

Politecnico di Milano
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Master of Science in Material Engineering and Nanotechnology

DESIGN AND MANUFACTURING OF A BIOINSPIRED COMPOSITE TUBE

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Abstract

The project at the base of this thesis' work was developed during an eight months internship which took place at the Institute of Polymer Materials and Plastic Technology in Clausthal-Zellerfeld, Germany. The project started with the goal of realizing a composite tube that could provide good performance under the loading condition of compression. A first design phase was carried out with the purpose of identifying an internal arrangement of fibres that could enhance the compression performance of the desired material. A structure selected was composed of zero degree tubular bundles of fibre, supposed to mimic the osteonic structure typical of the cortical bone. Once identified the desired internal arrangement of fibre, the focus was moved onto how to produce such tube. A considerable amount of time was dedicated to the development of a manufacturing technique that would enable the realization of such composite. For this purpose, tubes with natural fibres were produced with the aim of developing the technique, evaluating the results and its repeatability. The material from such tubes has been compared in compression and in fibre volume content to the one obtained from the filament winding technique. Once evaluated the characteristics of the tube to obtain and how to do it, a production phase followed and resulted in the realization of three types of artificial fibre composite tubes, two bioinspired and a comparative one. Specimens extracted from the material of such tubes were tested in compression to evaluate the response of the material subjected to such load. The materials produced were further compared by taking into consideration also the fibre volume content, which is an important parameter that needs to be considered. After the data elaboration, an improvement of the material's property was observed which confirmed the effectiveness of the chosen internal structure compare to the usual laminate composite. In the development of this thesis, a full description of the manufacturing process, the reasons for the selection of the structure, the production of the artificial fibre composite tube and the characterization of the material associated will be presented and analysed.

Sommario

Il progetto alla base di questa tesi è stato sviluppato durante uno stage di 8 mesi presso L'istituto di Materiali Polimerici e Tecnologie Plastiche di Clausthal-Zellerfeld, situato nella regione della Bassa Sassonia in Germania. Il progetto cominciò con l'obbiettivo di realizzare un tubo composito che potesse dare buone prestazioni quando sottoposto a sforzi in compressione. Una prima fase di design è stata affrontata con l'obbiettivo di identificare una organizzazione interna delle fibre che potesse andare ad aumentare le performance del materiale in compressione rispetto a compositi generalmente prodotti. Una struttura composta da tubicini di fibre di carbonio intrecciate e riempiti da fibre di vetro unidirezionali, pensata per rappresentare la disposizione osteonica caratteristica dell'osso corticale, è stata selezionata da lavori precedenti condotti da un gruppo di ricerca del dipartimento di Meccanica del Politecnico di Milano. Una volta identificata la disposizione delle fibre, l'attenzione si è postata su come produrre tale tubo composito. Una notevole quantità di tempo è stata dedicata allo sviluppo di una tecnica di produzione che permettesse di realizzare tale materiale con la struttura interna desiderata. Valutata l'organizzazione interna e la tecnica per ottenerla, ha susseguito una fase di produzione risultata con l'ottenimento di tre tipi di tubi compositi bio-inspirati. Campioni estratti da tali tubi sono stati testati in compressione per valutare la risposta del materiale sottoposto ad un tale tipo di carico. I materiali prodotti sono stati successivamente comparati prendendo in considerazione la percentuale della quantità volumetrica di fibre contenute, questo rappresenta un importante parametro da tenere in considerazione. In seguito alla rielaborazione dei dati, è stato possibile constatare un netto miglioramento delle proprietà del materiale, il che va a conferma dell'efficacia della struttura interna scelta rispetto al materiale comparativo. Nel procedere della stesura di questa tesi, una descrizione completa del processo di produzione, le ragioni legate alla struttura selezionata, la produzione dei tubi compositi in fibre artificiali e la caratterizzazione del materiale a loro associati saranno presentati ed analizzati.

Chapter 1.
Introduction to Biomimetics

To quote with the words of Janine Benyus , a pioneer of the field, "*Biomimetics is innovation inspired by nature".* Within this simple phrase lays the concept behind this approach to science and research, which consists in taking inspiration from a deep understanding of materials and functions found in nature with the aim of solving engineering problems and provide smart, innovative and sometimes unexpected solutions.

The word itself, Biomimetics or Biomimicry, is composed of two parts, which derive from ancient Greek, "bios", which means life and "mimesis", which means imitation.

Even though the interest that this approach has drawn from the scientific community is rather new, few decades, as it is its efforts to develop a more systematic translation of concepts from the biological to the technical world, its roots grow deep into history and several examples of mankind looking up to nature, both as a simple admiration of it, the story of Icarus and his wax wings in the Greek mythology, or as a tangible pursuit to reproduce its products, the attempt of the Chinese to make artificial silk 3000 years ago, can be found.

Coming back to more recent times, the term Biomimetics was coined in the 50s by Otto Schmitt and other denominations such as "bionics" "biophysics" "biologically inspired design" were used by many to describe essentially the same interdisciplinary path to the development of new ideas. The importance of a multidisciplinary team or the interaction between different scientific figures such as chemists, biologists, designers, physicists, material scientists and engineers is vital because it is thanks to the synergy of different skills that a complete understanding of a biological element, the extraction of its concepts and their application to a technological problem is possible.

Nature had 3.8 billions of years to develop itself so it is highly probable that the solutions it managed to find through evolution represent a good optimization to the problems it had to face in creating such complex and multifunctional structures. This way of thinking represents for us an opportunity, to learn from a more experienced teacher, and a challenge, to extract and use such relations between function and structure. The power of this approach is also its versatility, inspiration and insight might be acquired for several fields of research like architecture and design, lightweight construction and materials, surface and interfaces, fluid dynamics, robotics, communication etc. Studying a particular object from nature, also if it doesn't have a straightforward connection to the function, might shed some lights on the function that needs to be unraveled in order to provide a technical answer. In figure 1.1, it is possible to find some examples of objects from nature and their selected functions [3].

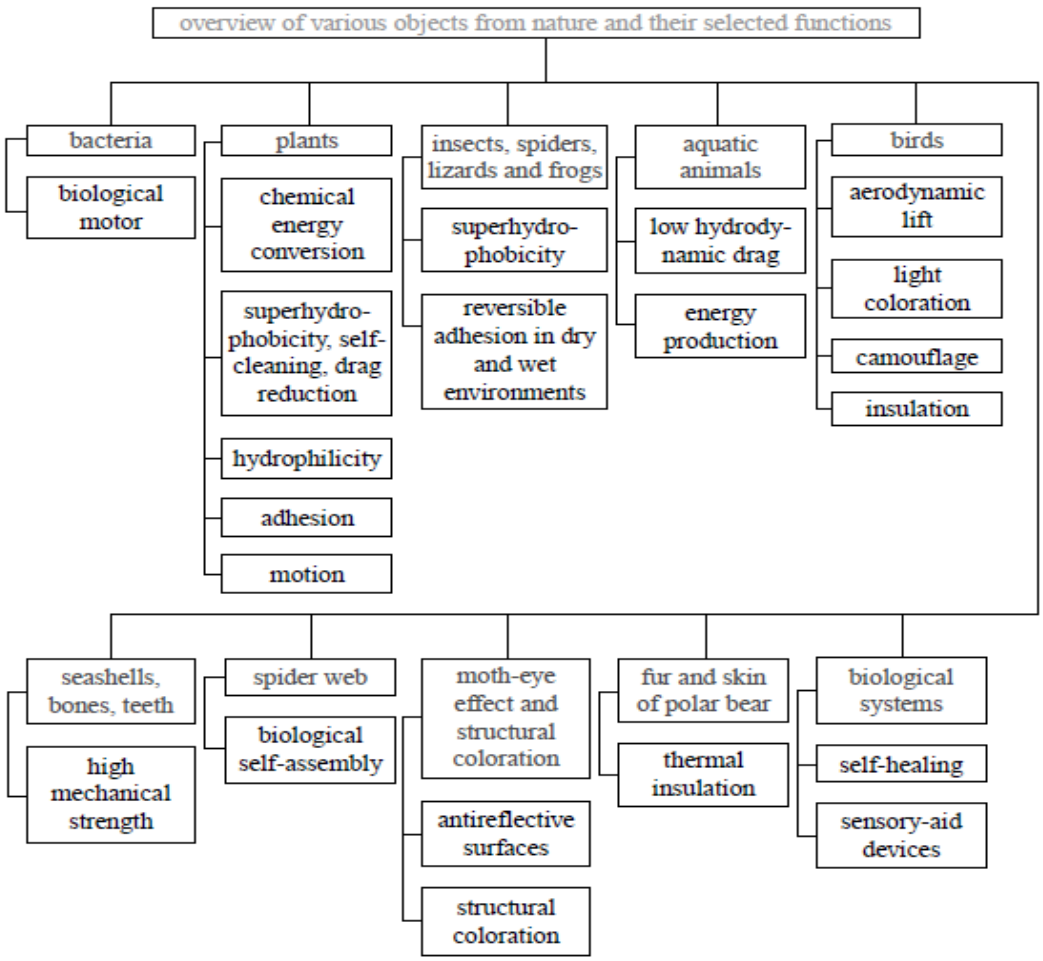


Fig. 1.1. An overview of various objects from nature and their selected functions.

1.1 Natural and man-made materials

Natural materials are composed of relatively few constituents which are used to synthesize a variety of polymers and minerals. On the other hand, man-made materials are characterized by the use of many more elements. This is underlined by the invention of materials with special properties, which are not used by nature, such as copper, iron, in their metallic forms and silicon semiconductors, distinctive of this information age. These types of materials require high temperature for fabrication and such energy is not available to biological systems, yet nature has developed a range of materials with remarkable functional properties. The choice of elements and the way materials are made are just two of many differences present between natural and artificial materials. However, the driver for change in biology and engineering may well be the same: resolution of a technical conflict [4].

1.1.1 Growth and functional adaptation

An engineer selects a material to fabricate a part according to an exact desired design, nature goes the opposite way and grows both the material and the whole organism using the principle of biologically controlled self-assembly. Growth is a process that can be influenced by external conditions including temperature, mechanical loading, supply of light, water or nutrition. A living organism must necessarily possess the ability to adapt to external needs while possible external influences on a technical system must be typically anticipated in its design phase. Therefore natural and artificial structures present different strategies in designing a material: “growth” and “fabrication”. In the case of engineering materials, the strategy is static as the choice is made at the beginning, according to the functional requirements and corrosive or deteriorating conditions, also taking into account possible changes during the service life. The fact that the natural material grows rather than being fabricated leads to the possibility of a dynamic strategy [6]. It is not the exact design of the organ that is stored in the genes but rather an algorithm to build it, the final product is therefore a result both of such recipe and the several external conditions. The advantage of this approach is that it allows flexibility at all levels:

1) It permits adaptation to the function while the body is still growing.
2) Allows the growth of hierarchical materials where the microstructure at each position of the part is adapted to the local needs.

Shape and microstructure become intimately related due to their common origin which is the growth of the organ. A simple example of this concept is the

change of shape of a tree due to a landslide or other external events, see figure 1.2.



Fig. 1.2. Adapting abilities of trees to external causes like man or nature.

