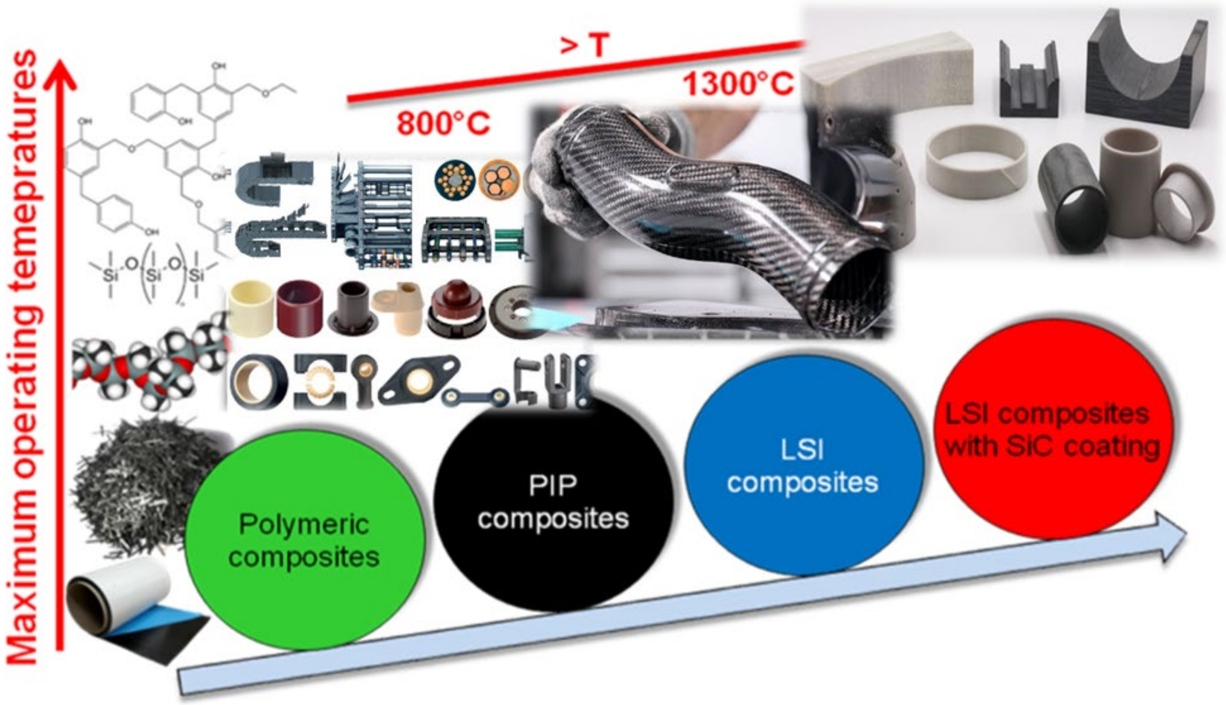

Figure 2.15. High temperature application of composites [35].

Where LSI means Liquid Silicon Infiltrate and PIP means Polymer Infiltration and Pyrolysis.

2.3.3. Missile aerospace systems

The use of composite materials in the primary structures of spacecraft has resulted from their successful use in the medium and long range missile technique. The first application of composite materials in the missile technique was the engine casing of the final propulsion stage of Vanguard projectiles, used in space exploration. The engine of the Altair X248 rocket was the first of a long writing that used composite materials such as epoxy resins reinforced with glass fibers (Figure 2.16).

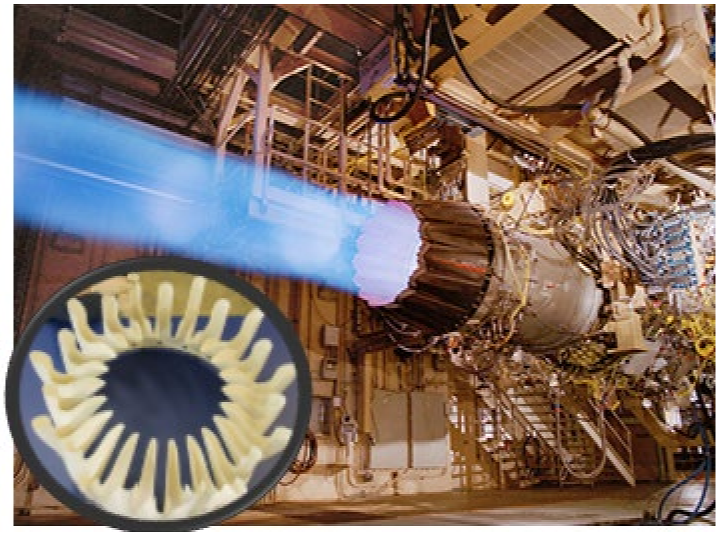

Figure 2.16. Application of composites in missile or rockets manufacturing [36].

Spatial structures must, as a rule, meet the following conditions: they must be light, durable, have a low coefficient of expansion and dimensional stability throughout their working life. The main components used in aerospace technology can be grouped into the following categories: structures, platforms, pressure vessels and tanks, housings [37].

2.3.4. Marine applications

Fiberglass-reinforced polymer matrices are used for the projection of the submersible part of ships, its surface being exposed to high pressure shocks of salt water. The choice of polymorphic matrix and reinforcing mesh is of paramount importance and has been the subject of a solution study that took into account steel, rubber and plastics reinforced with fiberglass, concluding that the latter composite material is preferable to traditional ones. The replacement of metal buoys with those made of composite materials was necessary due to the frequent damage to the vessels in case of collision with them. Polymer matrix composite materials such as polyethylene foams, polyurethane elastomers and glass fiber reinforced plastics were successively tested [38,39].

Boat builders for tourism purposes in Europe used polyvinyl chloride foams reinforced with fiberglass and polyester applied over the wooden skeleton of the boats.

2.3.5. Protection of buildings against the effects of earthquakes

Attempts to protect buildings against earthquakes are intended to reduce their disastrous effects. Ten years of intense research in this field have led to the idea that "foundation insulation" from the rest of the construction, through bearings made of rubber-based composite materials can reduce the effects of earthquake shocks on civil and industrial construction. Of course, the "insulation of the function" is not a miraculous solution in itself, it will be corroborated with soil conditions, geological and economic conditions and adequate education of the inhabitants [40].

2.3.6. Construction of machines and commercial applications

Elastomeric composites are widely used in the construction of cars and motor vehicles due to their low weight and elastic and strength properties, at least comparable to those of traditional materials [41].

2.3.7. Flexible gaskets

Flexible gaskets are generally designed in overseas layers, consisting of rubber and metal plates, constituting a composite structure that withstands the deformation loads resulting from the action of compressive forces. Such sandwich laminates have proven real advantages over conventional gaskets made of rubber with different properties [42].

2.3.8. Rubber laminates and non-metallic plates

The use of supports made of rubber and non-metallic plates comes to eliminate most of the difficulties that metal supports present. Thus, fiberglass and aramid fiber plates, Kevlar, Du Pont, incorporated in thermoplastic matrices were studied. Prefabricated fiberglass boards were the first to be used for this purpose, together with rubbers polychloroprene. Aramid fiber stiffened composites seem to be the most recommended for making non-metallic supports [43].

Composites reinforced with glass fibers aligned parallel to each other have maximum mechanical strength and rigidity in the direction of alignment. If the fiber distribution varies between 0° and 90°. the mechanical strength varies proportionally.

The bidirectional fabrics, taking angles of 0 ° and 90 °, contribute to the mechanical strength of the composite accordingly, with higher values in the direction of parallel alignment. If the distribution of fibers in the warp and weft direction is equal, the properties of the laminate are comparable in these directions (but not necessarily equal).

In unidirectional fabrics, glass fibers are distributed. mostly. in the direction of the warp, and the amount of embedded weft is minimal. in composites reinforced with unidirectional fabrics, the maximum mechanical strength corresponds directly to the highest concentration of fibers.

Reinforcement with fibers statistically distributed in the matrix, such as. in the case of felted materials. with cut fibers, gives the composite relatively uniform mechanical strength in all directions (approximately isotropic characteristics), but smaller.

Glass fiber-reinforced thermosetting polymers are also used in combination with other materials, such as: metal, wood, cellular polymers, resin-impregnated paper. in sandwich structures.

The faces of the sandwich structure can be metallic materials (aluminum alloys or stainless steel) and non-metallic materials (polymeric laminates with glass fibers or carbon fibers), for this purpose cut fiberglass felt, woven roving or unidirectional laminates are used. made of glass fibers, pre-impregnated with polyester, phenolic, epoxy, polyimide resins, etc. A common structure consists of a laminate with cross-layers, with the folds oriented at 0 ° and 90° to the fixed axis and a wood core or cellular polymer [44].

Sandwich materials with fiberglass-reinforced thermosetting polymers are widely used for applications where the main requirement is bending. An ideal sandwich material has thin, rigid, high-strength faces, a low-density core and a low cost price.

For special applications in the field of aircraft construction, for helicopters and spacecraft or in military equipment, where the strength / weight ratio is a priority, honeycomb core sandwich materials are used. In these materials, the core can be made of paper pre-impregnated with resin and especially of aluminum alloys or polymers reinforced with glass fibers.

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Chapter 3

Experimental Investigations in Ceramics Improvement

Further an experimental study conducted on the improvement of compressive strength and hardness of alumina based ceramic material is presented.

3.1. Materials and methods

In this study multiple types of ceramic composites have been developed using alumina, silica and nano-clay as raw materials. The main component of the developed ceramics was aluminium oxide, while silica, magnesia and nanoclay have been used as additions to increase the mechanical properties of the control sample.

3.1.1. Alumina

T60 / T64 alumina is a pure sintered alumina α material that has been densified by rapid sintering without the use of sintering additives at temperatures above 1800 °C. It is in the form of large, hexagonal crystals with lengths of up to 200 µm. Due to its microstructure with low open porosity and large crystals with closed spherical pores trapped during recrystallization, alumina T60 / 64 possesses excellent thermal stability and high resistance to thermal shock. The studied alumina has very high refractoriness, high mechanical strength and abrasion resistance, very good chemical purity, excellent dielectric properties and a very good resistance against alkaline and acid corrosion.

Alumina is the ideal material for the manufacture of highly refractory unshaped parts and is used in a variety of industries such as: steel, foundry, cement, petrochemical, ceramics and waste incineration. Other common applications include its use in electrical insulators, furnace components and as a support for catalysts. Alumina is an excellent product that can be used as a filler in epoxy systems or resins where high dielectric strength, good thermal conductivity or abrasion resistance are desired (Table 3.1) [1].

Properties of Almatis Alumina [1]:

- High refractoriness
- Good thermomechanical properties
- Volumetric stability
- High density, low permeability

- High resistance to thermal shock
- High chemical resistance
- High purity
- High mechanical stability
- High wear resistance

Table 3.1. The characteristics of T60 / T64 alumina [2].

Oxide composition, % wt.	**Particle dimension**	**-45 µ LI**	**-45 µ STD**	**-20 µ**
Al_2O_3 (ordinary)	99.5	99.5	99.1	99.3
Na_2O	≤ 0.40	≤ 0.40	≤ 0.60	≤ 0.40
SiO_2	≤ 0.09	≤ 0.09	≤ 012	≤ 0.15
Fe magnetic	≤ 0.02	≤ 0.02	≤ 0.30	≤ 0.02
Physical properties				
Raw density [g/cm^3]	≥ 3.50			
Aparent porosity [%]	≤ 5			
Water absorption [%]	≤ 1.5			

All data are based on test methods according to Almatis standards.
1) All sizes except - 45 microns LI, - 45 microns STD and - 20 microns.

3.1.2. Silica

Silicon dioxide is the most important component of glass and silicates, the most representative form of which is quartz. Silicon dioxide is practically insoluble in water or acids except hydrofluoric acid (HF) reaction in which silicon tetrafluoride (gas) having the formula (SiF_4) is released. And especially the amorphous form of silicon dioxide dissolves in alkalis (bases).

3.1.3. Magnesia

The magnesium oxide used as addition is a high purity compound of minimum 96%, this contains magnesium in the +2-oxidation state. Accordingly, this compound is considered an anhydrate of magnesium hydroxide. When combined with water, the magnesium hydroxide is created, which can be reverted to magnesia through heating, due to water evaporation. The magnesia contains Mg^{2+} and O^2 networks which creates ionic bonds. Moreover, the magnesia used is a non-flammable, hygroscopic substance. The table 2.6. summarize its chemical and physical properties [3].

Table 2.6. Chemical properties of magnesium oxide used in the manufacture of samples.

Chemical formula	MgO	**Specific weight**	40.30
Type	-325 powder	**Melting point**	2852°C
Boiling point	3600°C	**Density**	3.58 g/cm^3
Refractoriness index	1.736	**Solubility**	Insoluble in water and ethanol, soluble in acids and ammonia.

3.1.4. Nanoclay

Nanoclays are particles of layered silicate minerals. Depending on the chemical composition and morphology of the nanoparticles, nanoclays can be classified into the following categories: bentonite, montmorillonite, kaolinite, hectorite, and halloysite. Modified organic nanoclays represent an attractive class of organic-inorganic nanomaterials that can be used in the composition of polymeric nanocomposites as a rheological modifier, as a gas absorber or drug carrier.

Nanoclay-urile Montmorillonite

Montmorillonite is the most widely used type of nanoclay, used for combination with other materials. It consists of a thin layer of aluminosilicate with a thickness of 1 nm replaced by metal cations on multilayers of 10 µm. Depending on the modification surface of the nanoclay layers, it can be dispersed in a polymeric matrix to form polymer-clay nanocomposites. Within nanocomposites the nanoclay particles separate and form plates with very large dimensions of order nm × µm (Figure 3.1 a).

Halloysite is a natural aluminosilicate in the form of nanotubes. (Figure 3.1 b). Halloysite tubes are chemically similar to kaolin and have average dimensions of 15 × 1000 nm compared to carbon nanotubes. Halloysite tubes are hollow inside and can be used for controlled drug delivery as well as in other nanocomposite applications [57].

The nanoclay used in this study is powder with modified surface as it contains trimethyl ammonium stearyl in 25-30 %, wt. (Table 3.2).

Table 3.2. Physical characteristics of nanoclay.

Properties	**Specifications**
Color	White
Type	Powder
Loss on drying	< 3.0 %
Density	200 - 500 kg/m^3
Average size of particles	< 20 µ

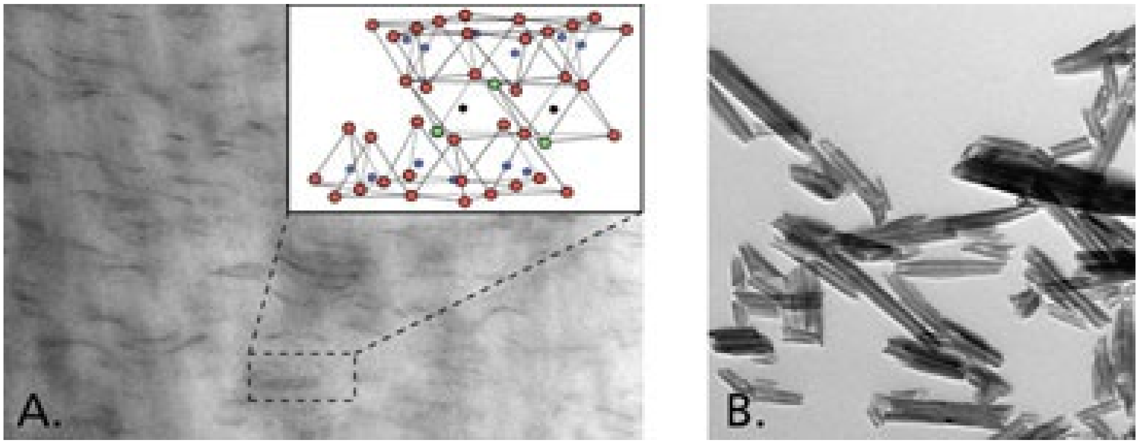

Figure 3.1. a) TEM micrograph of a 2% nylon nanocomposite - shows plates with nanometric thicknesses of clay dispersed throughout the polymer matrix. The insertion schematically shows the structure of the montmorillonite aluminosilicate layers. b) TEM micrograph of the halloysite clay nanotubes.

3.1.5. Screening/sifting process

The initial powder was sieved through several sieves with the following mesh sizes: 100 (150 µm), 140 (106 µm), 230 (63 µm), 325 (45 µm), using a vibrating sieve equipment Figure 3.2), Vibratory Sieve Shaker AS 200 with the following settings:

Sifting time: 20 min
Amplitude: 2 mm
Shaking range: 20 sec

Figure 3.2. The Sieve Shaker AS 200 sieving equipment [2].

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The analytical vibrators with sieve from the AS 200 series are used in research and development, in the quality control of raw materials, in intermediate and finished products as well as in production monitoring. The controllable electromagnetic drive system provides an optimal fit for each product. With the help of the equipment a large amount of screened particles can be obtained in a short time.

All AS 200 series sieve vibrators work by electromagnetic actuation, which is patented by RETSCH (EP 0642844). This device produces a 3D throwing motion that evens out the particles to be screened over the entire sieving surface of the sieve. Advantages: high sieving capacity, extremely smooth operation, short sieving time with high efficiency [2].

3.1.6. Determination of the density of alumina powder

The density of the raw material was calculated using the Le Chatelier densitometer. To reduce the deviation of the measurement method, the average representative value of three checks was used.

$$\rho_1 = \frac{74{,}39\ \mathrm{g}}{19{,}4\ ml} = 3{,}83\ \mathrm{g/cm^3}$$

$$\rho_2 = \frac{72{,}33\ \mathrm{g}}{19{,}1\ ml} = 3{,}78\ \mathrm{g/cm^3}$$

$$\rho_3 = \frac{68{,}82\ \mathrm{g}}{18\ ml} = 3{,}82\ \mathrm{g/cm^3}$$

$$\rho_\mathrm{m} = \frac{\rho_{1+}\rho_{2+}\rho_3}{3} = \frac{3{,}83 + 3{,}78 + 3{,}82}{3} = 3{,}81\mathrm{g/cm^3}$$

3.1.7. Particle size distribution analysis

After calculating the density, the particle size was determined using the COULTER LS 200 particle analyzer (Figure 3.3). The technical specifications are presented in the Table 3.3.