1.1.2 Hierarchical structuring

Hierarchical structuring is one of the consequences of the growth process of the organs [6], it allows

1) the construction of large and complex organs based on much smaller building blocks
2) the adaptation and optimization of the material at each level of hierarchy to yield outstanding performances.

There are many biological materials that can provide such a complex structure and present high performance functions. An idea of how wide this reality can be, it is given by the following examples taken from the natural world: bone, trees, spider silk, attachment systems of geckos, optical microstructures, skeleton of glass sponges, super hydrophobic surfaces etc. New functions may be obtained just by structuring a given material instead of choosing a new one to provide

such task. A clear example of his strategy is presented in figure 1.3, where it is highlighted the fact that within tissues with different material properties, such as for example bone, cornea and tendon, the fundamental building blocks, at a certain length scale, present striking similarity [5]. Structural features commonly found in different tissues are classified as universal. Universality is found predominantly at ultra-small scales, forming basic protein constituents while a great diversity of structure is typically found at larger length-scale where particular patterns are established. Research in literature provides evidence that the occurrence of hierarchical structures is crucial to combine seemingly disparate properties, such as high strength and high robustness.

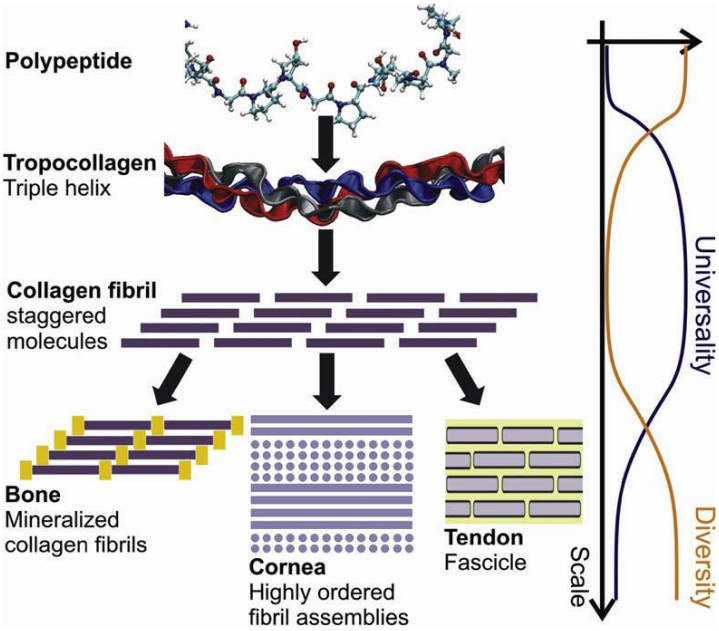


Fig. 1.3. Hierarchical structures of collagen-based biological materials [5].

Composite materials are a classical example for biomimetic translation of nature's wisdom into technology. Bone, plant stem, wood have highly optimized the use of fibers in the exact direction of the effective loads under which they are subjected. These became really interesting models for a material scientist or engineer, understanding the microscopic fiber's arrangement and the macroscopic shape as a function of the applied load and several other external output is the first step of this approach. Composites are present in both natural and artificial materials, even though the natural ones present an hierarchical

structure that is much more complex and at several scale levels, for man-made composite it is possible to obtain an hierarchical structure just on few levels.

1.1.3 Damage repair and healing

One of the most remarkable properties of biological materials is their capacity to self-repair. Even though this property has not yet been transferred to artificial materials, it represent a very interesting next step for the material engineering field that it is worth mentioning. There are different strategies associated with self-repair. At the microscale there is the concept of sacrificial bonds between molecules that break and reform dynamically, providing the possibility of plastic deformation. At higher levels many organisms have the possibility to remodel, for example in bone there are specialized cells that permanently remove material while others are depositing new tissues [6]. This cyclic replacement of bone material has a double advantage:

1) It allows a continuous structural adaptation to changing external conditions
2) It permits the replacement of damaged material wherever and whenever needed.

A schematic representation of the concept expressed above is represented in figure 1.4.

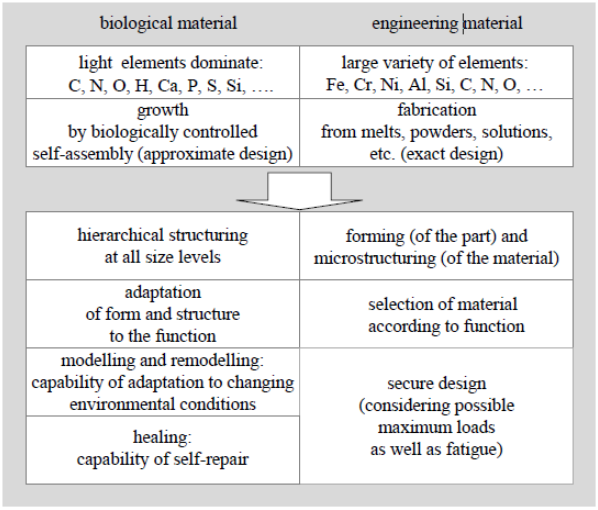


Fig. 1.4. Schematic summary of strategies pursued in the two types of materials.

1.2 Remarkable features of natural composite materials

There are endless examples in nature of composite materials starting from bone, shells, wood etc., nature tried for millions of years the vast possibilities of the structural design space and it is reasonable to believe that the structures they present now are the best answers nature found so far to fulfil some specific needs. An important aspect of such materials is the remarkable enhancement of their mechanical properties, compared to the ones of its constituents, that can be achieved through a carefully micro and nano designed structure. In figure 1.5, Ashby diagrams of the fracture toughness as a function of the elastic modulus are shown for several materials and their building blocks.

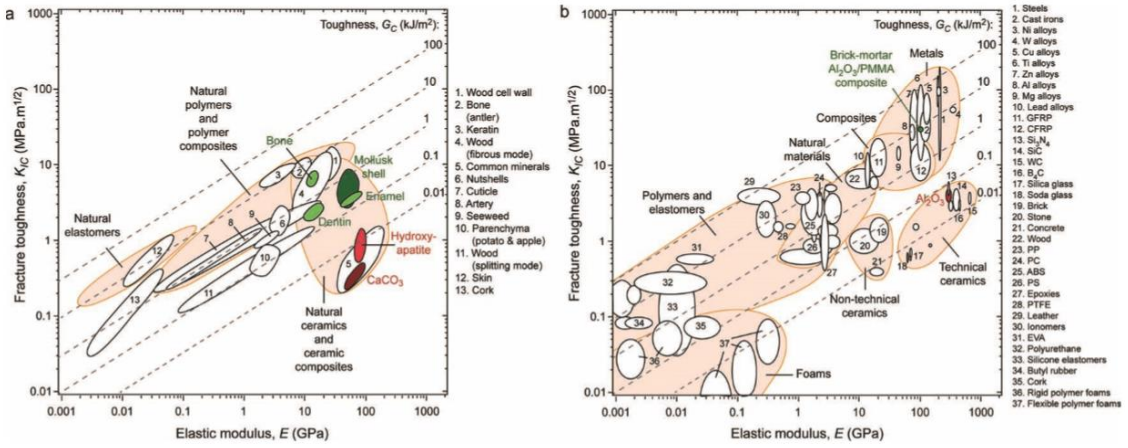


Fig. 1.5. Ashby diagrams displaying the fracture toughness K_{ic} and the toughness G_c of a wide range of a) natural and b)synthetic structural materials as a function of their elastic modulus [1].

It can be noticed that mollusk shells, for example, present values of fracture toughness at least one order of magnitude higher of calcium carbonate, $CaCO_3$, which is the main chemical element of shells, around 95%. This corresponds to a two order of magnitude increase in the energy required for fracture. Same considerations can be drawn for bone, dentin and enamel compared to their mineral phase hydroxyapatite [1]. This increase of mechanical properties is rarely obtained in artificial materials which present a much less structured internal arrangement. A crucial role for natural composite materials is played by interfaces and Nacre is a perfect example. Considering that his main constituent is a mineral, it may seem puzzling that it would be able of exhibiting a ductile-like behavior with relatively large failure strains. In order to achieve

such performance some requirements must be met [5]:

1) the interface must be weaker than the tablets, the mineral building blocks, otherwise they would fail in tension before any significant strain, showing a brittle behavior.
2) Some hardening mechanism must take place at the tablets' level in order to spread sliding through the material so the tablets have to climb obstacles to move on one another.

This clarifies that the performance of nacre is partly controlled by mechanisms at the interface between the tablets and, generally speaking, the importance of such interface mechanisms for composite materials.

Natural materials can tune their properties thanks to several structural parameters such as the geometry of the building blocks, their spatial and orientational distribution, type and distribution of chemical bonds, their 3D architecture. Furthermore they seem to be characterized by a multifunctional behavior, providing sometimes optical, magnetic, sensing and interfacial properties. The realization of outperforming advanced synthetic materials is conceivable if the elaborate structures of biological materials are combined with superior basic constituents. A deep understanding of the design principles utilized by nature to fulfill the mechanical demands and respond to the dynamic stimuli of the natural environment might help us address to current limitations of man-made composites and shed light to pathways to high performance bioinspired composites.



Fig. 1.6. Abalone shell and a spider web.

1.2.1 Examples

After presenting the main features that characterize materials grown by nature, it could be interesting to suggest a couple of examples, figure 1.6.

Seashells are composite material constituted mainly of inorganic minerals and a small fraction of biopolymers. The most studied is the Abalone shell which presents extraordinary mechanical properties, compared to the main constituent $CaCO_3$, thanks to his hierarchically organized structure.

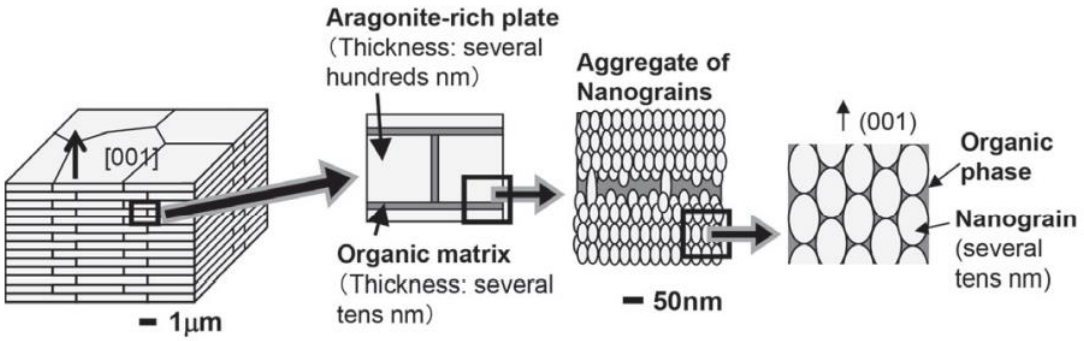


Fig. 1.7. Hierarchical structure of the Abalone shell at different length scale [7].

It is distinguished by the presence of a thick organic layer at the nanoscale, 20-30 nm, followed by 0.5-10 μm crystal bricks which constitute the microstructure, between which and a thick inner layer of 0.3 mm composed of nacre, also known as mother of pearl. Between the crystal "bricks" there is an elastic biopolymer, the combination of the two components inhibit transversal crack propagation, making the material strong and resilient [5].

Another example of how the hierarchical structure can allow the material to achieve great performances is the silk of the spider web. The web is insoluble in water, it can withstand significant weight and is resistant to atmospheric agents like wind or sunlight. Spider silk is three time stronger than steel with a tensile strength of approximately 1,2 GPa. The combination of strength and extensibility is give mainly by the domains of crystalline β-sheets and flexibles helices within the polypeptide chain, a representation of some of its hierarchical levels are represented in figure 1.8. The web has also the purpose of catching insect thanks to its stickiness.

It is also quite interesting how the spider creates its 2d web out of silk fibres. A study found in literature presents its results after analysing the spider web-

building behaviour using a computer model that constructed artificial webs with a rule-based simulation. They found that the web characteristics, such as the spiral distances, eccentricities and vertical hub location, were accurately simulated by the model This may be interesting for the modelling of biological self-assembly structures of complex materials from the local rules to the overall structure that is adaptive to the external conditions [3].

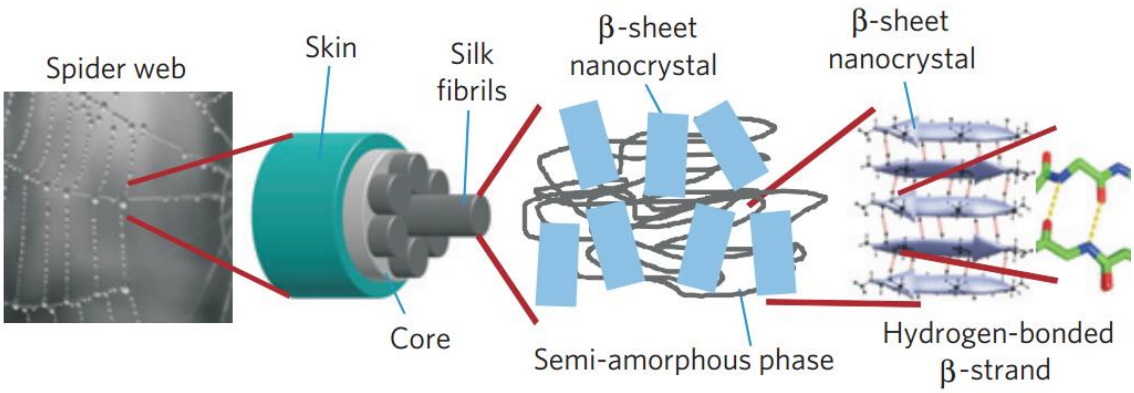


Fig. 1.8. Schematic of the hierarchical spider silk structure that ranges from nano to macro [8].

1.3 Translation of natural design principles

The transfer of a concept or mechanism from living to non-living system is not trivial. A simple and direct replica of the biological model is rarely successful, due to the different nature that characterize them, further adjustments in the optimum biological architecture might be in order because of differences in demands or in boundary conditions in engineering and natural environments. Often it's not clear which requests have been satisfied by the as-optimized structure. A phrase that summarizes well this concept was stated by Rich Huiskes who said: "If bone is the answer, what is the question?" [6] This underlines the need to understand the structure-function relationship of the biological material in his context. To conceive and develop high performance composite materials inspired by nature's design principles a systematic investigation of the following questions is in order [1]:

1) How do biological composites tune their local chemistry, structure and mechanics at multiple length scale to maximize their functional properties?
2) What are the key structure-function relationship in a given biological composite? What can we learn from such relationships, concerning the

physical and mechanical design principles underlying the optimum architecture of the natural material?

3) How can we produce artificial composite that incorporate the design principles of a given biological material?

1.3.1 Approaches

From literature [2], two main approaches of doing biomimetics emerged.

1) Top-down approach in which engineers start with a specific problem, like optimizing existing products or processes, and search for ways to solve it with the aid of biologists and their pool of biological knowledge. After identifying the most promising solutions with eventually additional structural and functional analysis, the engineer will abstract the concepts and create a modified and appropriate technical solution.
2) Bottom-up approach in which the whole process is turned on by the curiosity or interest of the researcher on a specific organisms from which new principles are discovered, analyzed and finally transferred into technical solutions.

1.3.2 Tools

Something that might help to ease the identification of certain biological species as model due to the fulfillment of particular desired functions, is the establishment of databases where engineers can find the analysis and explanations of the structure-function relations, after a deep and thorough understanding of such natural elements by biologists. The research of a function on such database should provide the engineer or scientist with potential models and allow such figure to identify the best suited archetype for the final goal of the project. Another tool that would help making the biomimetic approach less fortuitous and more systematic is the development of a logical framework that takes its origin from TRIZ [4]. It was developed in Russia, more or less 50 years ago, and it is known for its successful transfer of inventions and solutions from one field of engineering to another. This peculiarity makes it really appealing to the biomimetic approach which is basically the transfer of principles and mechanisms from biology to science and engineering. It consists in an accurate definition of a problem and its context by the association with a pair of opposing or conflicting characteristics. In order to standardize the process, many patents were analyzed and a list of 39 conflicting

characteristics was outlined. To make the process easier such conflicting couples were placed in a matrix with antithetic features along the top and the desired features arranged along the vertical axis. Once the "contradiction matrix" is available, the problem can be solved by separating it from its context so that solutions to any problems can be drawn from a very wide range of science and technology. One of the peculiarity of such method is that the more incompatible the contradiction features are, the stronger the solution will be revealed. The TRIZ method is rather complex, yet it's worth mentioning because attempts towards defining a matrix derived from biological effects have been done and some results show an analogy of only 12%, between biology and technology, in the principles which solutions to problems illustrate. While technology solves problems mainly by manipulating usage of energy, biology uses information and structure [4]. Such study shows what is the potentiality, yet unexplored, of the principles on which natural solutions are based.

1.4 Biomimetics in the industry

The biomimetic approach has proven itself of being able to help delivering smart and efficient solutions into the market. It has been estimated that the 100 largest biomimetic products had generated approximately US $1,5 billion over 2005-2008 and the annual sales are expected to continue increasing[3].

I would like to close this chapter with some of the most successful products and the corresponding biological models.



Fig. 1.9.The scaly pangolin and the Waterloo International Terminal.

The scaly pangolin inspired the design of the Waterloo International Terminal. The movement of the scales of the pangolin, allow air to circulate, regulating its temperature. As a result of the design, the layout and movement of the glass panels allow the surge in air pressure, from trains entering the station, to efficiently and safely escape, figure 1.9.

The yellow boxfish, found in tropical seas, inspired Mercedes-Benz to develop a car called the 'Bionic'. The boxfish was selected as the basis of the design, because its unusual shape dramatically reduces drag, meaning that it is extremely efficient. Mercedes Benz were also interested in its exoskeleton, which is light yet extremely rigid. They applied what they found through research and experiment, to the frame of the new car.



Fig. 1.10. The yellow boxfish and Bionic Mercedes Benz.



Fig. 1.11. Burrs and Velcro.

In 1941 Swiss engineer Georges de Mestral was walking his dog through a field, when he realised that the dog's fur and his trousers had numerous burrs attached to them. Burrs have natural hooks that attach to almost any type of fur and textiles. He took inspiration from this to invent Velcro, figure 1.11.

Engineers studied the efficiency of whale fins in water and as a consequence have developed a new type of blade for wind turbines. The new design reduces noise, which is a common problem and has increased the generation of electricity by 20%.This design of blade, is also being applied to fans, pumps and compressors, to make them run quietly and also to reduce the amount of electricity they consume.

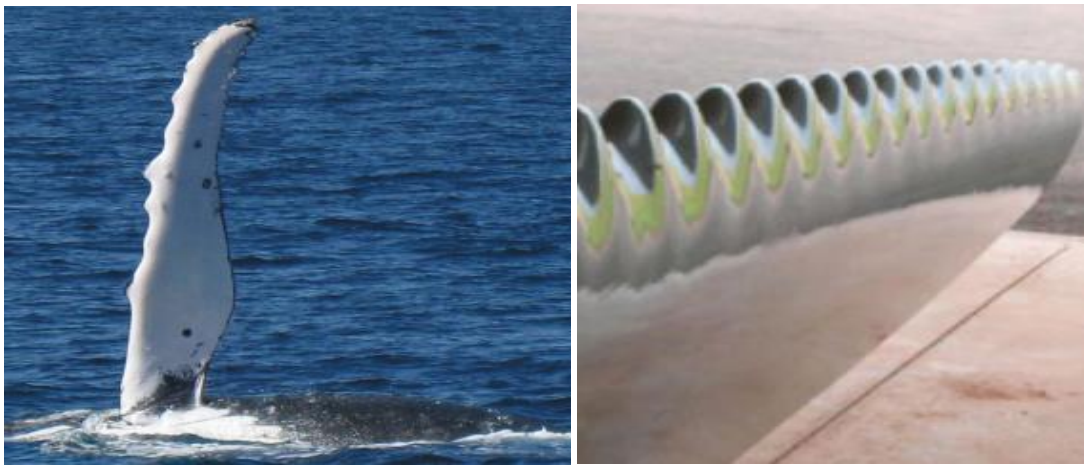


Fig. 1.12. Whale fin and the wind turbine blade inspired from it.

Chapter 2.

Introduction to Bone and its Properties

The nature-made model chosen for this project as a biomimetic source of inspiration and wisdom is bone, in particular the cortical one. The reasons for this choice will become clear through the scrolling of this chapter.

Bone represents one of the most studied biological material both due to the great efforts of medical research to cure diseases like osteoporosis or to provide better prosthesis and to the increasing engineering interest to unravel the concepts behind such a remarkable material.

Bone must satisfy several request to fulfil its tasks, first of all superior mechanical properties to sustain the whole structure and yet it has to be light to allow movements without the use of a lot of energy, strength is also important to avoid breaking after impacts and protect internal organs. Considering that it is a living material it has to be able of self-healing in case of fracture and dynamically respond to the internal and external constraints of a growing structure.

Bone is composed of two main constituents, a collagen matrix which provide elasticity and the ability of dissipating energy under mechanical deformation and hydroxyapatite mineral crystals that grant the load bearing capacity and which occupy more than 60% of the volume. It is possible to find also other types of substances including inorganic minerals components such as calcium, phosphorus, magnesium etc. , while among the organic ones it is possible to find proteoglycans, non-collagenous proteins, cytokines and growth factors. Particularly interesting are the cells involved in self-healing and repair

such osteoblasts, osteocytes and osteoclasts which continuously regenerate bone through a process of destruction and reconstruction.

In the last 50 years the scientific community has gained a much deeper understanding of this tissue and the internal structural reasons of its behavior. This insight has come mainly from the enabling of technology which has become able of reaching smaller length scales. Yet I would like to underline that from a reading of scientific literature it emerged that there is still a lot to uncover in terms of bone internal structure and its relation to the requirements of its functions.

2.1 Different types and functions of bones

An adult human skeleton consists of around 206 different bone with several shapes and dimensions through which it is possible to classify them:

1) Long bones, which are characterized by a shaft, the diaphysis, that is much longer than its width and by an epiphysis, a rounded head at each end of the shaft. They are made up mostly of compact bone and spongy, cancellous bone. Most bones of the limbs, including those of the fingers and toes, are long ones.