Description of equipment and method used for particle measurement (Laser diffraction)

The method of measuring, based on laser diffraction, the size of the particles benefits from an optical principle that detects small particles in the way of a light beam by capturing the light that is refracted by them. Depending on the angle of the incident beam and its intensity, the particle distribution can be deduced on the screen (Figure 3.4).

Table 3.3 Technical specifications.

COULTER LS series - Technical specifications	
Technology:	Fraunhofer laser diffraction, Polarization with differentiated intensity
Measurement range:	LS 100Q: 0.375 -m-948.2 µm
Measuring channels	LS200: 0.375 µm-2000 µm
Detectors:	LS230: 0.04 µm-2000 µm
Optical control:	All in one analysis
Laser:	LS 100Q: 84, LS 200: 92, LS 230: 116
Control Software:	LS 100Q: 126, LS 200: 126; LS 230: 132
Analysis time	Auto optical alignment
Amplifiers:	Solid State (750 nm)
Moisture:	Based on Microsoft Windows
Temperature range:	15-90 seconds
The power:	14 bit high resolution with (1-384), variable for large size
Sample modules	0-90% without condensation
Dimensions:	10-37 ° C

Figure 3.3. COULTER LS 200 particle analyzer.

The simplest flux model consists of a single-mode scattering of spheres that is displayed as a bright central point (known as the Airy disk), surrounded by dark, concentric light rings whose intensity decreases as it moves away from the center of the model, i.e. as the diffraction angle increases. The minimum diffraction depends on the particle size and is given by the size of the first closed ring, it is smaller the smaller the particle size (or, alternatively, depending on the size of the Airy disk) [4].

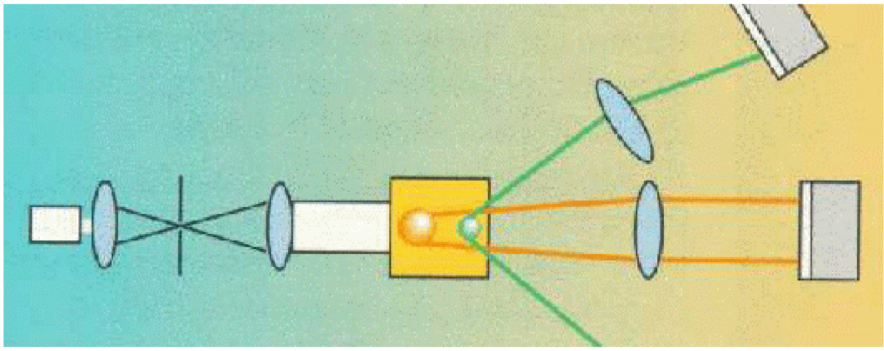

Figure 3.4. Schematic representation of the measuring principle used by the equipment.

With its dual optical system, the Coulter LS 200 can measure particle size without the need to change lenses. Moreover, the LS 200 is very flexible and can handle many different sample handling requirements.

The particle size after sieving is shown in Figure 3.5. Also, the Table 3.4 presents the volume of alumina particles.

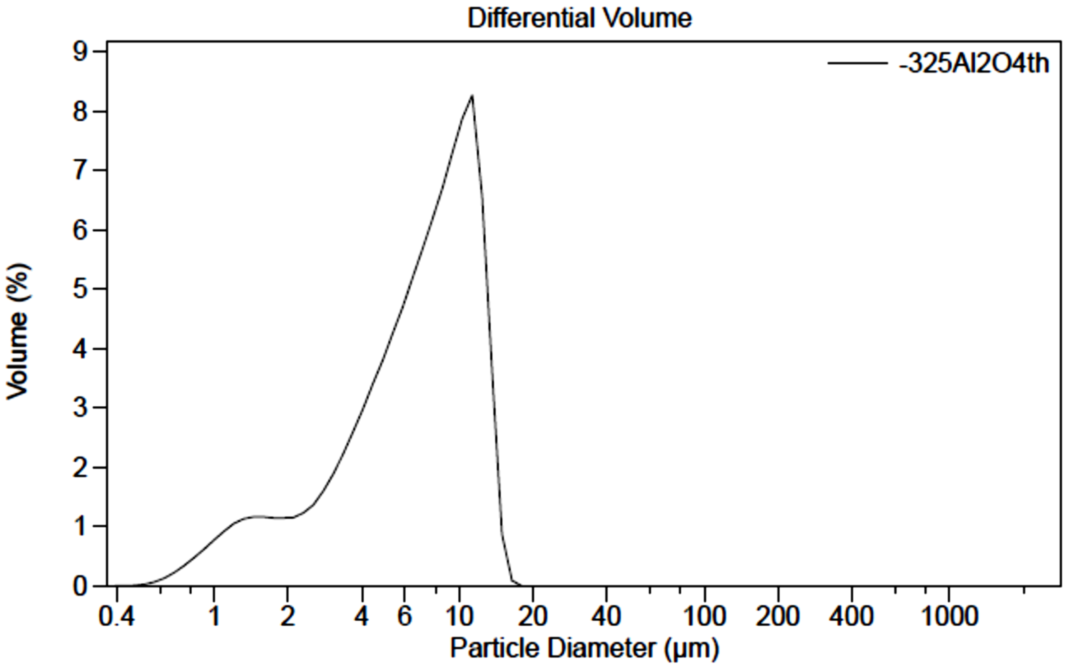

Figure 3.5. Particle size for alumina powder before grinding.

Table 3.4. The volume of alumina particles.

Particle volume						
	Quantity	Average (μm)	D.S (μm)	d10 (μm)	d50 (μm)	d90 (μm)
-325Al_2O_4th	100%	5.89	2.01	1.94	7.12	12.0

3.1.8. The milling process

Grinding was performed using the Retsch PM4 ball mill. Equipment description: Retsch ball mill, PM4 model with 4 agate cups for size reduction, grinding and homogenization of samples.

Technical specifications: Voltage: 220V, 50Hz; Power: 750W; Duration and speed can be adjusted electronically; The dimensions inside the grinding cups are (ø) x (H): 7.5 x 7 cm (300 ml); Machine room size (W) x (D) x (H): 81.5 x 64.5 x 110.5 cm.

Preparation of the mixture in order to reduce the size of the alumina powder: 85g of powder and 110g of balls of different sizes were introduced in each tank. Grinding lasted 8 hours at a grinding speed of 100 rpm. Every half hour the machine was stopped to return the composition to the bottom of the tanks, because it was expelled from the tank walls during grinding, this not being done in optimal conditions, and the particle size will be checked every hour. The decrease in particle size occurred according to the Table 3.5 and Figure 3.6.

Table 3.5. Volume of alumina particles after milling.

Particle volume						
	Quantity (μm)	Average (μm)	D.S. (μm)	d10 (μm)	d50 (μm)	d90 (μm)
D3_S2_1h	100%	5.98	2.09	1.84	7.27	12.8
D3_S2_2h	100%	5.57	2.05	1.75	6.74	11.8
D3_S2_3h	100%	5.53	2.05	1.72	6.65	11.8
D3_S2_4h	100%	4.60	2.01	1.47	5.73	9.34
D2_S2_5h	100%	3.22	1.94	115	4.05	6.31
(Average)	100%	4.98	2.03	1.59	6.09	10.4

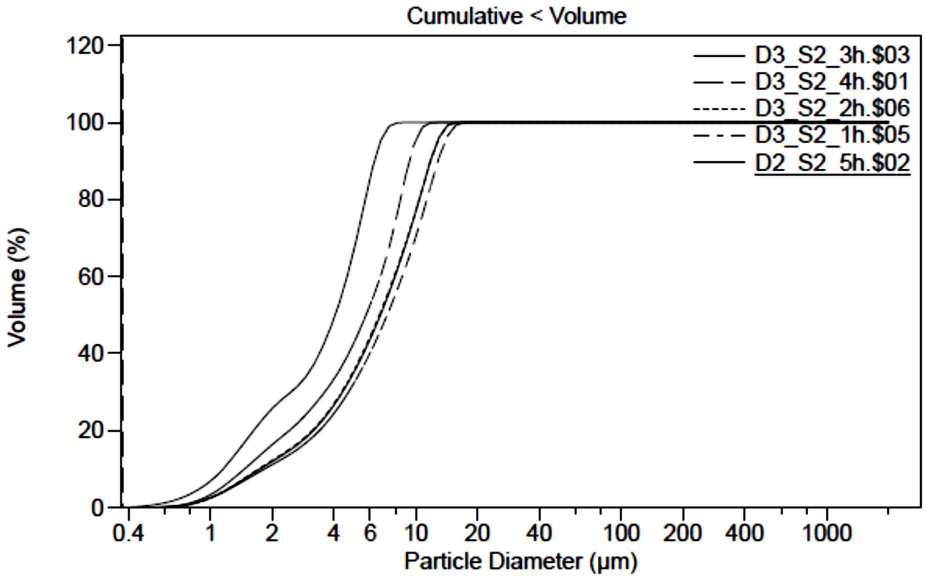

Figure 3.6. Particle size distribution of milled compounds.

3.1.9. The sintering processes

The stages of the sintering process can be described as follows: heating the oven to 300 °C, maintaining this temperature for 60 minutes, then raising the temperature to 900 °C at a speed of 10 °C / min, this temperature being maintained for 30 minutes. minutes followed by a further rise in temperature to 1500 °C at a rate of 5 °C / min, which is maintained for 120 minutes. This maintenance was followed by a slow cooling of the oven from 1500 °C to 900 °C. at a speed of 10 °C / min, then from 900 °C to ambient temperature at a speed of 1 °C / min. according to the Figure 3.7. The sintered samples are shown in the Figure 3.8.

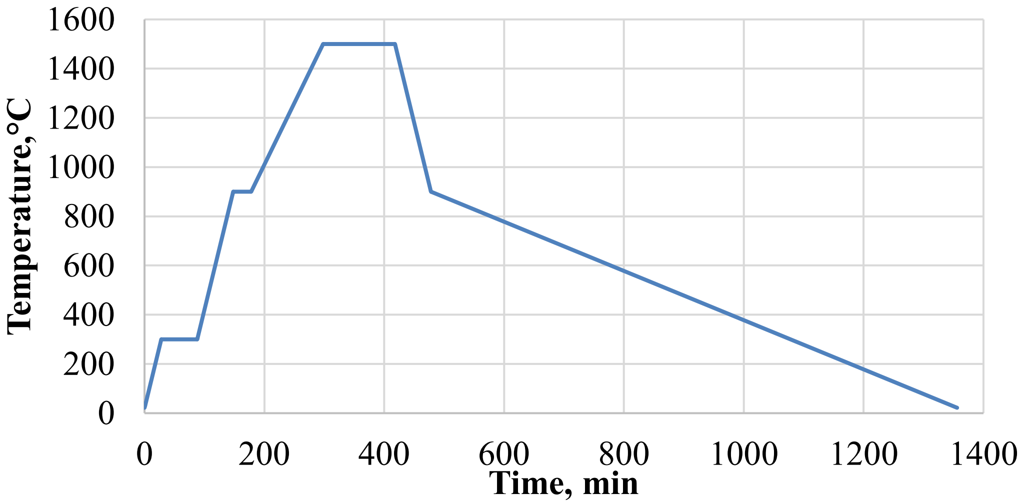

Figure 3.7. Temperature-time curve of the first sintering process.

Figure 3.8. Sintered samples (the samples diameter is 10 mm).

3.2. Experimental damples manufacturing

The samples have been manufacturing through a conventional pressing method. Therefore, established quantities of each component have been introduced in a closed die and pressed with different force.

3.2.1. Manufacture of alumina samples

10 samples of alcohol pre-impregnated alumina powder (Figure 3.9) with the following characteristics were manufactured: (Figure 3.10)

Sample 1: mass 3.25 g, pressing force 100 bar.

Sample 2: mass 3.25 g, pressing force 120 bar.

Sample 3: mass 3.00 g, pressing force 100 bar.

Sample 4: mass 3.00 g, pressing force 120 bar.

Samples A1 ÷ A6: mass 2.5 g, pressing force 100 bar.

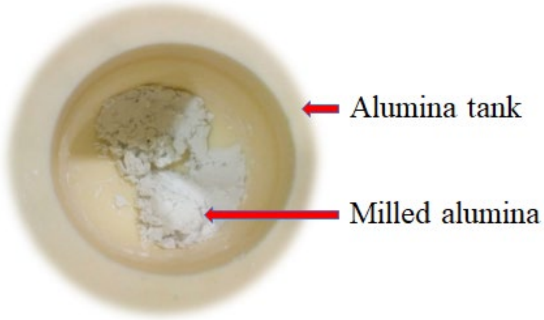

Figure 3.9. Mixing alumina with alcohol after milling.

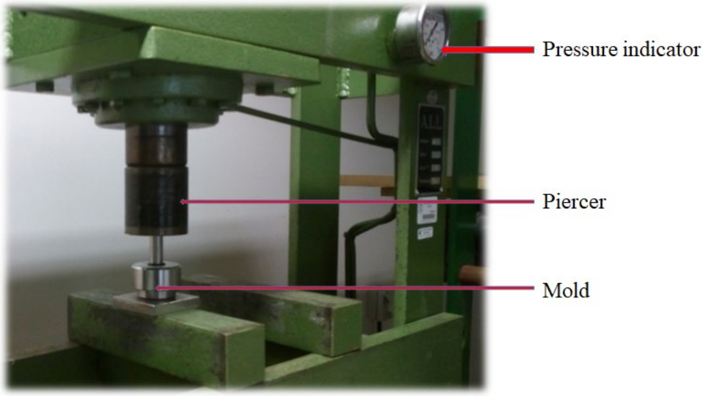

Figure 3.10. The press used for the manufacture of samples.

3.2.2. Manufacture of samples from alumina and magnesium oxide

These samples were made from a mixture of powders, containing 99% alumina and 1% magnesium oxide. To homogenize the composition, alcohol was added in the amount of 50% of the mass of the mixture and 100g of small balls, in one of the vessels of the Retsch PM4 machine, which will perform a cycle of 30 minutes at a speed of 100 rpm.

Samples AM1 ÷ AM10: mass 2.5 g, pressing force 100 bar.

3.2.3. Manufacture of samples from alumina and silicon oxide (molded)

Alumina powder was mixed with silicon oxide in the following proportions: 71.8% alumina and 28.2% silicon oxide (Mullite 3: 2). To homogenize the mixture, alcohol was added in an amount of 50% of its mass and 100g of small balls, then it was introduced into one of the vessels of the Retsch PM4 machine for 30 minutes at a speed of 100 rpm.

Samples M1 ÷ M12: mass 2.5 g, pressing force 100 bar.

3.2.4. Manufacture of samples from mullite and magnesium oxide

1% magnesium oxide powder was added to the mulch mixture. To homogenize the composition, alcohol was added in the amount of 50% of the mass of the mixture and 100g of small balls, in one of the vessels of the Retsch PM4 machine, which will perform a cycle of 30 minutes at a speed of 100 rpm.

Samples MM1 ÷ MM12: mass 2.5 g, pressing force 100 bar.

3.3. Experimental results

The obtained samples have been tested through diametral compression and hardness test. However, to clearly evaluate the behaviour of each sample, first, its physical properties must be determined.

Table 3.6. The physical properties of sintered samples.

Sample	**Diameter**	**Thickness**	**Dry weight [D]**	**Suspended weight [S]**	**Saturated weight [W]**	**Bulk density [B]**	**Mean density**
A1	13	6.45	2.322	1.684	2.519	2.78	2.8
A2	13	6.4	2.303	1.678	2.503	2.791	
A3	13	6.35	2.298	1.678	2.488	2.809	
A4	13	6.3	2.285	1.674	2.473	2.859	
A5	13	6.45	2.339	1.670	2.532	2.713	
A6	13	6.4	2.319	1.695	2.509	2.848	
AM1	13	6.2	2.247	1.623	2.435	2.767	2.805
AM2	13	6.25	2.247	1.635	2.436	2.805	
AM3	13	6.25	2.253	1.642	2.444	2.809	
AM4	13	6.2	2.249	1.635	2.436	2.807	
AM5	13	6.3	2.284	1.664	2.476	2.812	
AM6	13	6.25	2.264	1.650	2.453	2.819	
AM7	13	6.3	2.293	1.670	2.489	2.799	
AM8	13	6.3	2.286	1.667	2.476	2.825	
M1	10.8	9.4	1.655	1.099	1.922	2.01	2.003
M2	10.7	10	1.65	1.092	1.909	2.019	
M3	10.7	9.6	1.629	1.087	1.87	2.067	
M4	10.8	9.6	1.661	1.108	1.927	2.028	
M5	10.8	9.7	1.65	1.081	1.939	1.954	
M6	10.7	9.8	1.571	1.042	1.849	1.946	
M7	10.6	9.2	1.465	0.978	1.713	1.993	
M9	10.8	9.7	1.636	1.098	1.913	2.007	
MM2	11.3	9.8	1.654	1.107	2.113	1.788	1.912
MM3	10.8	9.8	1.798	1.202	1.823	1.825	
MM4	11.3	9.5	1.51	0.996	2.019	1.952	
MM5	11.2	9.7	1.714	1.141	1.82	1.953	
MM6	11.1	9.0	1.551	1.026	1.866	1.893	
MM7	11.0	8.9	1.566	1.039	1.816	1.936	
MM8	11.5	8.5	1.547	1.017	1.847	2.015	
MM9	11.1	9.6	1.592	1.057	1.902	1.911	
MM10	11.2	9.8	1.6	1.065	1.961	1.923	
MM12	11.0	9.8	1.656	1.1	1.964	1.929	

Notes: The bulk density, B, of a specimen in grams per cubic centimeter is the quotient of its dry weight divided by the exterior volume, including pores. Calculate B as follows: B, g/cm^3 = D/V. Exterior Volume, V. Obtain the volume of the test specimens in cubic centimeters by subtracting the suspended weight from the saturated weight, both in grams, as follows: V, cm^3 = W – S.

3.3.1. Samples characteristics

Table 3.6 presents the physical properties of the obtained samples, as can be seen the samples manufacturing process was very accurate, as the differences between the samples are in the range of 2-3%.

3.3.2. Diametral compression

The diametral compression test, also called the Brazilian disc test or the diametral compression test, is considered to be a safe and accurate method of determining the strength of brittle materials (Figure 3.11). The test is used to determine the compressive strength of concrete, rocks, carbides, ceramics, pharmaceutical materials in dosage form, and other fragile materials. If the material deforms plastically, special attention must be paid to the maximum value of the stress, as this can change significantly. By entering a prefix, the method can be used to find the factors that influence the stress or breaking strength. The diametrical compression test is also used to determine the crack resistance of compact powder materials. For powdered materials no prior processing of the sample shape is permitted, this must be obtained directly to the desired geometry. During the test, a thin disk is compressed along the diameter until it is destroyed. The compressive force induces a tension perpendicular to the pressed diameter, which is constant over a large region around the center of the disc. Compressive strength is considered to be the value of the force at which destruction is initiated [56].

Figure 3.11. Shimadzu equipment.

The tests were made using a Shimadzu machine with a loading velocity of 0.5 mm/min.

The values of the diametral compressive strength of the sample are presented in the Table 3.7.

Table 3.7. Diametral compressive strength of the samples.

Sample	Load, N	Average	Compressive strength, MPa	Average	Stdv.
1	2572.71	2537.79	17.26	17.4	3.21
2	1810.20		11.98		
3	3457.14		24.19		
4	2311.13		16.17		
A1	1030.38	2492.46	7.88	19.13	6.96
A2	2102.16		16.09		
A3	3524.14		27.18		
A4	2385.85		18.55		
A5	2574.39		19.55		
A6	3337.82		25.54		
AM1	1292.04	1935.45	10.21	15.14	2.46
AM2	2186.24		17.13		
AM3	2312.19		18.12		
AM4	1887.16		14.91		
AM5	2082.74		16.19		
AM6	1948.99		15.27		
AM7	2057.48		15.99		
AM8	1716.77		13.34		
M1	947.74	759.55	5.94	4.69	0.80
M2	674.97		4.02		
M3	591.19		3.66		
M4	851.82		5.23		
M5	782.86		4.76		
M6	688.49		4.18		
M7	838.73		5.48		
M9	700.58		4.26		
MM2	480.01	646.61	2.76	4.79	2.25
MM3	1530.59		9.21		
MM4	614.52		3.64		
MM5	696.23		4.08		
MM6	641.43		4.09		
MM7	1295.99		8.43		
MM8	622.46		4.05		
MM9	657.75		3.93		
MM10	435.39		2.53		
MM12	871.73		5.15		

The following figures show the graphs between force and displacement for the samples that showed a maximum value, the closest to the value of the average compressive strength of the category to which they belonged.

Figure 3.12 shows the behaviour of the sample A5 to diametral compression. As can be seen, the graph shows a significant variation at approximative 0.1 mm and ≈650 N and a linear increase until the maximum load of ≈2600 N. Moreover, both samples A5 and AM6 (Figure 3.13) exhibit a brittle behaviour.

The magnesium oxide addition in alumina samples resulted in a 30% decrease in compressive strength. Accordingly, it can be stated that, the magnesium oxide particles influenced the samples homogeneity, Moreover, they act as structural defects in the alumina matrix.

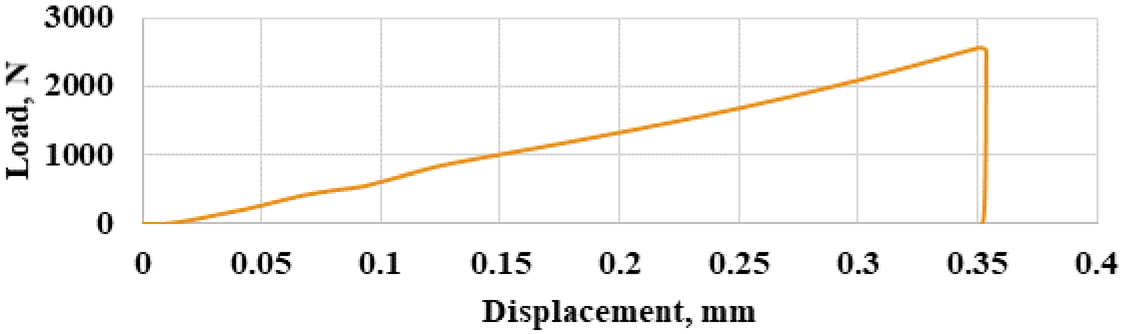

Figure 3.12. Diametral compression behavior of sample A5.

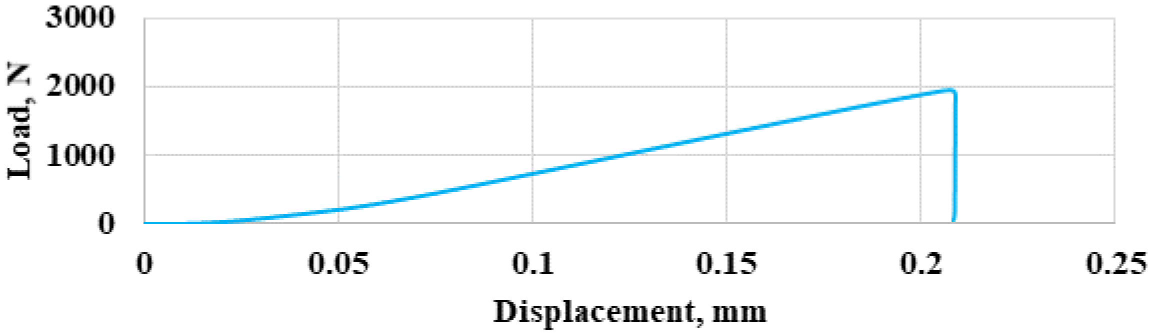

Figure 3.13 Diametral compression behavior of sample AM6.

In Figure 3.14 it can be clearly seen the differences between the compressive strength behaviour of sample AM6 and sample A5. Also, it can be observed that, in the point where the curve specific to A5 sample showed a fluctuation, the curve specific to AM6 showed a linear increase, which resulted in a much higher energy consumption for the same displacement. In other words, before breaking, at the same displacement, the AM6 sample accumulate much more energy than A5 sample (Figure 3.14) which can be correlate with a much denser sample, yet, it has a lower ultimate strength.

Compared to the behaviour of alumina samples, the mullite samples (Figure 3.15 and Figure 3.16) exhibit a completely different behaviour. Instead of linear increasing of load, the compressive curves specific to mullite samples exhibit multiple fluctuations, due to cracks occurrence. In the last 0.2 mm, before failure, the samples exhibit a significant increase in load, due to the cracks blocking resulting in a compact body.

Compared to AM samples, the magnesium oxide addition in mullite samples showed minimum influence. However, even the curve specific to MM12 sample is above the one specific to M5 sample, i.e., it resists to higher load, the average compressive strength of MM samples is 15% lower than M samples (Figure 3.17).

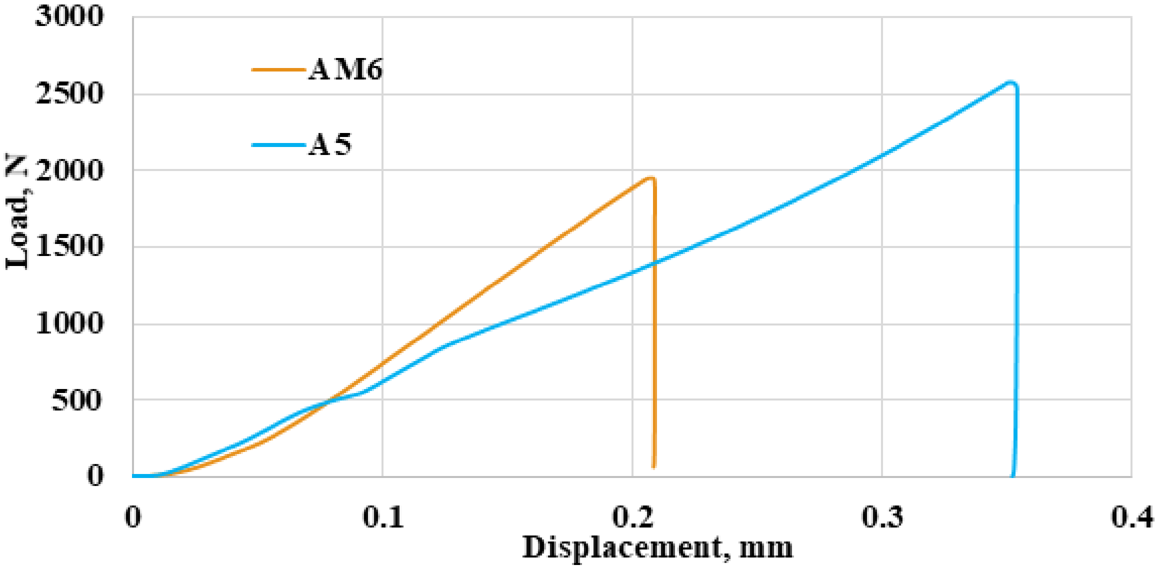

Figure 3.14. Graphic comparison between the diametrical compression behavior of samples A5 and AM6.

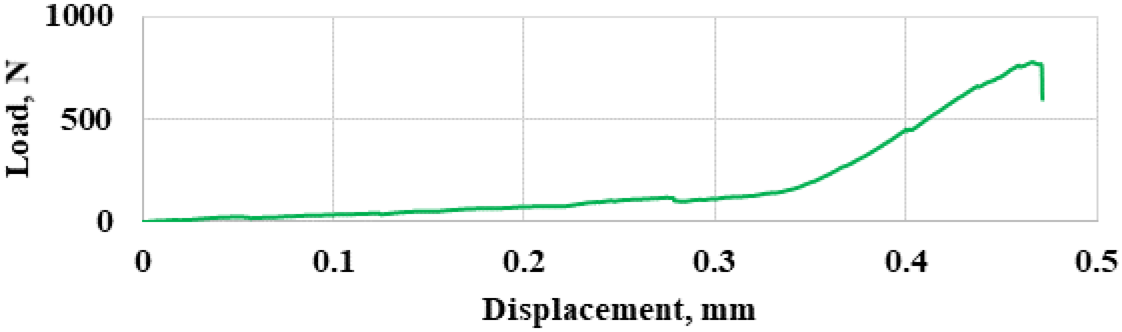

Figure 3.15. Diametral compression behavior of sample M5.

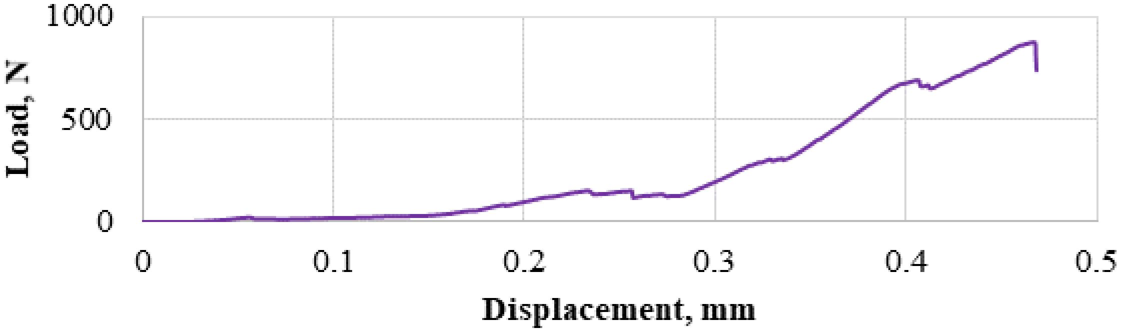

Figure 3.16. Diametral compression behavior of sample MM12.

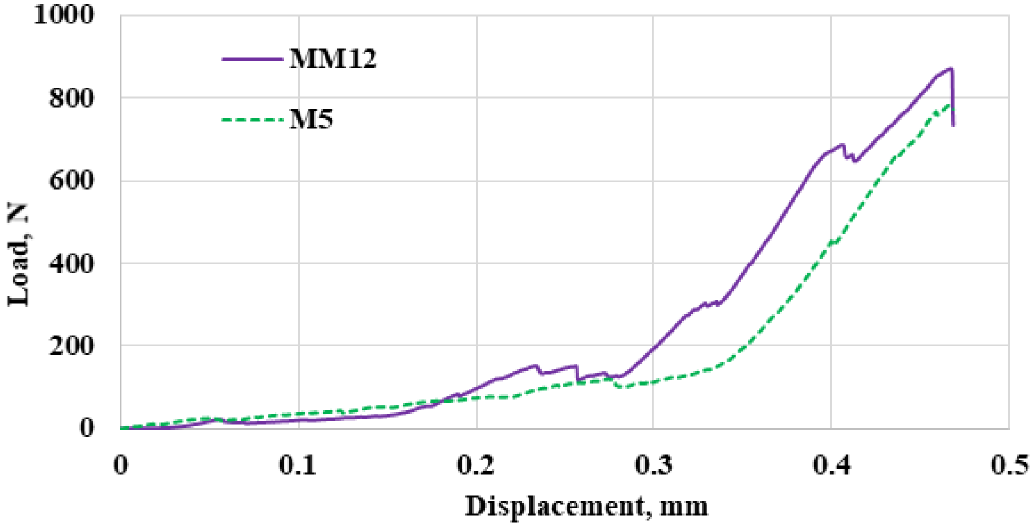

Figure 3.17 Graphic comparison between the diametrical compression behavior of samples MM12 and M5.

According to the obtained results, the magnesium oxide addition in mullite and alumina matrix resulted in a decrease in compressive strength of the samples.

3.3.3. Hardness testing by Vickers method

Vickers hardness was measured using Mitutoyo VK-C2 equipment. All measurements were performed on polished surfaces (mirror type). The surface of the sample was prepared by means of Struers RotoPol-21 equipment. In order to be able to use this equipment, the samples were introduced into epoxy resin in order to create a support with the diameter required by the machine.

Prior preparation of the sample substrates, it is required to polish the surfaces. The two parts of the sample holders were polished in order to obtain a flat fixing surface and to remove the excess resin from the sample surface on the other surface using abrasive paper disc equipment (Figures 3.18 and 3.19).

Figure 3.18. Sample embedded in epoxy resin, after surface polishing.

Figure 3.19. The first stage of sample polishing.

The samples have been polished following the steps described in Table 3.8, employing the materials from column 1 to 3 at the polishing parameters described in columns 4 to 6.

Table 3.8. Polishing method steps and parameters.

Disks	Gri, µm	Polishing media	Rotation, rot/min	Load, N	Time, min
MD-Plan	6	DP-Lubri. +DP-Spray (6 µm)	150	25	30
MD-Dac	3	DP-Lubri. +DIAPAT-M (3 µm)	150	20	30
MD-Dur	1	DP-Lubri. +P-Spray P (1 µm)	150	15	5
MD-Chen	OP-S	DP-U Suspension	150	10	10

The Vickers method consists in imprinting the surfaces with a diamond by pressing it on the surface of the tested specimen. 10 fingerprints were made on the surface of each sample with a loading force of 10•9.81 N (HV1) for 15 s. The tests were performed under laboratory ambient conditions. After impression, the length of the diagonals of the square obtained on the surface was measured using optical microscopy using a lens with a magnification of 20X (Figure 3.20). The Vickers hardness value was calculated using the following relation (eq. 3.1):

$$HV = \frac{1.85 \bullet F}{d^2} \tag{3.1}$$

Where:

HV = Vickers hardness;

F = Load force (N);

d = Arithmetic mean of the lengths of the two diagonals (mm).

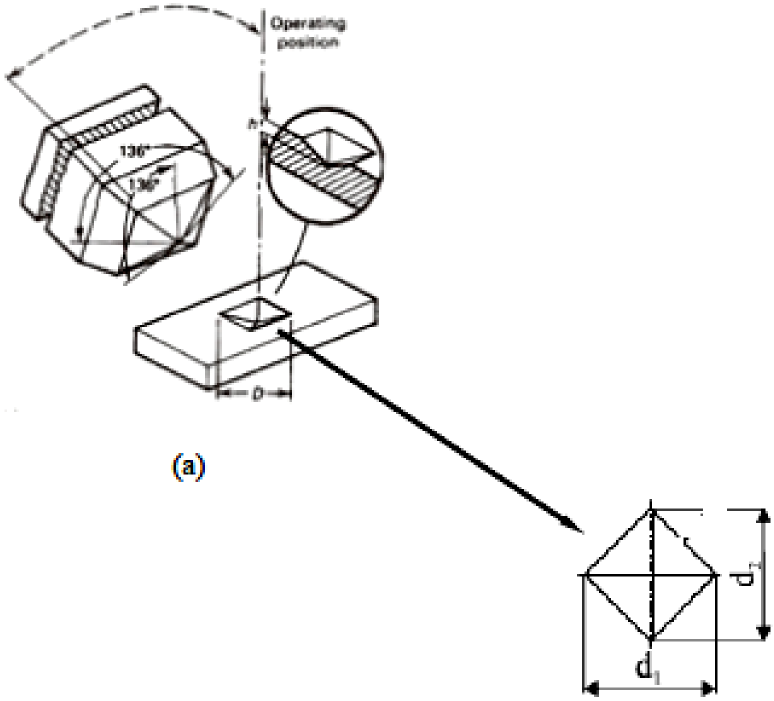

Figure 3.20. Hardness testing by Vickers method - Schematic representation of the method and shape of the indent.

The deviation, S, was calculated by means of the following equation (eq. 3.2):

$$S = \sqrt{\frac{\sum(HVm - HVn)^2}{n - 1}} \tag{3.2}$$

Where: HVm = mean value of the hardness (eq. 3.3);

$$HVm = \frac{\sum HVn}{n} \tag{3.3}$$

HVn = HV obtain by *n* indents;

n = number of indents.

The samples hardness values are shown in the Table 3.9.

Table 3.9. Samples hardness values.