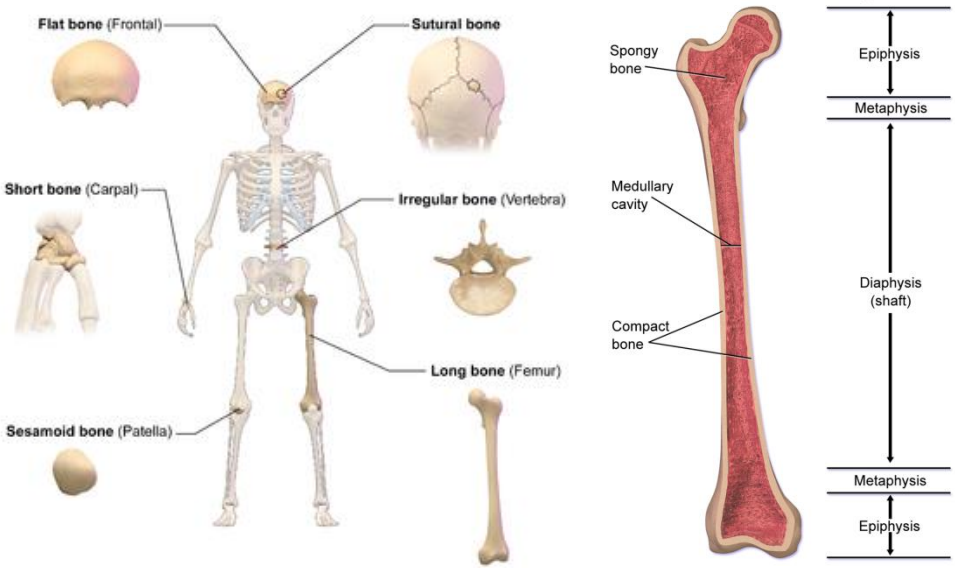


Fig. 2.1. Classification of bone by shape and structure of a long bone.

2) Short bones are roughly cube-shaped and have only a thin layer of compact bone surrounding a spongy interior. The bones of the wrist and ankle are short bones.
3) Flat bones are thin and generally curved, with two parallel layers of compact bones sandwiching a layer of spongy bone. Most of the bones of the skull are flat bones.
4) Sesamoid bones are bones embedded in tendons. Since they act to hold the tendon further away from the joint, the angle of the tendon is increased and thus the leverage of the muscle is increased.
5) Irregular bones are the ones that do not fit into the above categories. They consist of thin layers of compact bone surrounding a spongy interior. Their shapes are irregular and complicated. The bones of the spine, pelvis and some bones of the skull are examples of this category.

All these types of bones help the skeleton providing the needed functions which can be divided mainly in 3 categories:

Mechanical: 1) support of the weight of the whole structure,

2) protection for the organs,

3) facilitation of movements,

4) it facilitates hearing,

Synthetic: 5) it contains bone marrow,

Metabolic: 6) it stores calcium,

7) it helps regulate the acid-base balance.

To think that just one material is able to provide all of these functions is absolutely remarkable. The question is “How does it do it?”. The answer may take a first step from the considerations made in the previous chapter about a characteristic of biological materials, their hierarchical internal structure.

2.2 Hierarchical structure

As observed in the majority of natural tissues, bone also is characterized by a complex hierarchical structure that allows the differentiation of many bone tissues and not only, starting from common building blocks arranged in particular ways. One of the results of nature's efforts is an increase of the mechanical properties of the resulting tissues compared to the ones of the single building blocks. In the following picture it's possible to have an idea of how articulate the structure is and the main mechanisms for mechanical toughening at each dimensional level presented.

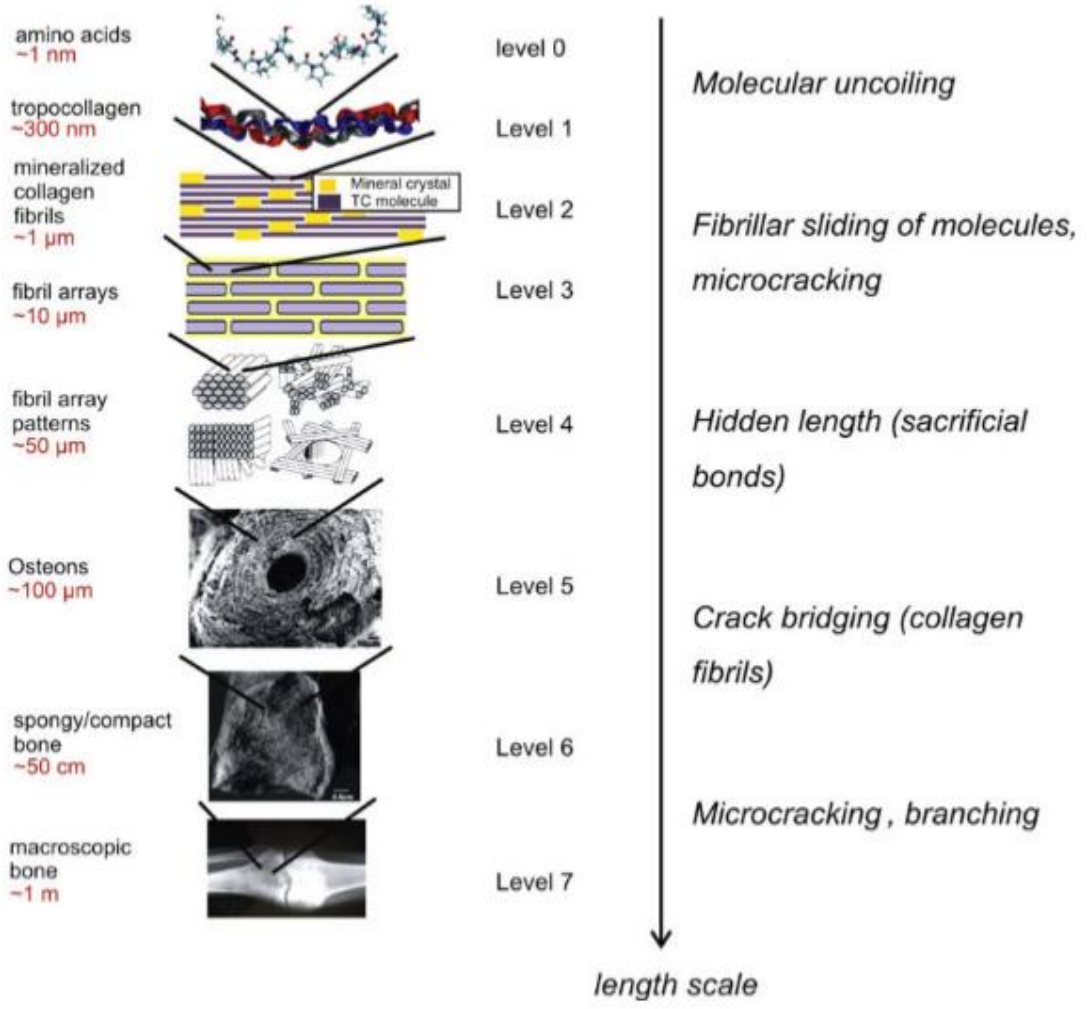


Fig. 2.2. Graphic representation of the hierarchical structure in bone [5].

A brief description of the several levels found in bone will be presented in the following paragraphs, but before , I would like recall a consideration proposed by John D. Curry according to whom the number of levels to consider is not fixed but it is dependent on the user and in particular to his specific interest and his availability of equipment [12].

2.2.1 Level 7: Macro-structure

Bone can be distinguished in cortical tissue, also known as compact bone, or in trabecular tissue, also known as spongy or cancellous. By taking into account long bones, such as the humerus, femur and tibia, it is possible to find both macrostructures. A long bone is composed of a central hollow cylindrical portion called diaphysis mainly composed of cortical tissue, responsible for the characteristics of stiffness and hardness of bone, and two rounded ends, called epiphysis, composed of trabecular tissue covered by a thin layer of cortical bone. The cortical tissue is about 80 % of the skeleton mass and ensure the stiffness and hardness of bone. The specific part of bone on which this project take is first steps for the creation of a bioinspired composite material is the cortical tissue, the following descriptions will focus mainly on this part of the bone structure.

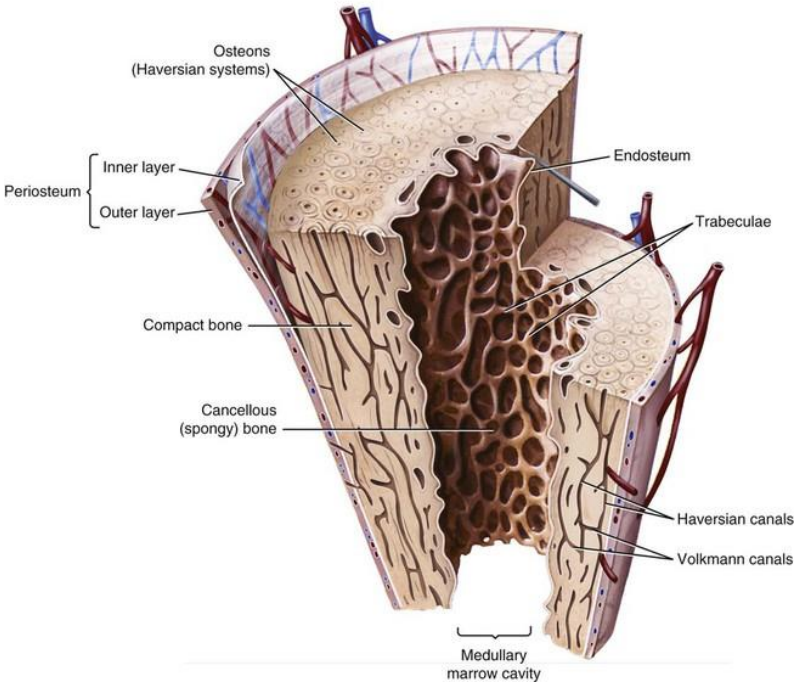


Fig. 2.3. Representation of the cortical and the trabecular bone.

2.2.2 Level 6: Cortical tissue

The cortical tissue, which is in the dimensional scale of the [mm] and more, is constituted of an intricate lamellar microstructure. Each lamella has a thickness ranging from 3 to 7 [μm] and contains fibers oriented approximately in the same direction. It is possible to distinguish two external elements, the inner and outer circumferential lamellae, in which the lamellae are extended around one or more osteons which are oriented longitudinally to the main load direction. Between the circumferential lamellae it is possible to find a dense structure constituted by hollowed cylinders named osteons in which the lamellae orientation is concentric. In the gaps between osteons there are the so called Interstitial lamellae, which are fragments of lamellae that are randomly oriented, the remains of the dissolution process of the old osteons operated by osteocytes. At this dimensional level a series of channels and cavities can be distinguished. In addition to the central hole at the center of the osteon, that allows the blood vessels to slide longitudinally, there are transverse channels called Volkmann canals that allow blood vessels to communicate with each other. Furthermore a series of smaller canals located within the osteon allow the gaps to communicate. This dense network of connections has the purpose to allow bone to be fed in all its volume. Consequently, even if it is clear that the hollows cause a stress intensification in the surrounding materials it should be considered that, on the other hand, they also play an important role in the low density of the material and, thanks to an optimized structure, to its ultimate fracture stress.