Sample	**D1**	**D2**	**(D1+D2)/2**	**HV,**	**Mean HV,**	**S,**
				GPa	**GPa**	**GPa**
A8	107.20	118.10	112.65	14.32	12.32	0.67
	129.10	132.70	130.90	10.61		
	120.10	119.10	119.60	12.70		
	119.50	121.90	120.70	12.47		
	127.80	127.70	127.75	11.14		
	125.30	125.50	125.40	11.56		
	113.40	115.80	114.60	13.84		
	122.00	124.70	123.35	11.94		
	120.20	120.50	120.35	12.55		
	119.50	126.00	122.75	12.06		
A7	124.40	127.20	125.80	11.48	11.88	0.13
	128.10	130.70	129.40	10.85		
	125.20	125.80	125.50	11.54		
	126.80	126.80	126.80	11.30		
	120.90	121.40	121.15	12.38		
	123.60	120.00	121.80	12.25		
	120.40	120.50	120.45	12.53		
	120.80	119.20	120.00	12.62		
	125.00	124.60	124.80	11.67		
	121.40	122.50	121.95	12.22		
AM9	121.20	121.90	121.55	12.30	12.39	0.03
	109.10	115.20	112.15	14.45		
	117.90	122.60	120.25	12.57		
	117.70	128.10	122.90	12.03		
	111.80	113.60	112.70	14.31		
	121.70	128.20	124.95	11.64		
	123.80	125.40	124.60	11.71		
	121.20	123.00	122.10	12.19		
	129.40	129.40	129.40	10.85		
	123.90	123.90	123.90	11.84		
AM10	127.80	128.40	128.10	11.07	11.46	0.13
	125.50	126.50	126.00	11.45		
	121.10	124.00	122.55	12.10		
	120.30	121.60	120.95	12.42		
	123.30	124.30	123.80	11.86		
	127.40	138.90	133.15	10.25		
	129.70	128.70	129.20	10.89		
	126.30	131.10	128.70	10.97		
	129.40	128.00	128.70	10.97		
	119.70	120.10	119.90	12.64		

M11	160.00	159.10	159.55	7.14	6.53	0.20
	168.10	157.10	162.60	6.87		
	149.90	156.60	153.25	7.74		
	152.60	1596.00	874.30	0.24		
	158.30	158.80	158.55	7.23		
	168.00	173.50	170.75	6.23		
	162.10	162.10	162.10	6.92		
	152.20	156.70	154.45	7.62		
	150.60	154.50	152.55	7.81		
	156.40	155.10	155.75	7.49		
M8	252.50	252.50	252.50	2.85	3.11	0.09
	249.30	261.70	255.50	2.78		
	232.80	232.30	232.55	3.36		
	240.40	243.00	241.70	3.11		
	228.60	242.30	235.45	3.28		
	243.90	243.90	243.90	3.05		
	240.60	241.40	241.00	3.13		
	256.60	259.10	257.85	2.73		
	245.30	241.90	243.60	3.06		
	218.20	223.10	220.65	3.73		
MM11	187.90	187.80	187.85	5.15	5.20	0.02
	187.90	187.20	187.55	5.17		
	195.20	189.40	192.30	4.91		
	183.50	190.10	186.80	5.21		
	191.40	192.40	191.90	4.93		
	182.40	180.10	181.25	5.53		
	181.50	188.20	184.85	5.32		
	189.10	194.30	191.70	4.95		
	178.30	185.60	181.95	5.49		
	181.30	189.30	185.30	5.29		
MM13	412.80	449.80	431.30	0.98	1.94	0.32
	402.80	405.80	404.30	1.11		
	397.20	399.70	398.45	1.14		
	224.50	231.50	228.00	3.50		
	204.80	211.70	208.25	4.19		
	312.00	315.00	313.50	1.85		
	258.60	258.60	258.60	2.72		
	388.30	416.70	402.50	1.12		
	458.00	469.10	463.55	0.85		

3.3.4. Manufacture of nanoclay samples and their characteristics after the sintering process

The required amount of nanoclay for each composition (2% and 4% of the amount of mixture) was added to a vessel over the alumina or the mixture of alumina and magnesium oxide. In order to homogenize, 10 balls with a diameter of 10 mm and alcohol were added until a viscous liquid was obtained. The vessel was introduced into the Retsch Planetary PM4 mill for half an hour at a machine speed of 150 rpm. After 30 minutes the mixture was subjected to ultrasound for 30 minutes. After the action of the ultrasound, the mixture was reintroduced into the mixing machine for another 30 minutes, at the same parameters of the equipment. After the second mixing the composition was subjected to ultrasound for another 30 minutes. It was then placed in the oven for drying until the viscosity of the composition required for the manufacture of the samples was obtained.

The samples were manufactured with the following characteristics:

Samples 2A1 ÷ 2A6: Alumina + 2% nanoclay, mass 2.5 g, pressing force 100 bar.

Samples 4A1 ÷ 4A6: Alumina + 4% nanoclay, mass 2.5 g, pressing force 100 bar.

Samples 2M1 ÷ 2M6: Alumina + 1% magnesium oxide + 2% nanoclay, mass 2.5 g, pressing force 100 bar.

Samples 4M1 ÷ 4M6: Alumina + 1% magnesium oxide + 4% nanoclay, mass 2.5 g, pressing force 100 bar.

The characteristics of the samples after the sintering process are shown in Table 3.10.

The outer volume, V, is measured in cm^3 *and is obtained by subtracting the suspended weight from the saturated weight in g, according to:* $V, cm^3 = W - S$

2A1-2A5 = Alumina with 2% nanoclay

4A1-4A5 = Alumina with 4% nanoclay

2M1-2M5 = Alumina + 1% magnesium oxide and 2% nanoclay

4M1-4M5 = Alumina + 1% magnesium oxide and 4% nanoclay

Table 3.10. Characteristics of the samples after the sintering process according to Figure 3.7

Sample	Diameter, Mm	Thickness, Mm	Dry weight, g	Suspended weight, g	Saturated weight, g	Density, g/cm^3	Average density
4M1	12.7	5.4	2.073	1.515	2.172	3.15	3.08
4M2	12.7	5.8	2.185	1.576	2.293	3.04	
4M3	12.7	5.6	2.143	1.567	2.248	3.14	
4M4	12.7	5.7	2.152	1.567	2.26	3.1	
4M5	12.7	5.8	2.2	1.584	2.31	3.03	
4M6	12.7	5.8	2.22	1.602	2.328	3.05	
2M1	12.8	5.8	2.15	1.569	2.289	2.98	2.97
2M2	12.8	5.7	2.145	1.571	2.284	3	
2M3	12.8	5.9	2.22	1.63	2.372	2.99	
2M4	12.8	5.8	2.175	1.588	2.322	2.96	
2M5	12.8	5.9	2.18	1.567	2.321	2.89	
2M6	12.8	5.8	2.172	1.595	2.319	3	
2A1	12.7	5.7	2.112	1.541	2.243	3	2.98
2A2	12.7	5.5	2.075	1.503	2.203	2.96	
2A3	12.7	5.7	2.137	1.567	2.269	3.04	
2A4	12.7	5.7	2.128	1.538	2.257	2.95	
2A5	12.7	5.7	2.135	1.561	2.27	3.01	
2A6	12.7	5.7	2.109	1.521	2.241	2.92	
4A1	12.7	5.5	2.084	1.524	2.207	3.05	2.99
4A2	12.7	5.6	2.106	1.533	2.225	3.04	
4A3	12.7	5.7	2.116	1.525	2.226	3.01	
4A4	12.7	5.6	2.109	1.535	2.222	3.06	
4A5	12.7	5.8	2.213	1.537	2.332	2.78	
4A6	12.7	5.6	2.117	1.537	2.235	3.03	

Note: The bulk density, B, of the samples in grams per cm^3 is the ratio between the dry weight and the volume of the sample (including pores). This was calculated using the following relation: B, g/cm^3 = D/V.

3.3.5. Testing nanoclay samples at diametric compression and hardness

The test was performed using the Shimadzu machine with a punch velocity of 0.5 mm/min. The values of diametral compression of the samples are shown in Table 3.11 and the diametral compression behavior is presented in Figures 3.21, 3.22, 3.23, 3.24, 3.25 and 3.26.

Table 3.11. Diametral compression of the samples.

Sample	Load, N	Average	Compressive strength, MPa	Average	Stdv.
2A1	2888.51	3001.90	25.41	26.63	6.28
2A2	3519.71		32.09		
2A3	1849.10		16.26		
2A4	3416.60		30.06		
2A5	3335.57		29.34		
4A1	2306.33	3409.49	21.03	30.50	6.49
4A2	3516.34		31.49		
4A3	3212.95		28.27		
4A4	3698.21		33.12		
4A5	4313.61		38.62		
2M1	2287.04	2785.52	19.62	23.84	7.42
2M2	3734.20		32.59		
2M3	1965.42		16.57		
2M4	2250.91		19.31		
2M5	3689.98		31.12		
4M1	2481.91	2855.09	23.05	25.22	4.88
4M2	3622.10		31.32		
4M3	2400.57		21.49		
4M4	2348.58		20.66		
4M5	3422.28		29.59		

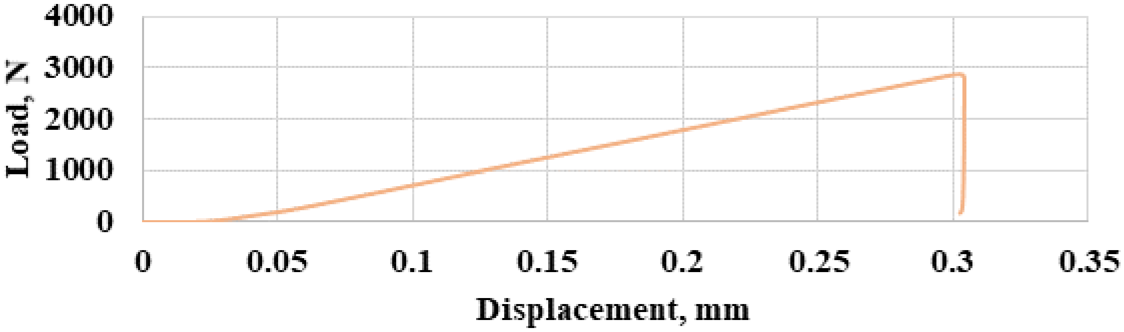

Figure 3.21. Diametral compression behavior of sample 2A1.

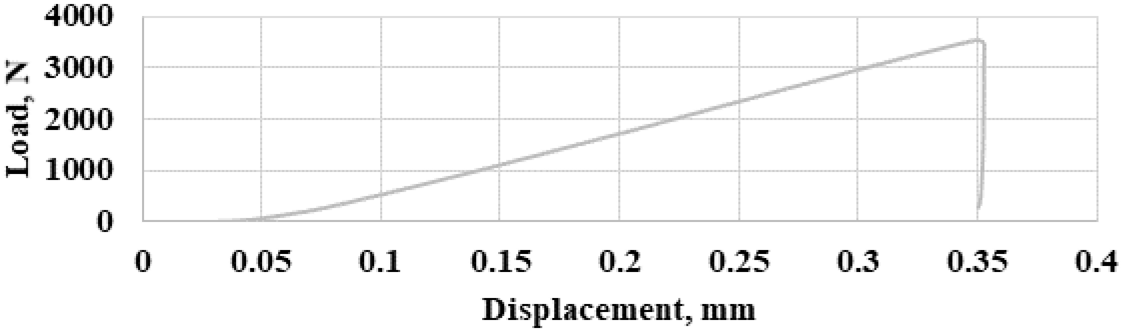

Figure 3.22. Diametral compression behavior of sample 4A2.

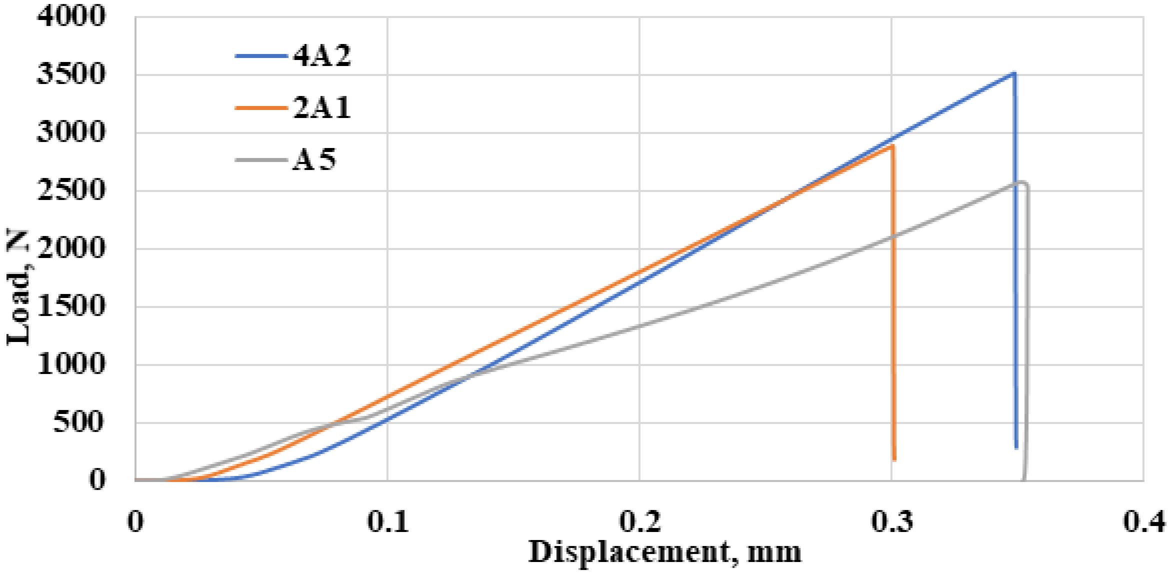

Figure 3.23. Comparison of diametral compression behavior between 4A2, 2A1 and A5 samples.

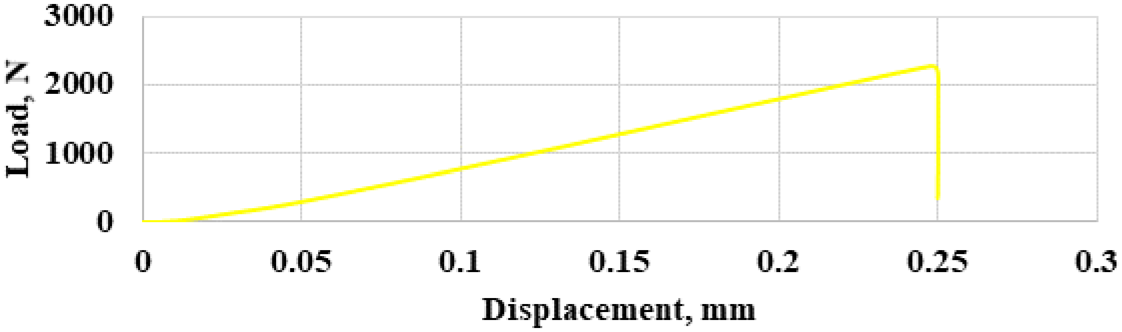

Figure 3.24. Diametral compression behavior of sample 2M1.

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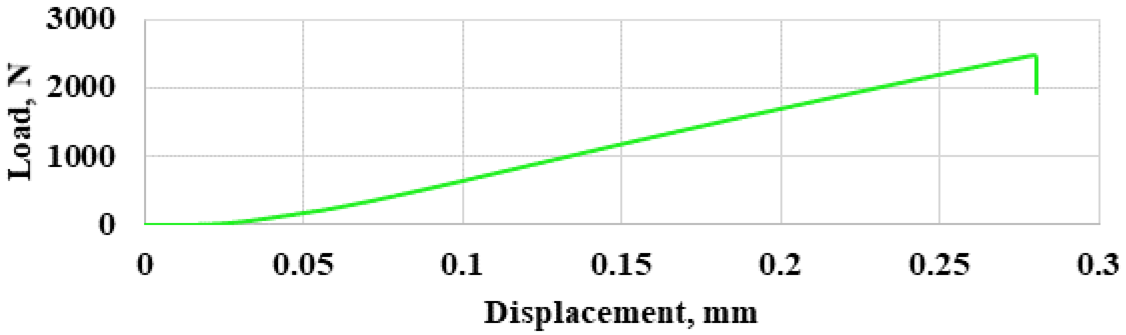

Figure 3.25. Diametral compression behavior of sample 4M1.

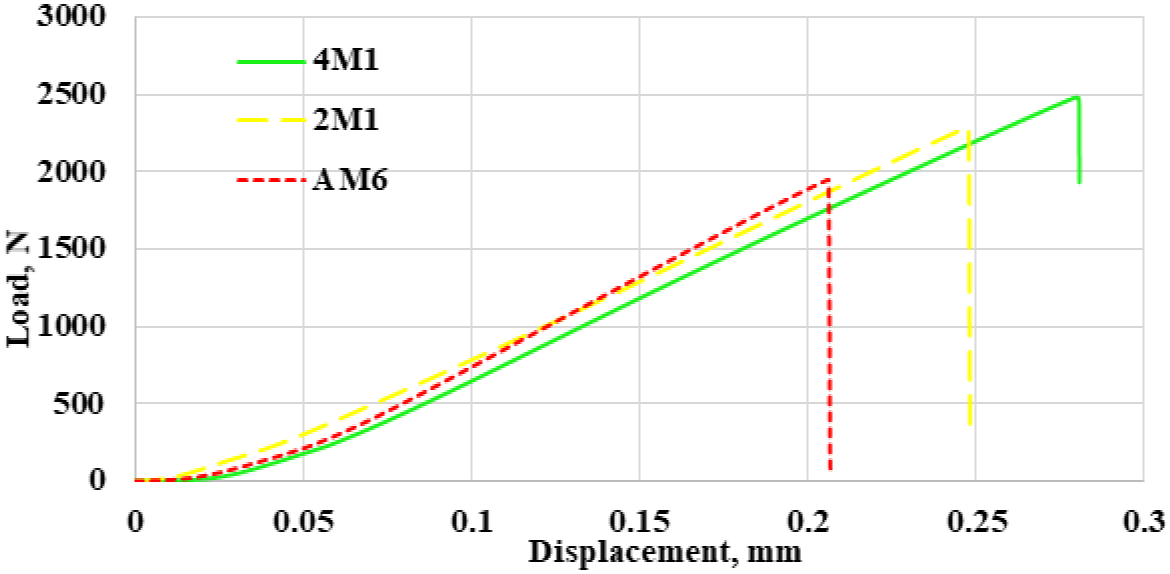

Figure 3.26. Comparison of diametral compression behavior between 4M1, 2M1 and AM6 samples.