2.2.3 Level5: Osteon

Among the three structures mentioned in the previous paragraph, the main one that constitutes the cortical tissue is the osteon also called Haversian system, which occupies about two-thirds of the volume and it is a key element in the biomimetic process presented in this thesis work. An osteon is a hollow cylinder with a diameter between 100 and 250 [μm] and a length of about 1 [cm], made of concentric lamellae and it is placed in the longitudinal direction of the bone. Around the outer edge of the osteon there is the cement line, a layer with a thickness between 1 and 5 [μm] of more mineralized tissue, made of collagen and HAP crystals, in which small cracks might nucleate.

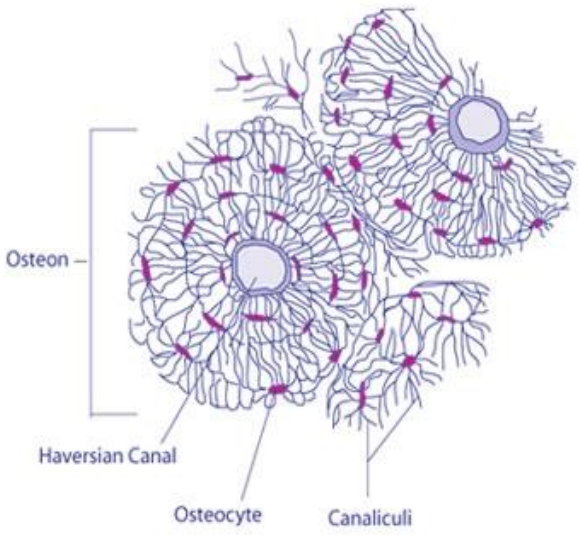


Fig. 2.4. Representation of the osteons and the lamellae that constitute it.

The lamellae are made of collagen fibers which are oriented in the same direction. The result of this micro-structure is a succession of fibrous layers with different orientation that together lead to a higher stiffness and a higher torsion resistance of the osteon.

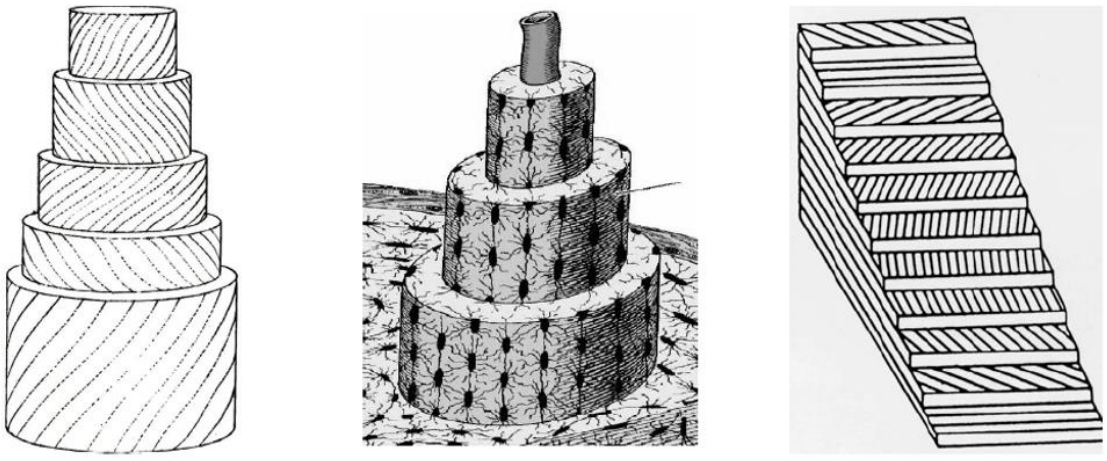


Fig. 2.5. Representation on the concentric lamellae structure of the osteon.

A study presented in literature demonstrated that a soft osteon promotes micro cracks propagation towards the osteon whereas hard osteon repel micro cracks. Considering that cement lines act as an interface for crack propagation, upon encountering a cement line, a crack would cease to travel transversely and follow the longitudinal boundary trapping the crack in the cement line, therefore increasing the fracture toughness of the bone [16].

2.2.4 Smaller Levels: collagen fibers, fibril, tropocollagen and polypeptide chain.

The next hierarchical levels are on the nano-scale. Indeed it was observed that the collagen fibers are constitute of smaller collagen fibrils all oriented on the same direction. The diameter of a collagen fiber is around 0.5÷3 [μm], while the fibrils one is between 10 ÷ 300 [nm]. A fibril could be described as a bundle of tropocollagen molecules bonded together thanks to hydroxyapatite crystals (10 [nm] thick and 225 [nm] long. The hydroxyapatite is really important for the resistance and fracture toughness of bone. Deepening further it is possible to describe a tropocollagen molecules as a triple-helix structure made up of polypeptide chains linked by hydrogen bonds. The tropocollagen molecules have a diameter of 2 [nm], while the single polypeptide chain have a diameter of 1 [nm]. By recent studies it is believed that this triple-helix structure could be related to the elastic properties of the bone tissue, in fact under the influence of a load the tropocollagen molecules tend to unrolls, leading to large strain, whereas the hydrogen bonds can be rebuilt, acting as sacrificial bonds. Indeed, the progressive formation and breakage of such bonds increases the energy to failure, allowing the preservation of the mechanical properties and the enhancement of fracture toughness.

2.3 Mechanical Properties of Bone

Experimental tests have shown that the mechanical performances of bone, greatly depend on humidity, age, type of bone, portion of the bone itself, temperature and load direction. Due to its structure, bone is highly anisotropic and it is possible to notice that it presents higher elastic properties longitudinally rather than transversally. In table 2.1 it is possible to find a comparison between the mechanical properties of bone and those of other common industrial materials.

Material	E [GPa]	ρ[g/cm3]	σmax [MPa]	σmax/ρ	E/ρ
Polyethilene	0,5	0,95	20-40	21-42	0,53
PMMA	3	1,18	70	59,3	2,5
Cortical bone	17,4	1,8	148	74	9,4
Structural steel	200	7,8	400	51,3	25,4
Alluminium (1100-H14)	70	2,7	110	40,6	25,4

Table 2.1. Comparison of mechanical properties of some industrial material and cortical bone.

Cortical bone presents lower absolute value of the elastic modulus, E [GPa], and of the fracture tensile stress, σ_{max} [MPa]. Yet its ratio between the maximum stress and the density, ρ [g/cm^3], is the highest of the other present in the table. Its lightness is related to its porous structure and its elevated maximum stress can be related to its compacted lamellar structure, a result of the evolution process.

2.4 Toughening mechanisms

The hierarchical structure highly influences the fracture toughness of bone and several toughening mechanisms can be observed at each dimensional level. In the sub-nanoscale the fracture behavior is given by the mutual sliding between the HAP crystals and the tropocollagen fibrils. At the nanoscale, the interface rupture of the fibrils interface increases the energy dissipation during the breaking of the material. These nanoscale deformation mechanisms contribute to the toughness of bone by creating plasticized area nearby the growing cracks, this phenomena are commonly called intrinsic toughening mechanisms. At the osteon level the mechanisms that mostly contribute to the fracture toughness are:

1) Constrained microcracking consists in the nucleation of microcracks in the vicinity of a main crack due to the elevated stress at its apex generally in the cement line. The cement line is the interface between the osteon and the matrix and in the cortical bone is a preferential path for the crack because it requires less energy. The formation of such microcracks allows a bigger fracture energy dissipation.

2) Crack deflection and crack twisting, the difference between the two is that in the first mechanism the deviation of the crack path occur in plane while in the second one the deviation leads the crack out of plane. In cortical bone, since the preferred direction of the crack propagation is parallel to the osteon due to the presence of the cement line, the best loading direction is the same one because in his way the load direction and the crack direction are perpendicular and it is possible to obtain a higher tenacity.

3) Ligament bridging, the presence of uncracked ligaments inside the path of a crack lead to a redistribution of the load and an increase in the fracture energy necessary to the crack to propagate.

Such mechanisms present an interesting example of how bone manages to provide toughness and resistance to fracture. By recalling the biomimetic approach to science introduced in the previous chapter, these could lead to the understanding and the use of nature design principles and it is worth pursuing if we aim at increasing the fracture toughness of a man made composite materials. The attempt to reproduce some of the fracture toughening mechanisms is part of this project and the procedure followed will be explained in the following chapters.

Chapter 3.
Composite Materials and Manufacturing Techniques

Fibres-reinforced composite materials are the result of a successful combination of two main elements, fibres and matrix, which leads to the creation of a new material with properties that are not obtainable by the single separate units. The fibres have the purpose of sustaining the loads to which the material is subjected, for this reason they present high strength and modulus, while the matrix maintains them into their specific positions, shelters the fibres from deterioration and allows the transmission of loads. The two elements do not merge together but preserve their chemical and physical identities, as a consequence, interfaces between them play an important role in the mechanical properties of the final material. This field of material engineering is at the basis of this project work and some of the key elements of such a wide technical domain will be presented throughout the chapter.

3.1 Characteristics of Composite Materials

Low density, strength-weight ratio, modulus-weight ratio, fatigue damage tolerance and fatigue strength are some of the characteristics that allowed fibre composite material to be used, or investigated for a future use, in the place of metallic materials for elements that require the lowest possible weight, for example in the field of aerospace or automotive. Fibres reinforced polymers rise then as an important class of structural materials.

As opposed to structural metals, such as steel and aluminium alloys, which display a non-dependence on the direction on which its mechanical properties are measured, fibre-reinforced composite are considered anisotropic, their

mechanical response is higher if evaluated along the fibre direction. Multidirectional reinforcement may present a more equitable set of properties but yet lower than the case of measurements along the direction of unidirectional fibres. Both cases may represent interesting edges over metallic materials on a unit weight basis. This peculiarity presents the drawback of generating more difficulty in the design phase due to the direction relation of the mechanical properties but it presents also the great possibility of reinforcing the material in the directions subjected to the highest stresses by means of a tailor-like design phase.

By observing both metal and composite materials some interesting points can be highlighted:

1. Most fibre reinforced composites present an elastic behaviour in their tensile stress-strain curve while metals are characterized by yielding and plastic deformation. However, due to its heterogeneous structure, some mechanisms of energy absorption occur at the microscale [17] and, depending on the type and severity of the external load, catastrophic failure may be avoided thanks to the occurrence of gradual deterioration.

2. In the efforts of replacing metal components with composite ones, the different mechanisms of damage development and growth must be thoroughly taken into account during the design phase.

3. Fibre reinforced polymers present a much lower coefficient of thermal expansion, if compared to the metal ones.

4. High internal damping is a peculiar characteristic of this kind of material, it leads to better vibrational absorption within the material itself and has the outcome of reducing the transmission of noise and vibration to neighbouring structures. This characteristic is really desirable, for example, for sport's gear and automotive applications where a lot of effort is put in resolving issues for the passenger comfort like noise and vibrations.

5. Fibre-reinforced polymers may absorb moisture or chemical from the environment in which they are placed and this could lead to the development of internal stresses due to dimensional changes. However, generally speaking, composite materials are known for their non-corroding behaviour compared to metallic materials.