The hardness of the samples was measured by the Vickers method using the Mitutoyo AVK-C2 equipment according to the previous procedure. The hardness values of ceramic samples with nanoclay addition are shown in Table 3.12.

Table 3.12. Hardness values of ceramic samples with nanoclay addition.

Sample	D1	D2	HV	(D1+D2)/2	HV, GPa	Mean HV, GPa	S, GPa
2A6	111.60	111.10	1495.00	111.35	14.66	14.27	0.13
	113.00	111.20	1475.00	112.10	14.46		
	109.40	109.20	1552.00	109.30	15.21		
	121.40	119.60	1277.00	120.50	12.52		
	111.20	111.60	1494.00	111.40	14.64		
	110.70	116.10	1442.00	113.40	14.13		
	114.60	117.20	1380.00	115.90	13.53		
	108.70	109.40	1559.00	109.05	15.28		
	118.20	119.80	1309.00	119.00	12.83		
	107.80	109.10	1576.00	108.45	15.45		
4A6	92.00	94.70	2128.00	93.35	20.85	18.78	0.69
	92.30	93.80	2141.00	93.05	20.99		
	97.40	101.90	1867.00	99.65	18.30		
	102.10	103.70	1751.00	102.90	17.16		
	103.80	104.00	1717.00	103.90	16.83		
	100.70	101.60	1812.00	101.15	17.76		
	100.20	101.00	1832.00	100.60	17.96		
	97.20	98.80	1930.00	98.00	18.92		
	95.60	96.10	2018.00	95.85	19.78		
	96.20	98.00	1966.00	97.10	19.27		
2M6	109.30	110.00	1542.00	109.65	15.12	14.59	0.17
	115.00	118.40	1361.00	116.70	13.34		
	109.50	111.00	1529.00	110.25	14.95		
	121.50	114.20	1335.00	117.85	13.08		
	111.50	113.00	1470.00	112.25	14.42		
	106.90	108.60	1597.00	107.75	15.65		
	105.70	110.90	1581.00	108.30	15.49		
	105.10	107.80	1636.00	106.45	16.04		
	112.40	114.10	1446.00	113.25	14.17		
	115.00	115.70	1392.00	115.35	13.66		
4M6	97.30	97.40	1956.00	97.35	19.18	20.41	0.41
	95.50	95.50	2033.00	95.50	19.93		
	91.20	93.60	2172.00	92.40	21.29		
	90.90	95.10	2144.00	93.00	21.01		
	92.30	95.20	2109.00	93.75	20.68		
	94.20	94.20	2063.00	94.20	20.48		
	92.90	95.00	2100.00	93.95	20.59		
	94.40	94.90	2070.00	94.65	20.29		
	93.80	94.10	2100.00	93.95	20.59		
	94.30	95.90	2050.00	95.10	20.09		

3.3.6. Samples obtained by another sintering process

The samples were manufactured with the following characteristics:

Samples 2A7 ÷ 2A8: Alumina + 2% nanoclay, mass 2.5 g, pressing force of 100 bar.

Samples 4A7 ÷ 4A8: Alumina + 4% nanoclay, mass 2.5 g, pressing force 100 bar.

Samples 2M7 ÷ 2M8: Alumina + 1% magnesium oxide + 2% nanoclay, mass 2.5 g, pressing force 100 bar.

Samples 4M7 ÷ 4M8: Alumina + 1% magnesium oxide + 4% nanoclay, mass 2.5 g, pressing force 100 bar.

3.3.6.1. The second sintering process

The sintering process can be described as follows: heating the oven to 300°C, maintaining this temperature for 60, then raising the temperature to 900°C at a speed of 10°C/min, this temperature being maintained for 30 minutes followed by a further increase in temperature to 1400°C at a speed of 5°C/min, which is maintained for 120 minutes. This maintenance was followed by a slow cooling of the oven from 1400°C to 900°C. at a speed of 10°C / min, then from 900°C to ambient temperature at a speed of 1°C/min, according to the Figure 3. 27. The characteristics of sintered samples according to the second sintering process are shown in Table 3.13.

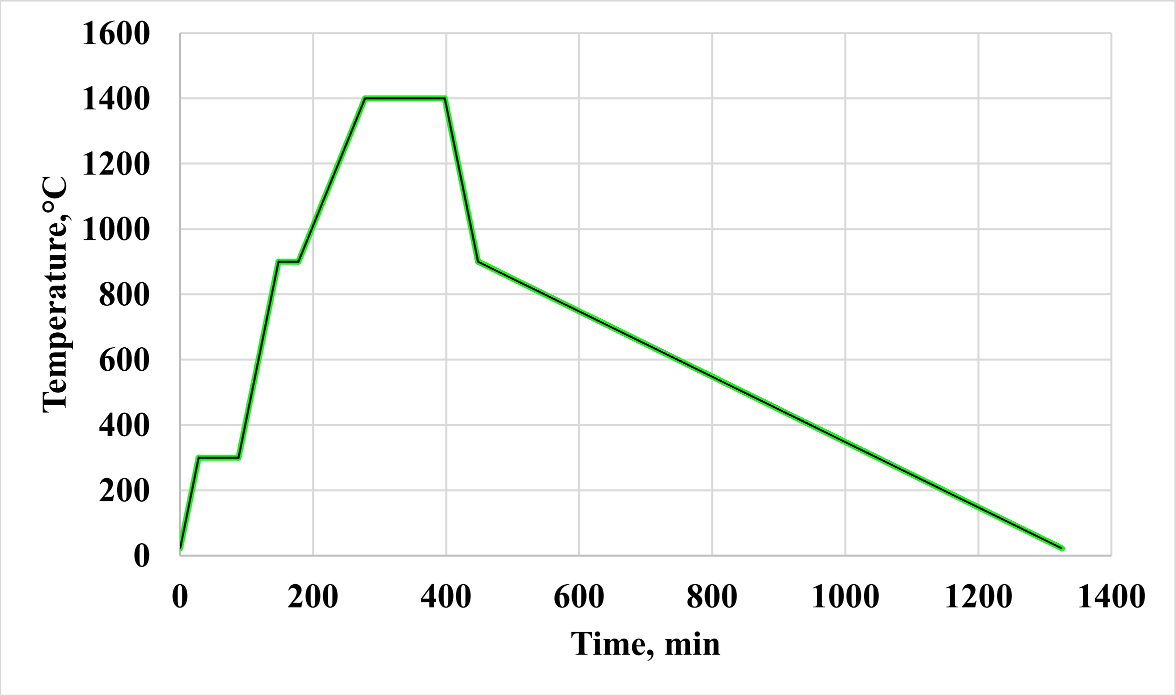

Figure 3.27. Temperature-time curve of the second sintering process.

Table 3.13. Characteristics of sintered samples according to the second sintering process.

Sample	Diameter, mm	Thickness, mm	Dry weight, g	Suspended weight, g	Saturated weight, g	Density, g/cm^3	Average density
4M7	12.9	6.5	2.496	1.791	2.646	2.91	2.9
4M8	12.8	5.9	2.249	1.617	2.390	2.9	
2M7	12.9	5.7	2.092	1.509	2.246	2.83	2.82
2M8	12.9	5.7	2.127	1.531	2.284	2.82	
4A7	12.9	5.7	2.091	1.503	2.229	2.88	3.01
4A8	12.9	5.7	2.138	1.547	2.227	3.14	
2A7	12.9	5.7	2.178	1.539	2.306	2.83	2.81
2A8	12.9	5.8	2.138	1.526	2.287	2.8	

3.3.6.2. Diameter compression

The test was performed using the Shimadzu machine with a punch velocity of 0.5 mm/min. The values of diametral compression are presented in the Table 3.14.

Table 3.14. Diametral compression test of the obtained samples.

Sample	Load, N	Compressive strength, MPa
2A7	2636.63	22.59
4A7	4147.74	35.93
2M7	3159.13	27.36
4M7	2577.44	19.58

The alumina with 2% nanoclay addition exhibit (Figure 3.28) a typical brittle behaviour. According to the graph, the sample showed a linear loading (no visible cracks formation) until failing at ≈2500 N and 0.27 mm.

When 4% of alumina was replaced with nanoclay, the diametral compression behaviour of the samples changed significantly. As can be seen from Figure 3.29, the 4A7 sample showed a linear increase in load up to ≈3500 N and ≈0.35 mm, above this point the curve slop changed and the displacement continued to increase while the load remains almost the same. Therefore, a crack appeared at the point of change which advanced through the sample, yet the failure behaviour was more specific to a flexible material than a brittle one. Considering this behaviour, it can be state that the use of this type of materials compared

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with pure alumina is much more recommended, since the failure occurrence can be observed and remediated in time.

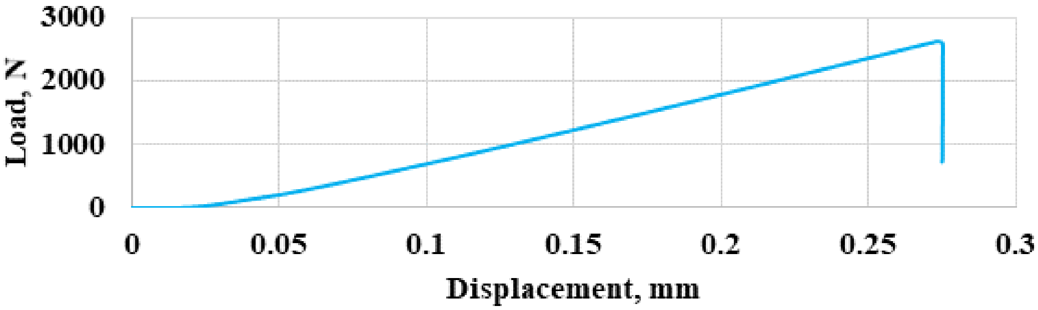

Figure 3.28. Diametral compression behavior of sample 2A7.

Nevertheless, when magnesium oxide was also introduced in the samples (Figure 3.30 and Figure 3.31), the behaviour was the samples changed to typical brittle. However, the compressive strength of this samples is much higher than the one of alumina with magnesia samples without nanoclay.

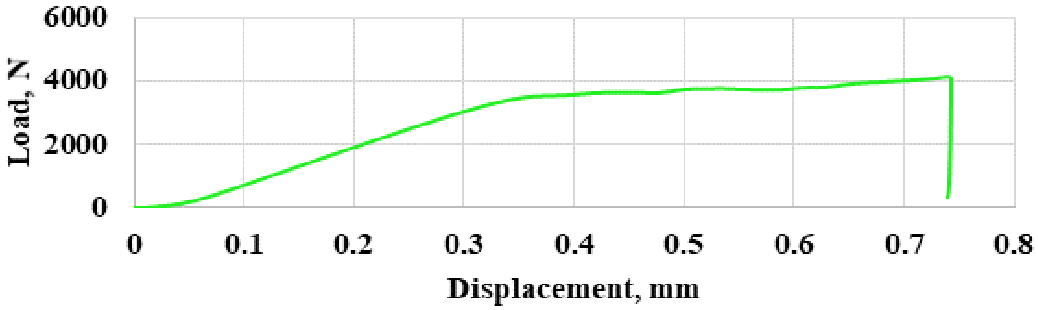

Figure 3.29. Diametral compression behavior of sample 4A7.

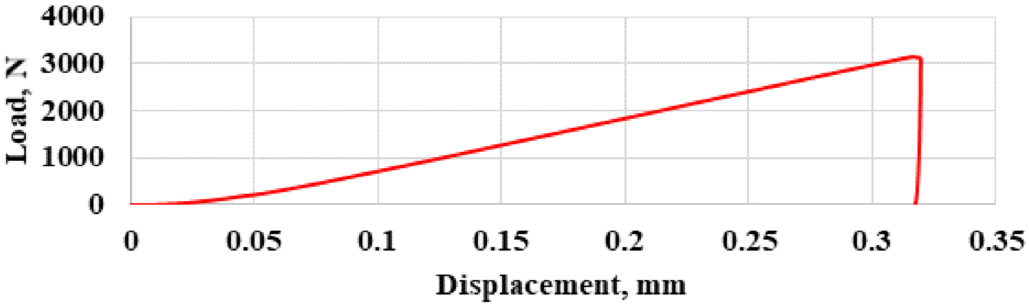

Figure 3.30. Diametral compression behavior of sample 2M7.

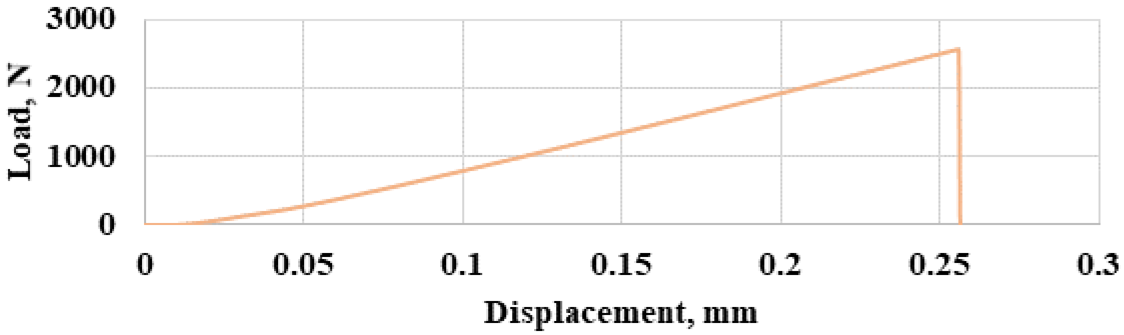

Figure 3.31. Diametral compression behavior of sample 4M7.

The comparison graphs between alumina and alumina with different addition of magnesia or nanoclay (Figures 3.32, 3.33, 3.34 and 3.35) exhibit the differences between the samples with the highest compressive strength and those with the minimum values. As can be seen almost all the samples exhibit typical brittle behaviour, without significant differences between their loading curve. However, in the case of 4A2 and 4A7 samples, it can be stated that, at the failure moment of 4A2 sample, the 4A7 sample continued to sustain its mechanical properties, in terms of compressive strength. Accordingly, its failure moment was mainly related with a higher displacement, while the load was almost the same.

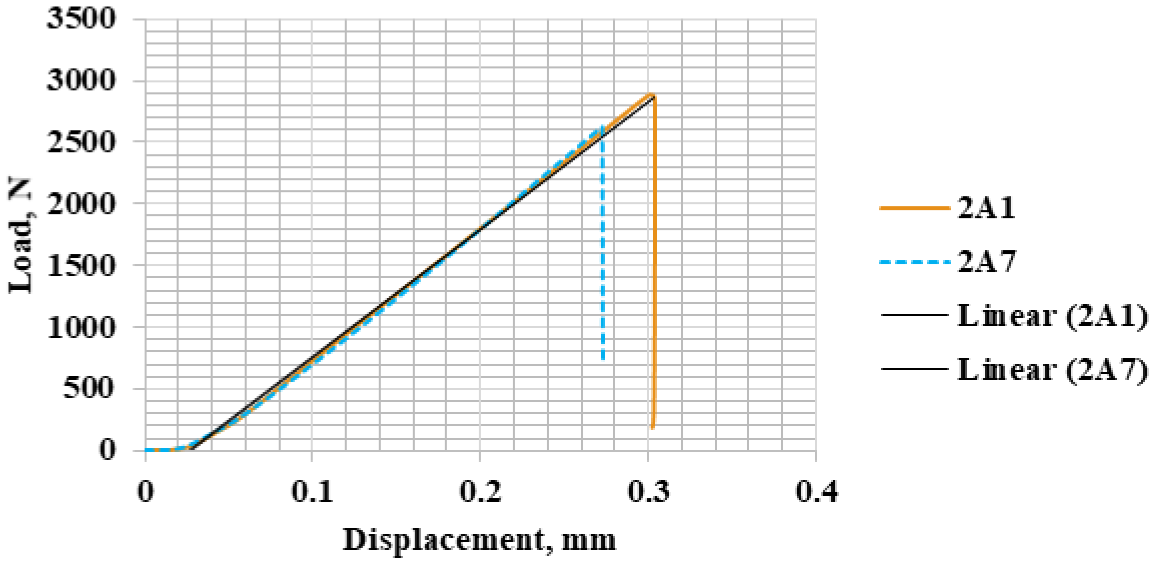

Figure 3.32 Comparison graph between diametral compression behavior of 2A7 and 2A1 samples.

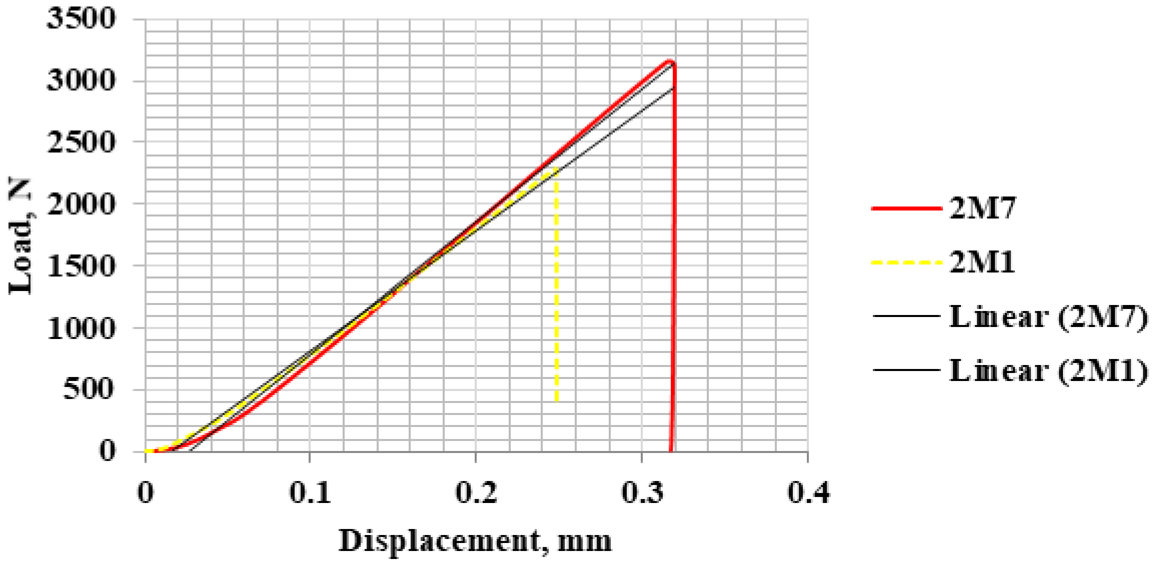

Figure 3.33. Comparison graph between diametral compression behavior of 2M1 and 2M7 samples.

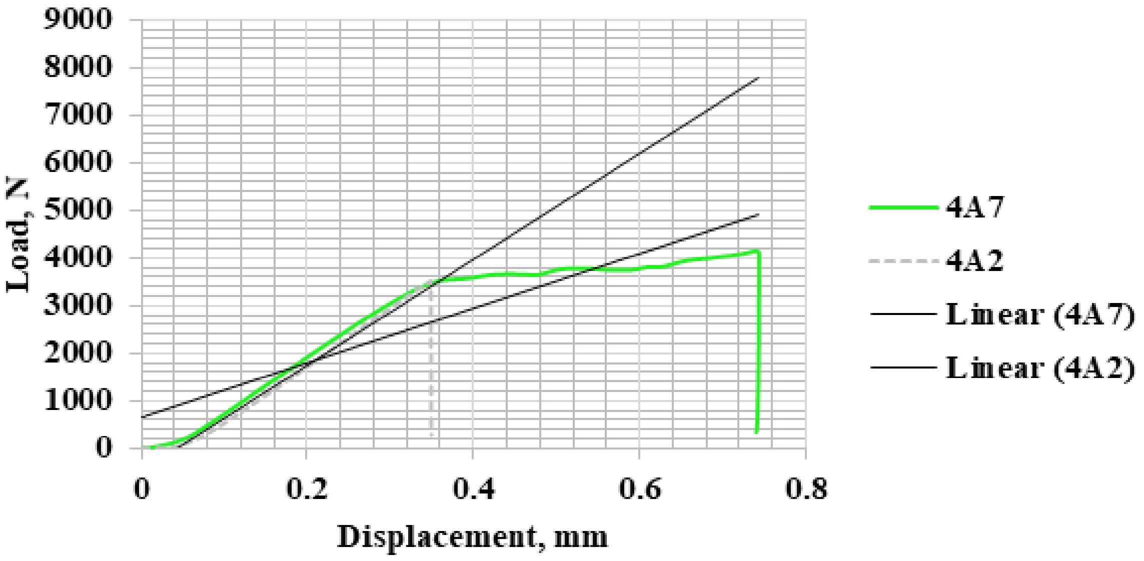

Figure 3.34. Comparison graph between diametral compression behavior of 4A2 and 4A7 samples.

The graph differences between 4M1 and 4M7 can be summarized as follows. In the case of 4M1 sample, the compressive test started with a small displacement during which the load didn't increase significantly, therefore, only few particles were presented on the sample surface which haven't been correspondingly cleaned. Therefore, if this part of the graph is removed, a clear overlapping between the 4M1 and 4M7 samples can be observed. Considering the small difference between the load values of these two samples, it can be stated that the mixture of alumina, magnesia and nanoclay was homogenous.

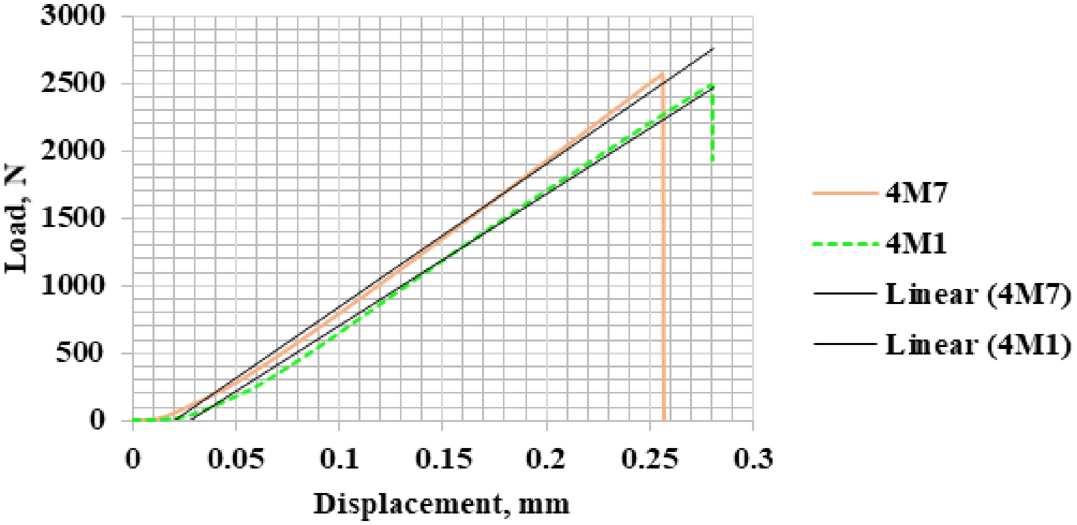

Figure 3.35. Comparison graph between diametral compression behavior of 4M1 and 4M7 samples.

3.3.6.3. Vickers hardness testing

Vickers hardness method was used to analyze the samples by means of Mitutoyo AVK-C2 equipment according to the procedure described previously.

The hardness values of sintered samples are shown in the Table 3.15.

Table 3.15. Hardness values of sintered samples according to Figure 3.27.

Sample	D1	D2	HV	(D1+D2)/2	HV, GPa	Mean HV, GPa	S, GPa
4A8	101.10	101.40	1808.00	101.25	17.73	18.43	0.23
	99.80	101.30	1834.00	100.55	17.97		
	104.00	104.80	1701.00	104.40	16.67		
	98.60	99.40	1892.00	99.00	18.54		
	94.30	95.80	2052.00	95.05	20.12		
	93.40	96.20	2063.00	94.80	20.22		
	101.40	102.50	1784.00	101.95	17.48		
	99.20	101.30	1845.00	100.25	18.08		
	101.00	100.30	1830.00	100.65	17.94		
	96.30	96.50	1995.00	96.40	19.56		
2M8	121.60	125.20	1218.00	123.40	11.93	13.64	0.57
	121.80	122.10	1247.00	121.95	12.22		
	108.50	108.60	1573.00	108.55	15.42		
	110.40	113.20	1483.00	111.80	14.54		
	108.90	107.60	1582.00	108.25	15.51		
	122.10	118.20	1284.00	120.15	12.59		
	118.80	121.10	1289.00	119.95	12.63		
	112.20	111.80	1478.00	112.00	14.49		
	114.00	115.60	1407.00	114.80	13.79		
	115.00	119.00	1354.00	117.00	13.28		
4M8	87.90	90.90	2320.00	89.40	22.74	20.35	0.80
	89.40	90.50	2291.00	89.95	22.46		
	92.30	93.30	2153.00	92.80	21.10		
	96.50	95.60	2010.00	96.05	19.70		
	96.60	94.50	2031.00	95.55	19.91		
	96.20	95.90	2010.00	96.05	19.70		
	96.90	96.10	1991.00	96.50	19.52		
	100.50	99.70	1850.00	100.10	18.14		
	93.70	95.20	2078.00	94.45	20.37		
	95.90	95.40	2026.00	95.65	19.86		
2A8	106.50	109.10	1525.00	107.80	15.64	14.92	0.24
	107.10	108.00	1603.00	107.55	15.71		
	104.80	107.30	1648.00	106.05	16.16		
	105.80	109.40	1601.00	107.60	15.70		
	108.20	111.00	1543.00	109.60	15.13		
	112.70	115.90	1419.00	114.30	13.91		
	114.90	118.30	1364.00	116.60	13.37		
	111.80	109.80	1510.00	110.80	14.80		
	113.70	114.90	1419.00	114.30	13.91		
	106.60	108.40	1604.00	107.50	15.73		

3.4. Conclusions

3.4.1. The samples with alumina and nanoclay addition

The diametral compression results of the alumina samples with 2% or 4% nanoclay addition are summarized in Table 3.16.

Table 3.16. Experimental results on compressive strength of alumina samples.

Sample	Composition	Load, N	Average, N	Compressive strength, MPa	Average, MPa
A5	Alumina	2574.39	**2492.46**	19.55	**19.13**
2A1	Alumina with 2 %, wt. nanoclay	2888.51	**3001.90**	25.42	**26.64**
4A2	Alumina with 4 %, wt. nanoclay	3516.35	**3409.49**	31.49	**30.51**

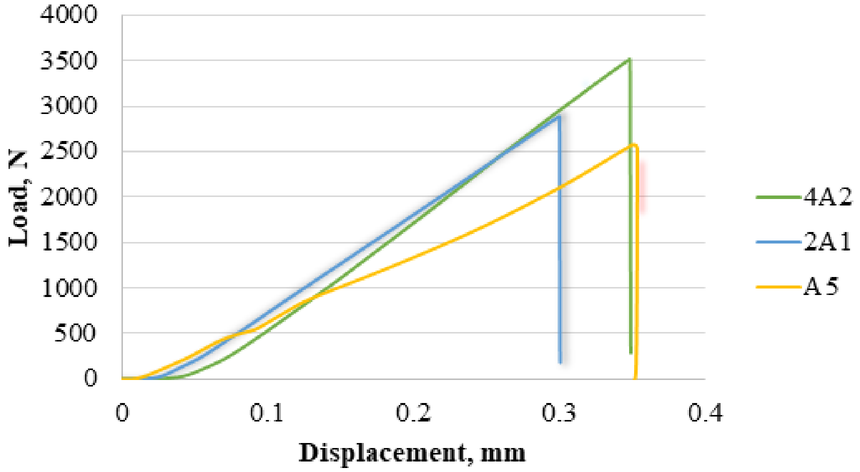

Figure 3.36. Diametral compression strength of alumina samples with nanoclay addition.

As can be observed from Figure 3.36 the compressive strength of alumina increases with increasing percentage of nanoclay added into alumina matrix.

The hardness values of alumina samples are presented in the Table 3.17.

Table 3.17. Hardness value of alumina samples.

	Proba A7	Proba 2A6	Proba 4A6
Maximum	12.62	15.45	20.99
Mean	11.89	14.27	18.78
Minimum	10.85	12.52	16.83

3.4.2. Conclusions on alumina samples with magnesium oxide

The diametral compression of the alumina samples with magnesium oxide is presented in Table 3.18.

Table 3.18. Experimental results on compressive strength of alumina samples + 1% magnesium oxide.

Sample	Composition	Maximum load, N	Mean	Compressive strength, MPa	Mean
AM6	Alumina + 1% MgO	1948.99	1935.45	15.27	15.14
2M1	Alumina + 1% MgO + 2% nanoclay	2287.05	2785.52	19.62	23.85
4M1	Alumina + 1% MgO + 4% nanoclay	2481.91	2855.09	23.05	25.23

It is observed that the compressive strength of alumina + 1% MgO increases with increasing percentage of added nanoclay.

The diametral compression test values of alumina samples + 1% magnesium oxide are presented in Figure 3.37. Also, the values of hardness for same samples are shown in Table 3.19.

Table 3.19. Hardness values for alumina samples with 1% MgO addition.

	Proba AM9	Proba 2M6	Proba 4M6
Maximum, HV	14.45	16.04	21.28
Mean, HV	11.88	14.59	20.41
Minimum, HV	10.85	13.08	19.18

The hardness value is increased by the addition of nanoclay.

From the analysis of the experimental data there is a decrease of the mechanical resistance to compression and an increase of the hardness by the addition of 1% MgO.

Moreover, if we define the relative increase of a property as the ratio between the value obtained for the composite with MgO and the value obtained for the composite without MgO, accordingly, the MgO addition influences the mechanical properties of alumina-based composites.

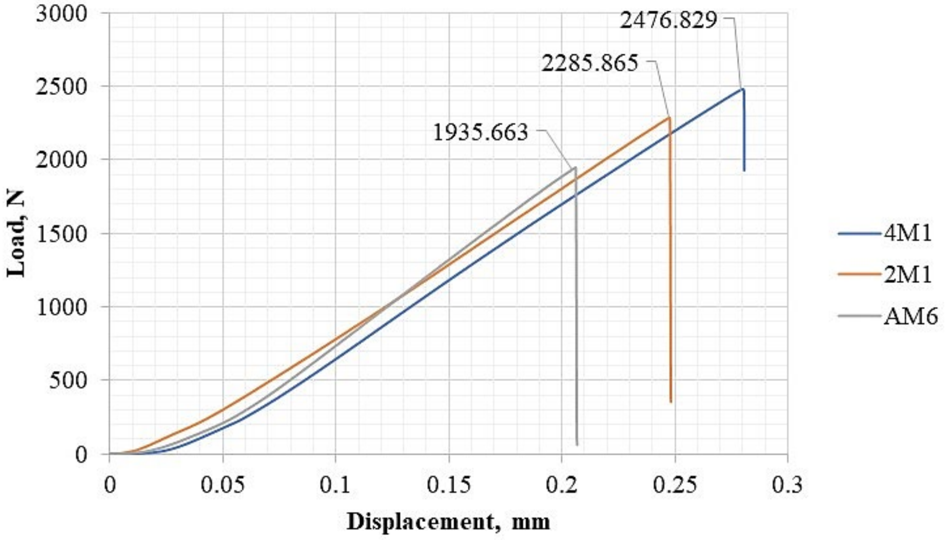

Figure 3.37. Diametral compression test.

The degree of hardness increase is observed as well as the way in which the compressive strength decreases. For example, for the 2% nanoclay composite, the hardness increases by 2% and the compressive strength decreases by 23% only by the addition of 1% MgO.

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Appendix 1. Samples and their characteristics

Sample	Composition	Compressive strength MPa	Hardness, HV	Density, g/cm^3
A1	m=2.5 g; Al_2O_3	7.88		2.78
A2	m=2.5 g; Al_2O_3	16.09		2.79
A3	m=2.5 g; Al_2O_3	27.18		2.81
A4	m=2.5 g; Al_2O_3	18.55		2.86
A5	m=2.5 g; Al_2O_3	19.55		2.71
A6	m=2.5 g; Al_2O_3	25.54		2.85
A7	m=2.5 g; Al_2O_3		11.88	
A8	m=2.5 g; Al_2O_3		12.32	
AM1	99% Al_2O_3 and 1% magnesia	10.21		2.77
AM2	99% Al_2O_3 and 1% magnesia	17.13		2.81
AM3	99% Al_2O_3 and 1% magnesia	18.12		2.81
AM4	99% Al_2O_3 and 1% magnesia	14.91		2.81
AM5	99% Al_2O_3 and 1% magnesia	16.19		2.81
AM6	99% Al_2O_3 and 1% magnesia	15.27		2.82
AM7	99% Al_2O_3 and 1% magnesia	15.99		2.80
AM8	99% Al_2O_3 and 1% magnesia	13.34		2.83
AM9	99% Al_2O_3 and 1% magnesia		12.39	
AM10	99% Al_2O_3 and 1% magnesia		11.46	
M1	m=2.5 g; 71.8% Al_2O_3 and 28.2% silica	5.94		2.01
M2	m=2.5 g; 71.8% Al_2O_3 and 28.2% silica	4.02		2.02
M3	m=2.5 g; 71.8% Al_2O_3 and 28.2% silica	3.66		2.07
M4	m=2.5 g; 71.8% Al_2O_3 and 28.2% silica	5.23		2.03
M5	m=2.5 g; 71.8% Al_2O_3 and 28.2% silica	4.76		1.95

M6	m=2.5 g; 71.8% Al_2O_3 and 28.2% silica	4.18		1.95
M7	m=2.5 g; 71.8% Al_2O_3 and 28.2% silica	5.48		1.99
M9	m=2.5 g; 71.8% Al_2O_3 and 28.2% silica	4.26		2.01
M11	m=2.5 g; 71.8% Al_2O_3 and 28.2% silica		6.53	
M8	m=2.5 g; 71.8% Al_2O_3 and 28.2% silica		3.11	
MM2	71.8% Al_2O_3 + 28.2% silica +1% MgO	2.76		1.79
MM3	71.8% Al_2O_3 + 28.2% silica +1% MgO	9.21		1.83
MM4	71.8% Al_2O_3 + 28.2% silica +1% MgO	3.64		1.95
MM5	71.8% Al_2O_3 + 28.2% silica +1% MgO	4.08		1.95
MM6	71.8% Al_2O_3 + 28.2% silica +1% MgO	4.09		1.89
MM7	71.8% Al_2O_3 + 28.2% silica +1% MgO	8.43		1.94
MM8	71.8% Al_2O_3 + 28.2% silica +1% MgO	4.05		2.02
MM9	71.8% Al_2O_3 + 28.2% silica +1% MgO	3.93		1.91
MM10	71.8% Al_2O_3 + 28.2% silica +1% MgO	2.53		1.92
MM12	71.8% Al_2O_3 + 28.2% silica +1% MgO	5.15		1.93
MM11	71.8% Al_2O_3 + 28.2% silica +1% MgO		5.20	
MM13	71.8% Al_2O_3 + 28.2% silica +1% MgO		1.94	
4M1	Al_2O_3 + 1% MgO + 4% nanoclay, m=2.5 g	23.05		3.15