6. The composite technology requires less energy and lower pressures for the production processes in comparison with metallic materials, in addition it presents advantages in the possibility of reducing the number of components and hence the number and costs of manufacturing and assembly operations. Furthermore it leads to the possibility of eliminating finishing operations like for example grinding.



Fig. 3.1. Bicycle frame in metal and composite material.

3.2 Fibre-based classification

Fibre-reinforced composite materials might be classified according to the dimension of the fibres of which they are composed of and their orientation. Continuous, long fibres composites can be further divided in unidirectional, bidirectional and multidirectional orientation. The choice of arrangement is owed to a proper design in accordance with the actual loading conditions of the piece in production. A high performance in the longitudinal direction is at the expenses of the transversal one, so the need to reach the better compromise is essential. Short fibre composite materials can be divided in unidirectional or randomly fibre oriented. The advantage of short fibres randomly arranged is the possibility of obtaining the same mechanical and physical properties in all the directions in the plane of the lamina. By comparing unidirectional short and long fibres, the last type is the one that presents the highest longitudinal strength and modulus.

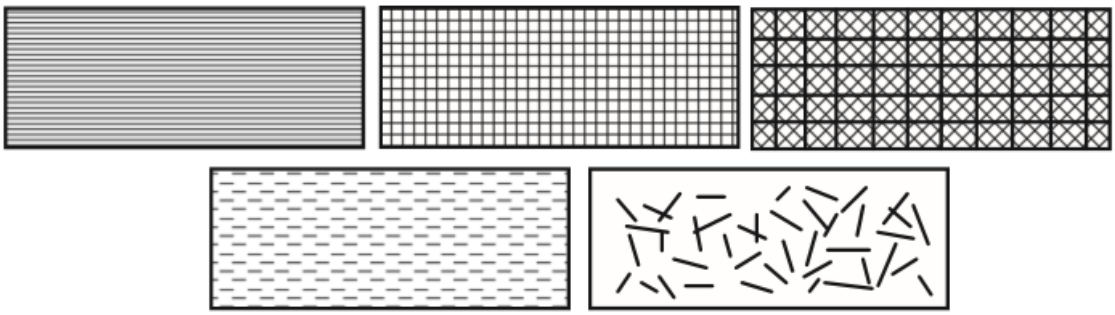


Fig. 3.2. Different types of fibre reinforcement on orientations.

3.3 Main constituent elements

The fundamental elements that are surely found in a composite material are the matrix, which operates as a binder for the fibres, and the fibres that constitute the reinforcement of the matrix. Other elements may be present in fibre–reinforced composite materials like coupling agents, coatings and fillers. The first two ingredients have the purpose of improving the wettability of the fibres in contact with the matrix and endorse the bonding between the two main elements throughout the whole material. The effect of stronger interfaces is a better load transfer between the fibres and the matrix. Fillers are engaged to reduce costs and improve the dimensional stability in some polymeric matrices. An appropriate choice of the fibre type, volume fraction, length and orientation is very important because it influences many characteristics of a composite material

1. density
2. tensile strength and modulus
3. compressive strength and modulus
4. fatigue strength and fatigue failure mechanisms
5. electrical and thermal conductivities
6. costs

Fibres present a very small diameter and to ease their handling they are commercially found in bundles produced by assembling together a large number of continuous fibres, either in twisted or untwisted form. The almost linear tensile stress-strain diagram up to the breaking point, the low strain to failure and the brittle failure mode make the fibres susceptible to damage during handling or in contact with other surfaces. The high tensile strengths presented by the reinforcing fibres is generally attributed to their filamentary form in which there are statistically fewer surface flaws than in the bulk form [17]. Generally speaking the tensile strength is higher than the compressive one.

3.3.1 Glass fibres

Glass fibres are one of the most used type of reinforcement for a polymeric material. There are different kinds which differ in the composition and the manufacturing costs, E-glass and S-glass. There is another type called C-glass used in case of the need of higher corrosion resistance to acids. Glass fibres present an amorphous and isotropic structure, a three dimensional internal architecture constituted of a long network of silicon, oxygen and other atoms randomly placed. The bundles of untwisted fibres are wound in a cylindrical package, such roving, is generally used in continuous molding operations, such as filament winding and pultrusion.



Fig. 3.3. Different forms of glass fibre available in the market.

Some of the advantages are the low costs, a high tensile strength, a high chemical resistance and excellent insulating properties. On the other hand, between the disadvantages there are a relatively low tensile modulus and high density compared to commercial fibres, susceptibility to abrasion during

handling, relatively low fatigue resistance and high hardness which causes excessive wear on molding dies and cutting tools.

3.3.2 Carbon fibres

Commercially available carbon fibres present wide range of tensile modulus values, from 207 [GPa] to 1035 [GPa]. The costs for this type of fibre are higher if compared to glass fibres, this is the reason why glass fibre is much more widespread in its use. From an internal structure point of view, carbon fibres contain a blend of amorphous and graphitic carbon and their high tensile modulus is a result of the graphitic form in which carbon atoms are arranged in crystalline structure of parallel planes [17]. The highly anisotropic behaviour of the physical and mechanical properties is due to the difference in the strength of the bonds of the atoms in the same layer, strong covalent bonds, and the inter layer weak bonds, van der Waals type forces. Some of the advantages of this type of fibre are the exceptionally high tensile strength–weight ratios as well as tensile modulus–weight ratios, a very low coefficient of linear thermal expansion, which provides dimensional stability in such applications as space antennas, high fatigue strengths and high thermal conductivity, even higher than that of copper. Drawbacks might be the low strain-to-failure, a low impact resistance, high electrical conductivity and high costs.

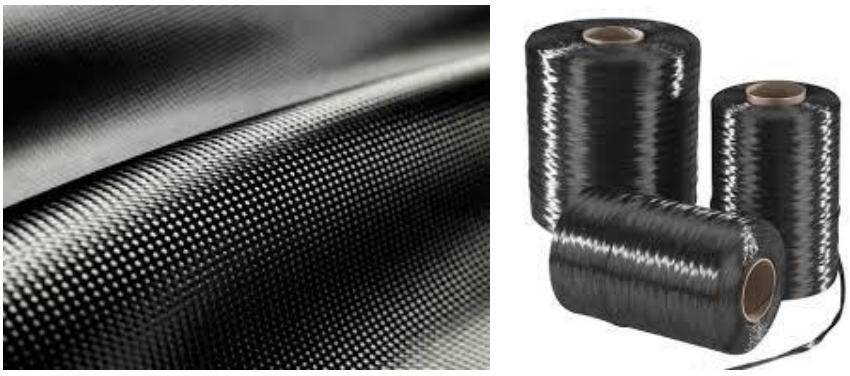


Fig 3.4. Examples of carbon fibres, fabric and rods of bundles.

3.3.3 Aramid fibres

The most known type of aramid fibres is Kevlar 49. Aramid fibres presents a highly crystalline structure with the amine group and an aromatic ring in the

repeating unit which gives a higher chain stiffness and a better mechanical and thermal stability. Aramid fibres present the highest tensile strength-to-weight ratio and the lowest density among the present reinforcing fibres, good resistance to impact damage and a negative coefficient of thermal expansion. As negative aspects it can be considered the low compressive strength and the difficulty in cutting or machining.

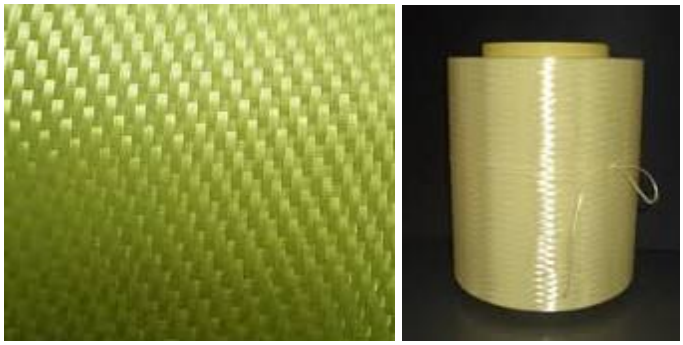


Fig.3.5. Examples of Aramid Fibres.

3.3.4 Natural Fibres

Examples of natural fibres are jute, flax, hemp, remi, sisal, coconut fibre (coir), and banana fibre (abaca), they are grown as agricultural plants in various parts of the world and are commonly used for making ropes, carpet backing, bags, and so on. They are composed of cellulose micro fibrils dispersed in an amorphous matrix of lignin and hemicellulose [17], the cellulose ranges from 60 to 80 wt% and the lignin content is in the range of 5–20 wt%, furthermore the moisture content in natural fibres can be up to 20 wt%.

Natural fibre-reinforced polymers have recently attracted increasing interest from the industry thanks to some of their peculiar characteristics

1. They are environment-friendly, which means that they are biodegradable and they require much less energy for their production if compared to synthetic fibres,

2. Natural fibres' densities range from 1.25 to 1.5 [g/cm^3] compared to 2.54 [g/cm^3] for E-glass fibres and 1.8–2.1 [g/cm^3] for carbon fibres.

3. The modulus–weight ratio of some natural fibres is greater than that of E-glass fibres, which means that they can be competitive in stiffness-critical designs.

4. Natural fibre composites provide higher acoustic damping than glass or carbon fibre ones and therefore are more suitable for noise attenuation.

5. Natural fibres are much less expensive than glass and carbon fibres.

However, there are some limitations in natural fibres. The tensile strength of natural fibres is relatively low, they present a low melting point and moisture absorption. Natural fibres start to degrade at temperature higher than 200 °C, which leads to odour, discoloration, release of volatiles and deterioration of mechanical properties.



Fig. 3.6. Examples of natural fibres.

3.3.5 Matrix

The matrix has to fulfil several tasks in the material:

1. Maintain the fibres in position

2. Allow the transfer of stresses between the fibres

3. Shelter the fibres from chemicals and moisture

4. Protect the surface of fibres from mechanical degradation.

Even though the matrix does not play a main role in the material's tensile load carrying ability, its influence in the compression properties is not to be under estimated, in fact it provides lateral support against the possibility of fibre buckling under compressive loading. It is also important for the inter lamina and in-plane shear properties of the composite material. The interaction between matrix and fibres is to be envisaged in designing damage tolerant structures. The processing and defects in the composite material depend strongly on the processing characteristic of the matrix such for example the liquid viscosity, the curing temperature and the curing time.

3.3.5.1 Different types of polymeric matrixes

Among the different types of matrix there are the thermoset polymers, mainly used for long fibre-reinforced composite material thanks to their low viscosity, thermoplastic polymers, often used with short fibres even if there is an increasing interest towards them thanks to their recycling possibility. Other types of matrixes are the metallic and ceramic ones which are mainly used in high temperature applications. The focus in this paragraph will be directed towards polymer matrixes and in particular thermoset ones because of the material chosen for this project work.

The two types of polymeric materials potentially used as matrix, thermoset and thermoplastic, present a major difference which affects their internal structure and their behaviour. The polymeric chains of a thermoplastic material are linked between themselves with weak intermolecular forces, van der Waals and hydrogen bonds. This allows the chains, once the material is heated, to flow on each other and acquire a new shape if a pressure is applied on them. Thermoset's chains, on the other hand, are chemically linked to each other by chemical cross-links, forming a rigid, three-dimensional network structure, the result of this architecture is that thermosets cannot be melt with the supply of heat.