4M2	Al_2O_3 + 1% MgO + 4% nanoclay, m=2.5 g	31.32		3.04
4M3	Al_2O_3 + 1% MgO + 4% nanoclay, m=2.5 g	21.50		3.14
4M4	Al_2O_3 + 1% MgO + 4% nanoclay, m=2.5 g	20.66		3.10
4M5	Al_2O_3 + 1% MgO + 4% nanoclay, m=2.5 g	29.59		3.03
4M6	Al_2O_3 + 1% MgO + 4% nanoclay, m=2.5 g		20.41	3.05
2M1	Al_2O_3 + 1% MgO + 2% nanoclay, m=2.5 g	19.62		2.98
2M2	Al_2O_3 + 1% MgO + 2% nanoclay, m=2.5 g	32.60		3.00
2M3	Al_2O_3 + 1% MgO + 2% nanoclay, m=2.5 g	16.58		2.99
2M4	Al_2O_3 + 1% MgO + 2% nanoclay, m=2.5 g	19.31		2.96
2M5	Al_2O_3 + 1% MgO + 2% nanoclay, m=2.5 g	31.12		2.89
2M6	Al_2O_3 + 1% MgO + 2% nanoclay, m=2.5 g		14.59	3.00
2A1	Al_2O_3 + 2% nanoclay, m=2.5 g	25.42		3.00
2A2	Al_2O_3 + 2% nanoclay, m=2.5 g	32.10		2.96
2A3	Al_2O_3 + 2% nanoclay, m=2.5 g	16.27		3.04
2A4	Al_2O_3 + 2% nanoclay, m=2.5 g	30.06		2.95
2A5	Al_2O_3 + 2% nanoclay, m=2.5 g	29.35		3.01
2A6	Al_2O_3 + 2% nanoclay, m=2.5 g		14.27	2.92
4A1	Al_2O_3 + 4% nanoclay, m=2.5 g	21.03		3.05
4A2	Al_2O_3 + 4% nanoclay, m=2.5 g	31.49		3.04
4A3	Al_2O_3 + 4% nanoclay, m=2.5 g	28.27		3.01
4A4	Al_2O_3 + 4% nanoclay, m=2.5 g	33.12		3.06
4A5	Al_2O_3 + 4% nanoclay, m=2.5 g	38.63		2.78
4A6	Al_2O_3 + 4% nanoclay, m=2.5 g		18.78	3.03

4M7	Al_2O_3 + 1% MgO + 4% nanoclay, m=2.5 g, the second sintering process	19.58		
4M8	Al_2O_3 + 1% MgO + 4% nanoclay, m=2.5 g, the second sintering process		20.35	2.91
2M7	Al_2O_3 + 1% MgO + 2% nanoclay, m=2.5 g, the second sintering process	27.37		2.90
2M8	Al_2O_3 + 1% MgO + 2% nanoclay, m= 2.5 g, the second sintering process		13.64	2.83
4A7	Al_2O_3 + 4% nanoclay, m= 2.5 g, the second sintering process	35.93		2.82
4A8	Al_2O_3 + 4% nanoclay, m= 2.5 g, the second sintering process		18.43	2.88
2A7	Al_2O_3 + 2% nanoclay, m= 2.5 g, the second sintering process	22.59		3.14
2A8	Al_2O_3 +2% nanoclay, m= 2.5 g, the second sintering process		14.92	2.83

Chapter 4

Experimental Investigations in Composites Improvement

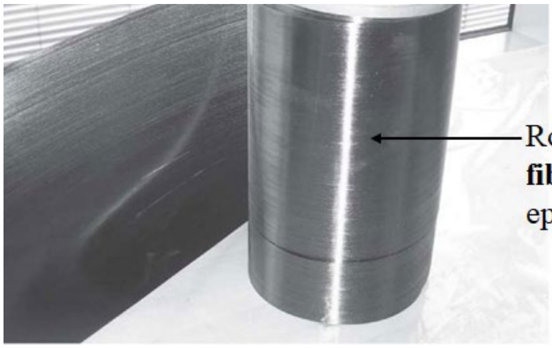
Further an experimental investigation on carbon fiber composites improvement was design and realized. Accordingly, the flexural strength of composites with sandwich structure was analysed.

4.1. Materials

The tests were performed on TEXIPREG® HS 110 REC made of carbon fibers with epoxy resin matrix. The epoxy matrix carbon fiber roll is kept at low temperatures so that the resin does not lose its properties, as it does not have a stable chemical structure. The roll is covered on one side with a layer of paper to protect it from impurities on the work table [1].

Storage: The roller is stored at temperatures below or equal to -18 °C, sealed in the original packaging. It can be handled to create samples at temperatures up to 20 °C.

Before using the prepreg roll, remove the roll from the freezer and allow it to warm to room temperature for 6 hours, sealed in the original package, then open.

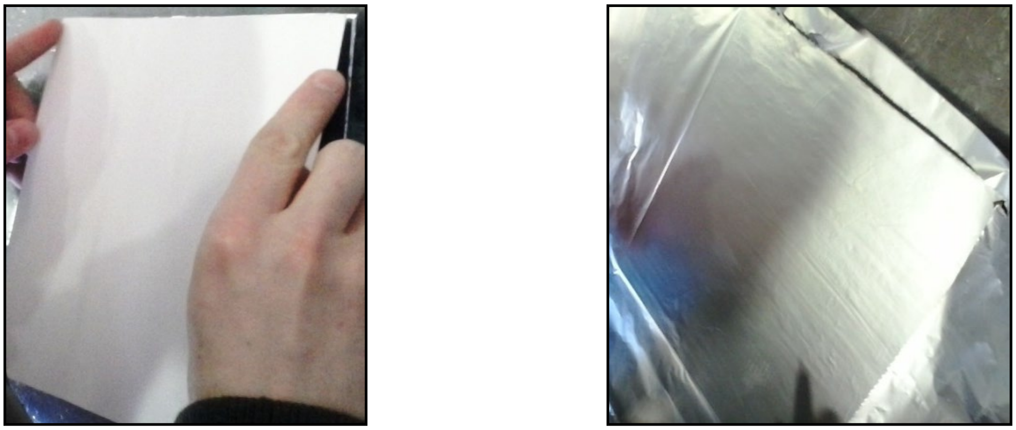


Roll of unidirectional **carbon fibers** pre-impregnated in epoxy resin

Figure 4.1. Pre-impregnated carbon/epoxy roll.

To perform the test, 10 units of epoxy resin 520 and one unit of epoxy hardener 523 are required, which are also stored in the refrigerator. The pre-impregnated carbon / epoxy roll must be cut into pieces of 200x200 mm (Figure 4.1), with the orientation of the carbon fibers at 0°and 90°, respectively.

Then, the pieces are glued together, the difference between the layers will be only the orientation of the fibers, because they must be glued, in pairs, at 0° and 90° respectively. Two samples are performed for this experiment (Figure 4.2).



a) b)

Figure 4.2. Samples manufacturing, layers positioning: a) sample containing pre-impregnated carbon/epoxy layers; b) sample containing pre-impregnated carbon/epoxy layers and aluminum layers.

The first sample is composed only of pre-impregnated carbon / epoxy layers, and to the second sample are added aluminum layers.

Properties of carbon / epoxy roller- TEXIPREG® HS 110 REC

High-strength carbon fibers pre-impregnated (pre-preg) with REC-modified epoxy resin, suitable for both compression and vacuum molding. They adhere directly to honeycomb materials and do not require any additional adhesive film for sandwich molding.

It is widely used in the manufacture of fishing gear and sports equipment. The pre-preg is a high resistance T300 carbon fibers unidirectional orientated at 0°, with a weight of 110 g/m2 in dry state mixed with a resin. The resin is a type REC epoxy, with 12 minutes setting time at 125 °C. The system fiber—resin (pre-impregnated) consists of 32 % by mass of resin, with a specific weight of 162 g/m2 and loss on ignition lower than 1% at 150 °C exposed for 10 minutes, also, it has an average viscosity, with a durability of 30 days. After 60 minutes pressing at 5 bars and 125 °C, the created pre-preg exhibits the following physical and mechanical properties. According to ASTM D792 standard, it has a 1.52 g/cm2 specific weight, at a 53% by volume content of carbon fibers. The roll layer has a 0.12 mm thickness, while the tensile strength exhibited reaches 1700 MPa, when tested according to ASTM D3039 standard. According to the same standard requirements, the

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composite elongation is 1.3 %, while the modulus of elasticity is 150 GPa [1]. Tested according to ASTM D790 (L/d=16) standard, the bending strength reaches 1400 MPa, and the bending modulus is 130 GPa. Its shear strength is 60 MPa, tested according to ASTM D2344. However, since the operating conditions and the experimental set-up parameters have high influence on the final characteristics of this type of composites, the values are only informative, they can be strongly affected by the process conditions. In order to obtain exact values regarding the properties of the manufactured composite, specific tests must be performed on it.

4.2. Methods

The composites have been manufactured through the vacuum infusion method. This method consists of obtaining high quality composites by inserting the resin and reinforcing it on an open mold with the help of a vacuum pressure.

This pressure acts on the layers by pushing the resin, ensuring a fast and complete impregnation of the industrial fabrics. As can be seen from Figure 4.3 the composite is positioned on a porous layer, which can be easily peeled after composite setting and assure a clear passing of the resin excess to the absorbent material that will capture the unnecessary amount of resin.

The resin excess is removed from the composite due to the vacuum pressure, that will squeeze the layers together through the vacuum bag, which will easily copy the form of the composite, while the composite is positioned on a component with the desired shape of the final product.

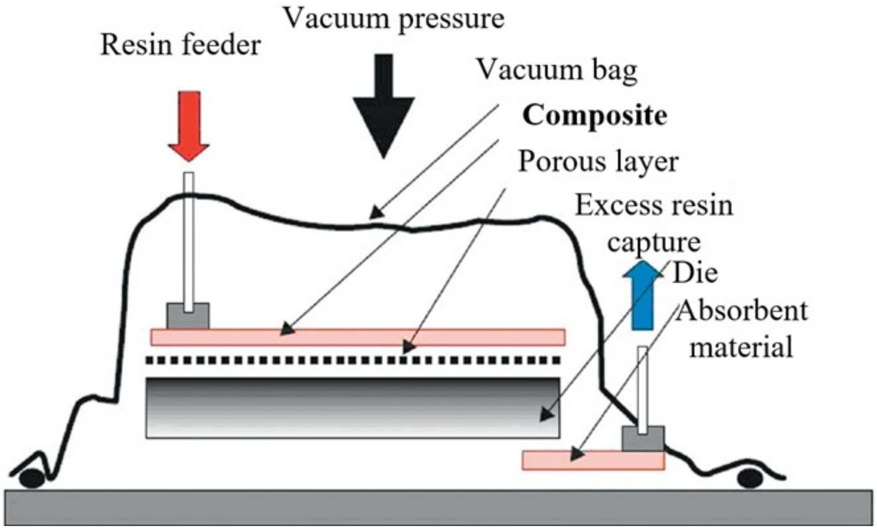
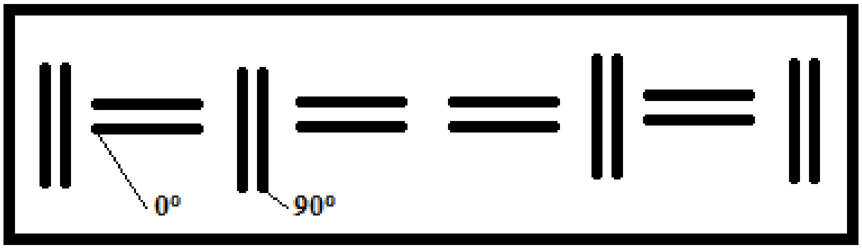
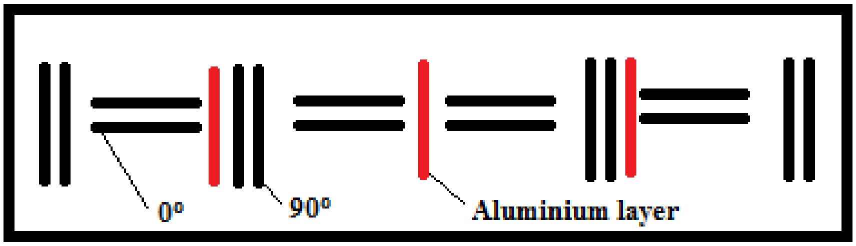


Figure 4.3. Schematic representation of composites manufacturing by vacuum infusion method [2].

Figure 4.4.a and Figure 4.4 b show the orientation of the fibers in the layers that make up the two samples.



a) $[(90^\circ)_2(0^\circ)_2(90^\circ)_2(0^\circ)_2(0^\circ)_2(90^\circ)_2(0^\circ)_2(90^\circ)_2]$



b) $[(90^\circ)_2(0^\circ)_2(Al)_1(90^\circ)_2(0^\circ)_2(Al)_1(0^\circ)_2(90^\circ)_2(Al)_1(0^\circ)_2(90^\circ)_2]$

Figure 4.4. Orientation of fibers in the sample layers: a) sample from pre-impregnated carbon/epoxy layers; b) sample from pre-impregnated carbon / epoxy layers and aluminum layers.

After laying the layers (Figure 4.2), the laminate is placed in a bag composed of two lower layers, the first of high temperature resistant plastic and the second of non-adhesive material so it will not stick to the laminate, it can be easily removed at the end of the manufacturing process (Figure 4.5).

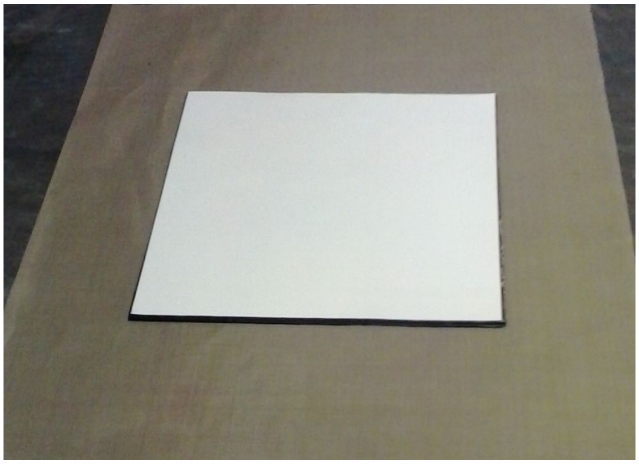


Figure 4.5. Laying the semi-finished product on the two layers.

Because during the manufacturing process the excess resin will come out of the carbon fibers, the laminate is surrounded by an absorbent material composed of synthetic fibers, fixed with high temperature resistant adhesive tape (Figure 4.6), after which they are covered with two layers, they being of the same materials as the first two but placed in reverse order. Through the second layer of plastic foil a hole is made for the insertion of the part which will be subsequently connected to the vacuum pump.

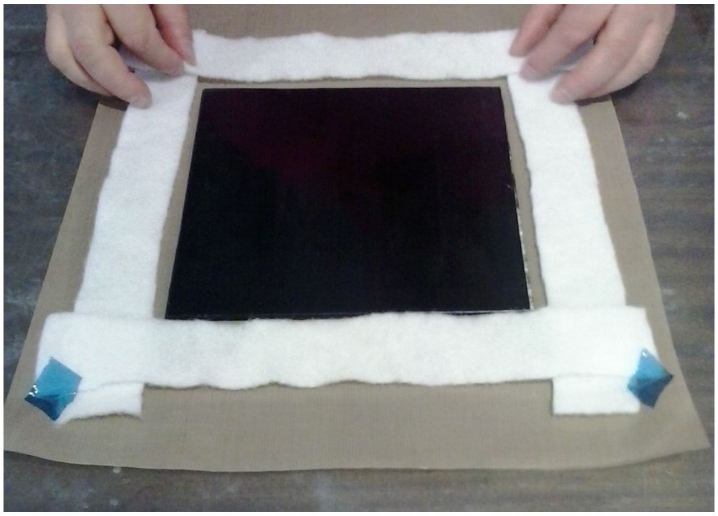


Figure 4.6. Laying and fixing the porous material.

In order for the bag to be vacuumed, the two layers of plastic are glued together (Figure 4.7).

Figure 4.7. Sealing the bag and fixing the upper component of the previously inserted part.

The bag is then autoclaved (Figure 4.8) and connected to the vacuum pump via the previously inserted part.

Figure 4.8. Autoclave.

Then, a vacuum pressure of 0.5 bar will be applied to remove the air from the bag (Figure 4.9).

Figure 4.9. Removing air from the bag.

Subsequently, the autoclave will close (Figure 4.10) forming a temperature of 125 ° C in the enclosure with a heating rate of 3-5 ° C / min (Figure 4.11), then a pressure of 2-7 bar will be applied on the bag for 90 minutes. After 90 minutes, the machine will be brought to room temperature.

Figure 4.10. Closing the autoclave.

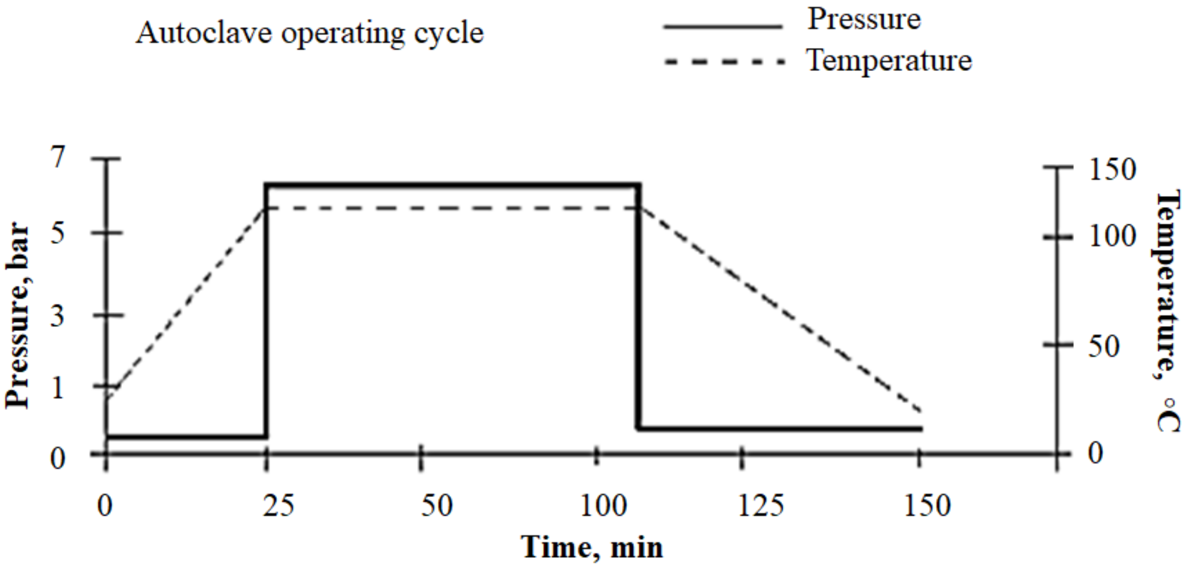


Figure 4.11. Autoclave operating cycle.