Two characteristics of polymeric solids can be pointed out, which are not found in metals under normal conditions, their mechanical properties strongly depend on temperature and on the loading rate. As far as it concerns the temperature dependence, near to glass transition temperature the behaviour of the material changes from hard, sometimes brittle, to soft, highly viscoelastic, that leads to a reduction of some orders of magnitude of the modulus.

The main concern in the choice of a matrix are its mechanical properties, the most valued ones are:

1. The tensile modulus, which affects the compressive strength of the composite

2. The tensile strength, which governs the intraply cracking in a composite laminate

3. The fracture toughness, which influences ply delamination and crack growth.

For polymer matrixes also a good dimensional stability at elevated temperatures, which means a high glass transition temperature, and resistance to moisture and solvents, which means that the polymer should dissolve, swell or other degradation mechanisms when in hot–wet environments, are desirable qualities. Thermoset polymers, also known as resins, are usually composed of low molecular weight chains which results in a liquid with low viscosity. This characteristic is really useful because it favours the wetting of the fibres without the need of high temperature or high pressure. The wetting of the fibres' surface is important for a good interaction between fibres and matrix and hence good mechanical performances. Resins also exhibit thermal stability and chemical resistance. Some disadvantages of working with thermoset polymers are:

1. Limited storage life at room temperature

2. Long fabrication time in the mold, due to the curing process

3. Low strain-to-failure which also contributes to their low impact strengths.

The thermoset polymer chosen for the production on the composite material in this project is epoxy resin which is a low molecular weight resin containing epoxide groups, a three member ring of one atom of oxygen and two carbons. Even though it presents higher costs and longer the curing time compared to other polymers, it presents some interesting advantages

1. Absence of volatile matters during cure

2. Low shrinkage during cure

3. Excellent resistance to chemicals and solvents

4. Excellent adhesion to a wide variety of fillers, fibres and other substrates.

These peculiarities make this material appealing and allowed it to become quite widespread in the industry.

3.4 Manufacturing processes

There are many different techniques regarding the production of composite materials, the one proposed in the following paragraph are some of the most common processes for the production of composite tubes.

3.4.1 Filament winding

Filament winding is a fabrication technique mainly used for manufacturing open or closed end structures like cylinders, tanks and also plates. The process involves winding filaments under tension over a rotating mandrel. The mandrel rotates around the spindle, while a delivery eye on a carriage traverses horizontally in line with the axis of the rotating mandrel, laying down fibres in the desired pattern or angle. The filaments are impregnated in a bath with resin as they are wound onto the mandrel until it is completely covered with the desired thickness. Depending on the resin system and its cure characteristics, often the rotating mandrel is placed in an oven or under radiant heaters until the part is completely cured.

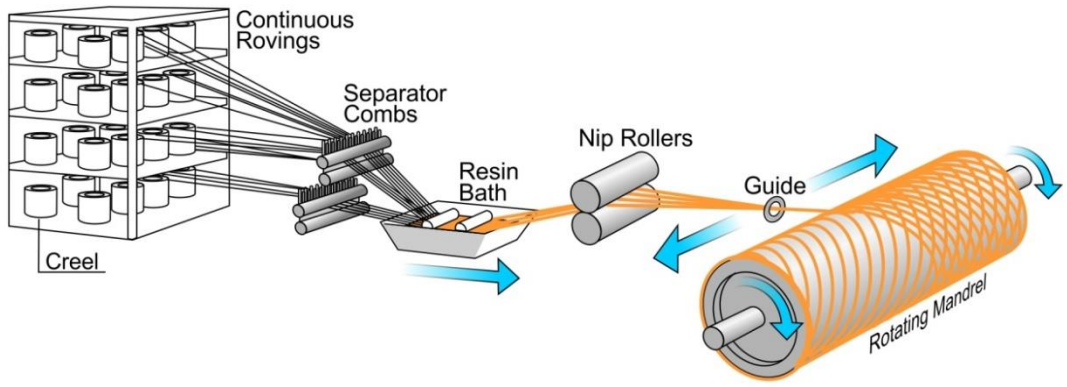


Fig. 3.7. Representation of the filament winding process [19].

Filament winding is well suited to automation, and there are many applications, such as pipe and small pressure vessel that are wound and cured without any human intervention. The controlled variables for winding are fibre type, resin

content, wind angle and thickness of the fibre bundle. The angle at which the fibre has an effect on the properties of the final product. A high angle "hoop" will provide circumferential strength, while lower angle patterns, polar or helical, will provide greater longitudinal tensile strength.

3.4.2 Pultrusion

It was developed in the 50's by the person considered by many to be "the father of composites," W. Brandt Goldsworthy. The term pultrusion combines the words, "pull" and "extrusion". Extrusion is the pushing of material through a shaped die. Pultrusion is a process in which dry, continuous fibers are pulled through a bath of resin and then through a die. The raw resin is almost always a thermosetting resin and is sometimes combined with fillers, catalysts and pigments. The fibre reinforcement becomes fully impregnated with the resin such that all the fibre filaments are thoroughly saturated with the resin mixture. The die serves two purposes, it forces the bundle of wet fibres to conform to the shape you want to create and, since the die is heated, it will cure the resin to set the bundle of fibres into its final shape. After the composite comes out of the die, it is allowed to post-cure while being pulled to the saw where it will be cut to stock lengths. Most often the reinforcement is fiberglass, but it can be carbon, aramid or a mixture.

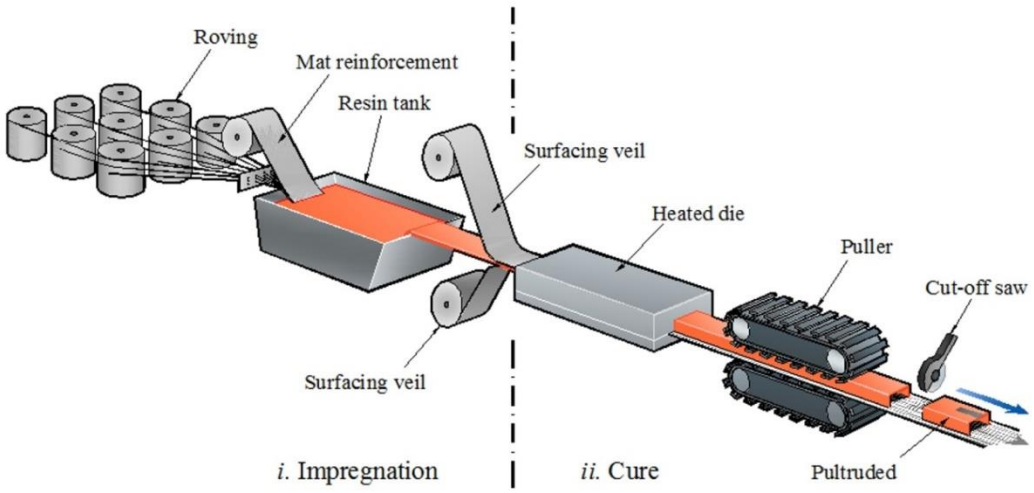


Fig. 3.8. Representation of the pultrusion process [19].

A variation is Pull-braiding where, in addition to the continuous fibres being pulled into the die, a braiding machine will apply a layer of fibres that acts to improve structural properties in other fibre orientations and adds an aesthetically pleasing look to the profile. With this technique present a high stiffness to weight ratio, also known as specific stiffness, that it allows materials of different mass to be compared quickly in rigidity-sensitive applications where weight is still a factor. Usually carbon fibres do extremely well in this area, being about 3 times stiffer than steel and aluminium for a given weight. One of the advantages of this method is the ability to easily make tubes with specific wall-thicknesses, the inside diameter of a pultruded tube is determined by a mandrel, which is easy to exchange for a different sized one, making it easy to produce tubes with varying wall thickness. Furthermore it's easy to machine due to the fact that the products are light structures.

3.4.3 Roll Wrapping

Roll-wrapping involves the applying of resin pre-impregnated composite fibre cloth around a mandrel. The outer diameter of the mandrel thus determines the inner diameter of the final tube. The mandrel and cloth are then spiral wrapped with a consolidation tape under tension to hold the laminate in place during the curing phase. After curing, the mandrel is extracted to leave the tube ready for machining or finishing as necessary. Since the matrix and resin are premixed in a temperature and particle controlled environment, the fibre volume fraction can be tightly controlled, an important parameter that affects the mechanical properties of the final product. The ease of adding or subtracting reinforcement make it possible to create custom end products to fit nearly any composite tube need.

3.5 Applications

The major structural application areas for fibre-reinforced composite materials include aircraft and military applications , automotive, sporting goods, marine and infrastructure. They are also used in electronics, for example for printed circuit boards, building construction, for example or floor beams, furniture, like chair springs, power industry, as for Eolic turbines, oil industry, for example in offshore oil platforms, medical industry, for example for bone plates for fracture fixation, implants, and prosthetics, and in many other industrial products.

I would like to focus the attention on some of the latest products in the automotive industry as an examples of the possible achievements.

In the picture below, figure 3.9, it is possible to see some of the elements present in the car which can be produced in composite material with natural fibres as reinforcement.



Fig. 3.9. Elements in a car proposed constituted of natural fibre reinforced composite materials.

Lotus “Eco Elise”, realized few years back, is an example of car produced and assembled with natural components, recyclable or biodegradable and where each manufacturing and assembly process is optimized to reduce the energy consumption. The materials incorporated include hemp, used on the car composite body panels, spoilers and seat, eco wool for the upholstery and sisal

for the carpet. Furthermore the Eco Elise's hemp hard top incorporate a set of solar panels to provide power for the car electrical system.



Fig. 3.10 Picture of the Lotus "Eco Elise".

The Lamborghini "Sesto Senso" has 80 % of the car in carbon fibre reinforced polymers. Featuring a skin one-third the thickness of the previous CFPR sports car body panels, the car's monocoque achieves the required rigidity via integrates stiffeners [18]. The achieved design objectives are the reduction of the weight by 40%, a cut of the acceleration time from 3.4 seconds to 2,5 seconds to reach 100Km/h and finally increase the power to weight ratio. Here below presented a picture of this automotive jewel.



Fig. 3.11. Lamborghini "Sesto Senso".

Chapter 4.

Development of the Manufacturing Process

The need to place long fibers parallel to the central axis of a tube and the goal to achieve a fibre volume content of around 60%, similar to the cortical bone, led us to the development of an idea that turned into a new manufacturing process that we called “squeeze-winding”. This technique has been defined through a series of attempts where each trial was critically analyzed, always keeping in mind the desired response, considering positive and most importantly negative aspects which were for us the incentive to modify the design in order to obtain a result that matched the expected effect. The idea behind it is that it would be possible to increase the percentage quantity of fibers in the whole material by reducing the quantity of the other main element, the matrix. The method devised mainly consists in a preplacement of fibres, first along the axis and then with a certain angle, with the purpose of compressing the inner ones after the appliance of the resin and the squeezing of the extra resin out. This is the general concept we based our work on and it will evolve through this chapter.