After completion of the manufacturing cycle the sample can be removed from the machine (Figure 4.12) and subjected to tests without requiring prior treatment.

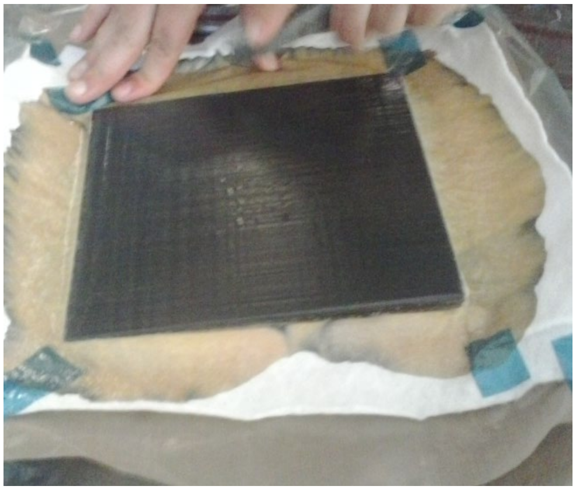


Figure 4.12. The carbon fiber composite after curing.

Sample handling suggestions:

- Handle the sample only with gloves.
- Store the sample in ventilated and dust-free rooms.

4.3. Samples testing

The samples were cut into pieces of different sizes depending on the test that will be performed on it, as follows:

Pre-engaged carbon / epoxy laminate.

No holes:

- Sample T1: width 23.8 mm, thickness 2.2 mm;
- Sample T2: width 23.1 mm, thickness 2.3 mm;
- Sample T3: width 23.6 mm, thickness 2.2 mm.

With hole in the middle of the sample (hole diameter is 5 mm):

- Sample H1: width 27.9-5 = 22.9 mm, thickness 2.4 mm;
- Sample H2: width 26.0-5 = 21.0 mm, thickness 2.4 mm.

Laminate from pre-bonded carbon / epoxy layers and aluminum layers.

No holes:

- Sample A1: width 22.4 mm, thickness 2.3 mm;
- Sample A2: width 23.1 mm, thickness 2.4 mm;
- Sample A3: width 23.4 mm, thickness 2.4 mm.

With hole in the middle of the sample (hole diameter is 5 mm):

- Sample AH1: width 23.9-5 = 18.9 mm, thickness 2.5 mm;
- AH2 sample: width 22.2-5 = 19.2 mm, thickness 2.5 mm.

Since this type of composites are mainly used in aircraft manufacturing. The aim of performing tests on samples with holes is to simulate the behaviour of products with different types of damages. In this case composites with round damages (5 mm hole).

Bending strength of the obtained composites

The samples were subjected to bending using the Instron 1341 machine (Figure 4.13) under normal environmental conditions, the distance between the supports being 40 mm, according to ASTM D790 and the hammer velocity being 2 mm/min.

Figure 4.13. Instron 1341.

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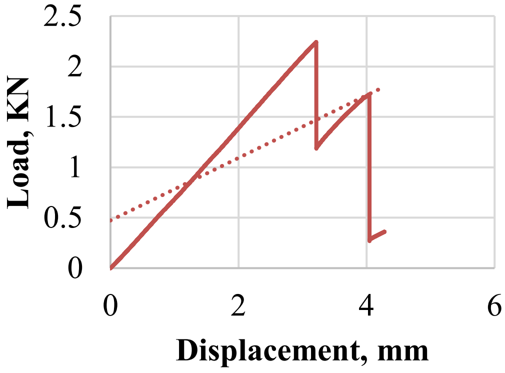

Figure 4.14. Load/displacement graphic of T1 sample.

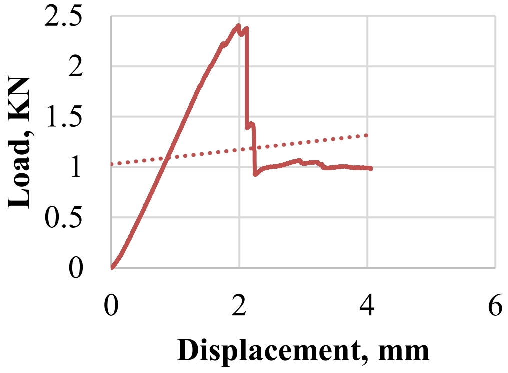

Figure 4.15. Load/displacement graphic of T2 sample.

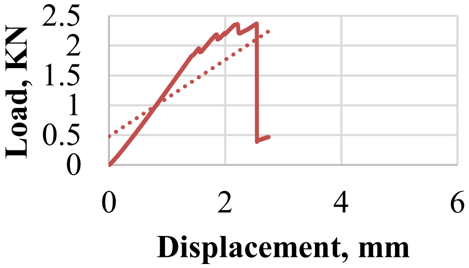

Figure 4.16. Load/displacement graphic of T3 sample.

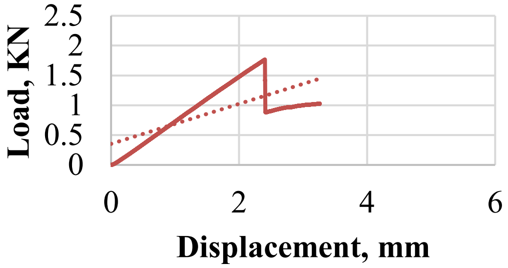

Figure 4.17. Load/displacement graphic of Sample H1.

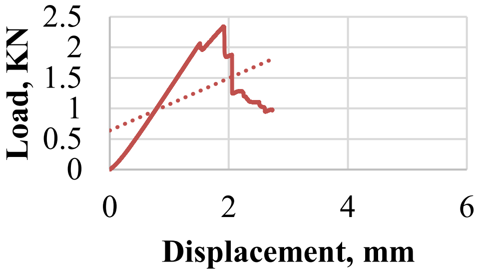

Figure 4.18. Load/displacement graphic of H2 sample.

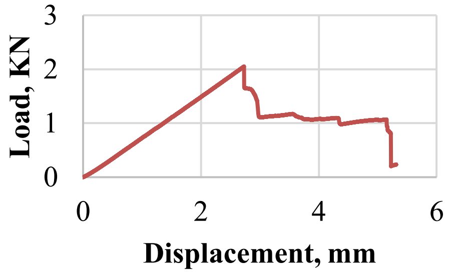

Figure 4.19. Load/displacement graphic of A1 sample.

Figure 4.20. Load/displacement graphic of A2 sample.

Figure 4.21. Load/displacement graphic of A3 sample.

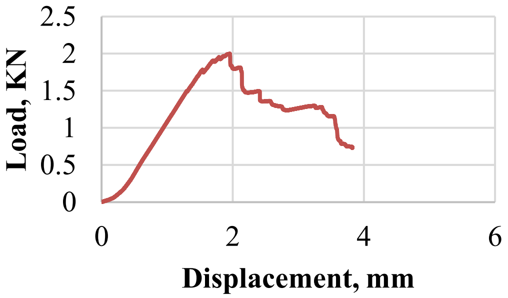

Figure. 4.22. Load/displacement graphic of AH1 sample.

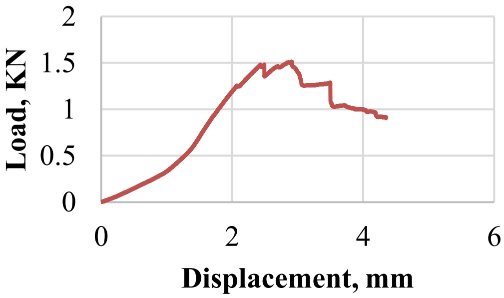

Figure 4.23. Load/displacement graphic of AH2 sample.

The effects of the hole or the aluminum layer can be highlighted using the following graphs:

The value of bending strength was calculated using the following relation (eq. 4.1):

$$6 = \frac{3 \cdot F \cdot 40}{2 \cdot b \cdot g^2} \tag{4.1}$$

Where: F– force; b - width [mm]; g - thickness [mm].

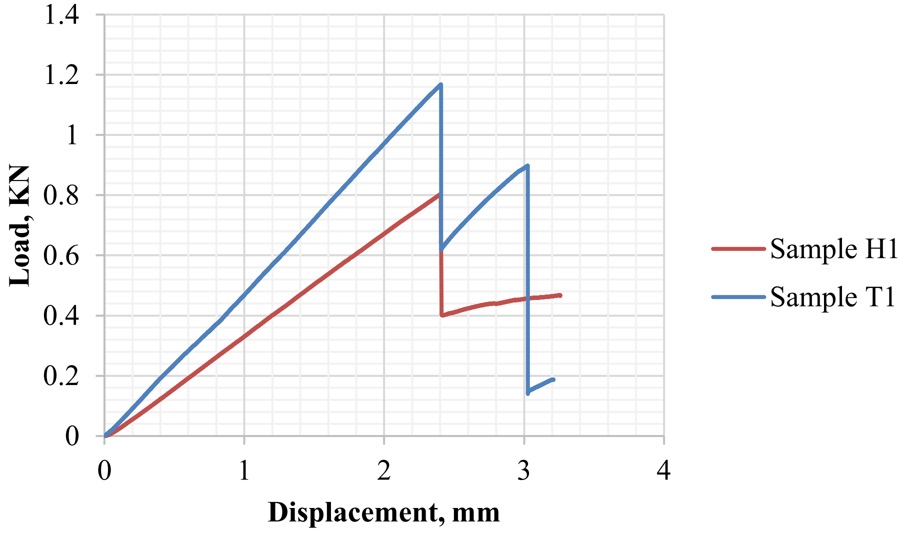

Figure 4.24. Load/displacement graph comparison between T1 and H1 samples.

Figure 4.24 shows that the orifice sample has lower bending strength because the hole decreases the usable area of the sample and cancels the strength of the fibers once sectioned.

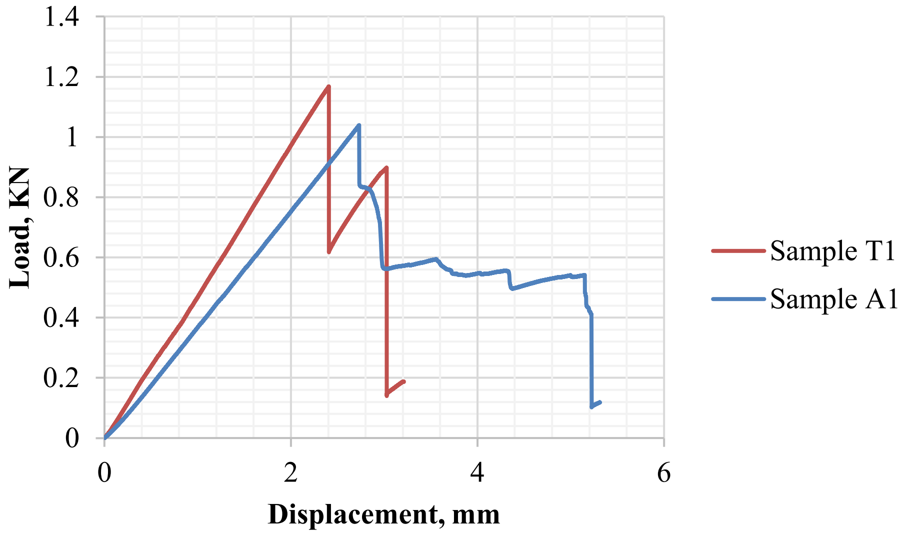

Figure 4.25. Load/displacement graph comparison between T1 and A1 samples.

Figures 4.25 and 4.26 show that samples with aluminum layers have a lower value of maximum tensile strength than samples with impregnated carbon / epoxy layers only, but it can also be seen that it is more tenacious than other.

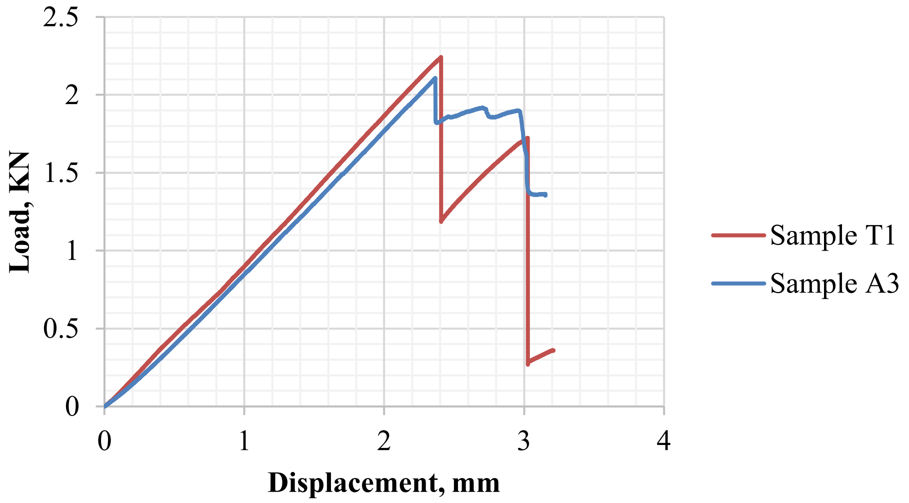

Figure 4.26. Load/displacement graph comparison between T1 and A3 samples.

The bending strength of the samples is shown in the Tables 4.1and 4.2.

Table 4.1. Bending resistance of samples without holes.

Material	**Bending strength [MPa]**	**Average value of bending strength [MPa]**	**Deviation [MPa]**
Carbon / epoxy laminates	1.17	1.20	0.04
	1.18		
	1.24		
Fiber / metal laminates	1.04	1.06	0.14
	1.21		
	0.94		

Table 4.2. Bending strength of hole samples.

Material	**Bending strength [MPa]**	**Average value of bending strength [MPa]**	**Deviation [MPa]**
Carbon / epoxy laminates with hole	0.80	0.98	0.25
	1.16		
Laminated fiber / metal with hole	1.02	0.89	0.18
	0.76		

After the test (Figures 4.28 and 4.30), with the help of a microscope, we can observe the effect of bending the samples (Figures 4.27 and 4.29).

Figure 4.27. Carbon / epoxy laminate after test.

Figure 4.28. Carbon / epoxy hole laminate after test.

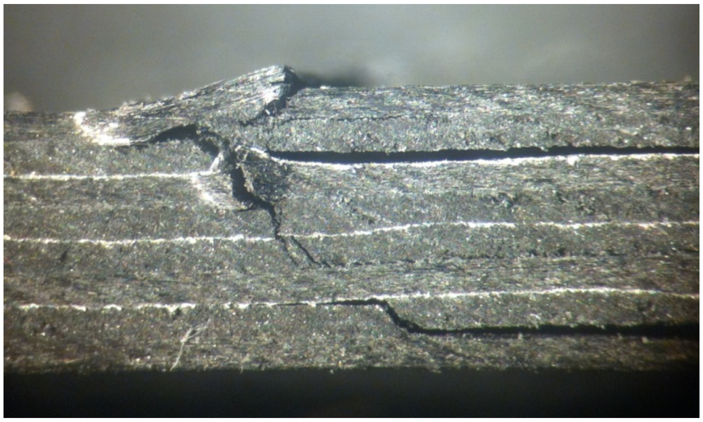

Figure 4.29. Laminate with aluminum layers after the test.

Figure 4.30. Laminate from fiber layers and aluminum layers after test.

In laminates with aluminum layers, it can be seen that the crack propagates along the aluminum layer, resulting in the application of aluminum layers is a disadvantage for this type of laminate.

4.4. Conclusions

The concept of composite material allows the new one to be strictly directed towards the expected results, as well as the creation of materials with certain imposed properties, so that the technical parameters of an element are satisfied by the qualities of a special material created for it.

Composite materials are of high interest and have extensive applications in industry. This topic was discussed in the paper, both in terms of fabrication and bending deformation.

In this chapter, several types of composites based on carbon fiber have been studied, to which aluminum foil has been added.

Thus, 4 types of composite test specimens were studied, as follows:

- Pre-engaged carbon / epoxy laminate;
- Pre-engaged carbon / epoxy laminate with 5 mm hole in the middle of the sample;
- Pre-bonded carbon / epoxy laminate and aluminum coating;
- Laminated from pre-engaged carbon / epoxy layers and aluminum layers with 5 mm hole in the middle of the sample.

Table 4.3. Synthesis of experimental data on comparison between tests.

Sample	Bending strength [KN/mm^2]	Observations	
T1	1.17	No holes	Pre-engaged carbon / epoxy laminate
T2	1.18		
T3	1.24		
H1	0.80	With hole (d = 5 mm) in the middle of the sample	
H2	1.16		
A1	1.04	No holes	Laminated from pre-bonded carbon / epoxy layers and aluminum layers
A2	1.21		
A3	0.94		
AH1	1.02	With hole (d = 5 mm) in the middle of the sample	
AH2	0.76		

From the analysis of Table 4.3 the following conclusions can be highlighted:

- by introducing aluminum layers into the laminated composite made of pre-bonded carbon / epoxy layers, the maximum bending stress decreases on average by 11.67%;
- for hole samples: by inserting the maximum bending stress into the laminated composite made of pre-bonded carbon / epoxy layers, the maximum bending stress decreases on average by 9.18%;
- by drilling holes in the laminated composite made of pre-engaged carbon / epoxy layers, the maximum bending stress decreases on average by 18.33%;

- by drilling holes in the laminated composite made of pre-engaged carbon / epoxy layers, the maximum bending stress decreases on average by 16.04%.

Moreover, maximum results were obtained for samples T3, H2, A2 and AH1. If these samples are compared, the maximum value was obtained for the T3 sample, now seen in Table 4.2.

Table 4.2. Synthesis of experimental data: maximum values.

Sample	**Bending strength [KN/mm^2]**	**Observations**	
T3	1.243857	**No holes**	Pre-engaged carbon / epoxy laminate
H2	1.162474	With hole (d = 5 mm) in the middle of the sample	
A2	1.208278	No holes	Laminated from pre-bonded carbon / epoxy layers and aluminum layers
AH1	1.016616	With hole (d = 5 mm) in the middle of the sample	

Under these conditions, by drilling the 5 mm hole, the resistance decreases by 6.45%, by introducing Aluminum, it decreases by 4%. The lowest value for bending strength is obtained for sample AH1 (resistance decreases by 17.74% compared to sample T3).

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