4.1 Set up’s arrangement

The first step of the project was to design the components for the set up that would allow us to put in practice the production of the composite tube. This choice was based on the requirements for the final piece and hence on the way thought to achieve them. In the following picture, figure 4.1, the elements that will be used in all of the attempts are presented.

Starting for the top left of the picture there is a) one of the disks for the placement of the fibres, b) the internal part of the plug, c) the external part of the plug, d) plastic tube used as mold and e) the ring attached to the pierced disk, used to fasten it to the plug. All these components were kept together with a series of screws.

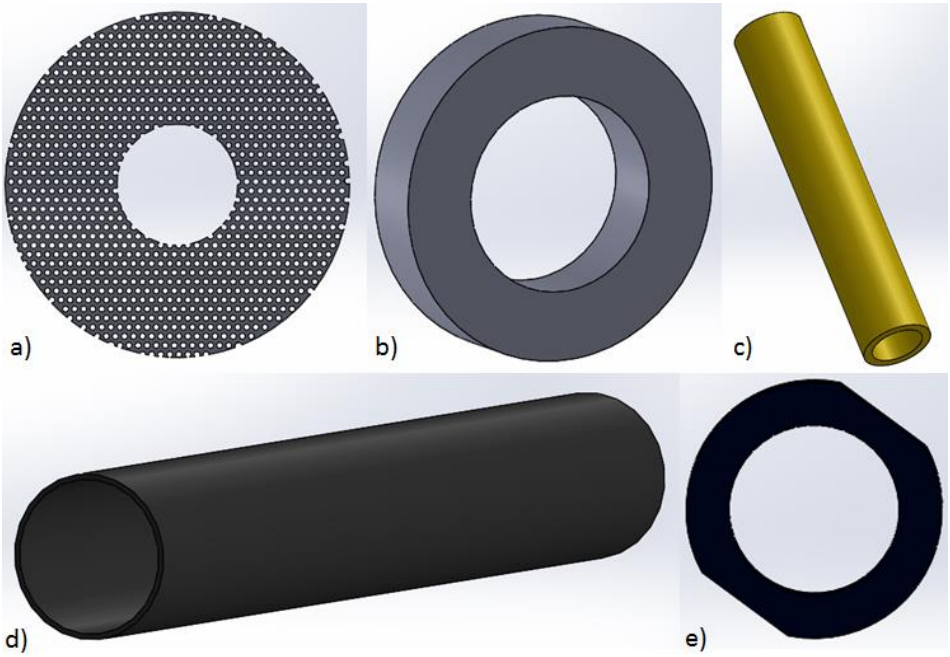


Fig. 4.1. Solid work representation of the set up's components.

4.2 First attempt of production

4.2.1 Description of the design

During the design phase it was decided to hold three layers of zero degree fibers in place by using a disk with several holes through which they could easily pass. Two additional layers of fibers, resulting at an angle of around +45° one and -45° the other, with respect to the axis of the tube, were taken into consideration to fulfill the task of compacting together the 0° fibers and to squeeze part of the resin out by been wrapped around them. It is reasonable that it would be possible to obtain a higher fiber volume content thanks to the "squeeze" aspect of the process and hence to obtain better mechanical performance. These two extra layers required two more disks at each side of the tube as a support and as

a mean by which applying the torque. In the following picture, figure 4.2, there is a drawing of the whole set up assembled for the first production experience.

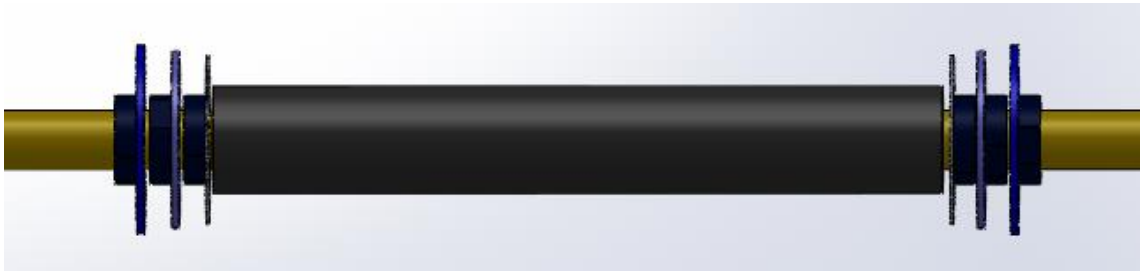


Fig. 4.2. Solid work representation of the set up for the first attempt in the production. The three disks at each side of the tube will be called throughout the chapter, starting from the left, outer, middle and inner disk.

The development of the manufacturing process was carried out with natural fibers, Jute, impregnated with Epoxy Resin. The choice of using this kind of fiber was led by the need to test the designed process with component as cheap as possible.

The tubular "mold" with three disks at each side, fixed to its ends with the help of plugs, was attached to the winding machine as stable support.

The "squeeze-winding" process consists mainly of four phases:

1. The placing of the fibers around the tubular mold,

2. The impregnation of the fibers by spreading the resin and applying the torque in the two different directions, clockwise and anti-clockwise.

3. The curing of the resin while keeping the tube spinning thanks to the winding machine,

4. The removal of the tubular mold, the cutting of the edges and the recovery of all the used components.

After placing the mold on the support, a layer of transparent film was applied on the tubular shape to ease the removal of the produced tube and prevent the resin from settling on the top of screws that could have been removed and reused afterwards. The placing of the fibers has been done manually with the use of a thread and a needle and it happened to be the most time-consuming

step of the whole process. The thread was necessary to make a handle able to pull the fiber on the other side of the disk since the eye of the needle was too big to fit in the holes of the disk. Three layers of 0° fibers were proposed in this first trial as a good compromise between a non-too thin thickness of the final tube and the need to well impregnate all the underlying layers. Other two layers were placed, fixed at the extremities of the correspondent supporting disks, the ones that were supposed to be turned after the impregnation . The middle and external disks were placed directly next to the inner disk, so the extra length of the fibers, necessary to achieve an angle of 45° starting from 0° during the appliance of the torque, was let loose on one side, as it is possible to see in figure 4.3.



Fig. 4.3. Photo of the production in progress of the tube before applying the torque at the two most external disks.

This choice was done in order to prevent the fibers from changing position and getting entangled with the others. The time necessary for each layer of fibers was around 5 hours, so a total of about 25 hours was needed for the placing of the fibers for one tube. Before beginning with the second step, a cover on the floor and on some components of the winding machine was in order because we expected some dripping of the resin. The epoxy resin, RIMR135, was mixed with a slow hardener, RIMH137, that would take eight hours pot life, time enough to complete the second step. The mass proportion used in the mixture was 100:32

between the resin and the hardener and the total volume of the mixture prepared was 2 liters. The spreading of the resin was carried out with the use of a paint-brush. The resin presented a low viscosity and it was observed a non-negligible dripping, yet this characteristic favored the impregnation of the natural fibers, which quickly turned into a darker color. The torque was applied with the use of clamps, first on one side of the tube and then on the other, for both the layers with tilted fibers. Due to the manual application of the torque and the tendency of natural fibers to relax it was not possible to measure the applied torque intensity but its appliance was carried out, for all the tubes, by the same person trained in handling manual tools so that a good degree of repeatability could be expected. Another potential advantage of the appliance of the torque and the resulting flow of resin towards the outside is that this flow could carry out bubbles of entrapped air. To better compact all the layers two metal wires were wrapped around the edges of the tube, that were successively removed by cutting the edges of the produced tube. Once the resin had been completely spread on the fibers, peel ply was used to cover them, a thin film that had the purpose of providing a smooth final surface of the tube and at the same time allowed the flow of the extra resin through it, see picture 4.4 for a visual clarification.

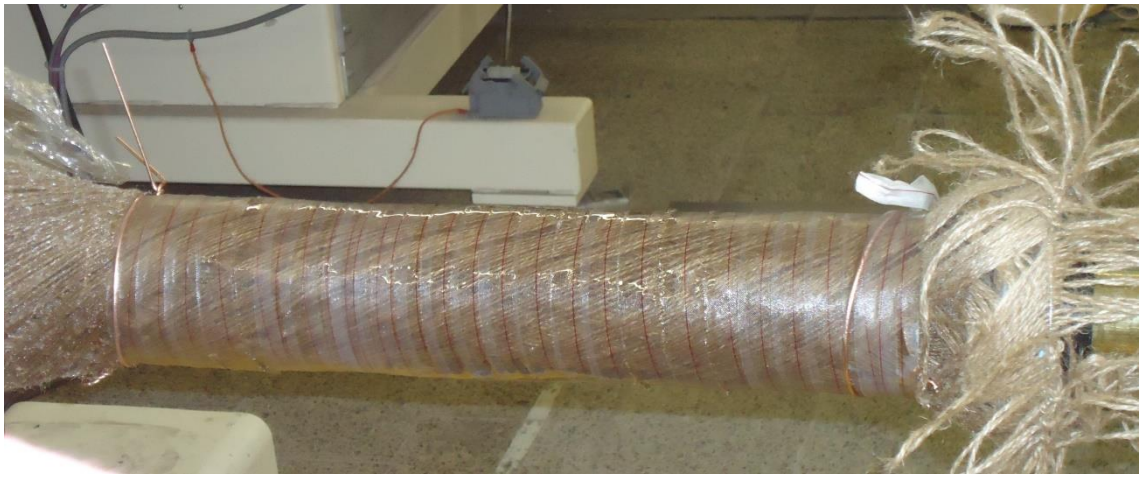


Fig. 4.4. Appearance of the semi-result of production after the second step.

The structure had been kept spinning at low velocity for the whole curing process, thanks to the winding machine on which it was mounted. Once the resin was completely cured, after 24 hours, we proceeded in the last step, removing the produced tube from the mold which we performed thanks to the use of a press. Once freed, we cut the edges of the composite tube to have a section perpendicular to the axis.



Fig. 4.5. First tube produced with the ancestor of the ”squeeze winding” process.

4.2.2 Evaluation of aspects to improve

This first attempt presented several issues. The main problem was the fact that the whole process was conducted manually and the fibers were placed one by one through the holes of the disks, pulled with the help of a needle and a thread. This influenced the amount of time necessary to perform the first step, the precision with which the zero degree angle was obtain and the presence in the tube of regions more dense than others, this step required an improvement to reduce time and efforts. During this project we did not switch to an automatized system, even if from a conceptual point of view wouldn’t be hard to design. The concern was on proving the effectiveness of the process itself, choosing a compromise between resources used and time of production.

The appliance of the torque presented also some difficulties, the ring attached to the disk to fix it on the plug resulted to be slippery and it was also problematic to reach the ring itself due to the presence of fibers. Furthermore the fibers placed on the middle and external disk, presented a high friction both with the fibers beneath, the ones at 0° already impregnated, and with the metal of the holes within which they were fastened. This lead to a displacement of the fibers on the tube surface, the need to straighten them up and the result was an irregular surface, as can be notices in figure 4.5. All of these elements lead to a

compactness of the fibers not satisfactory, that is why we had to use 2 metal wires at the edges, even though they very not easy to fasten, and furthermore it proved that the squeezing part of the process needed to be reviewed.

Another effect of the high friction of the fibers with the holes of the disk was the non-achievement of a constant angle of +/-45° as expected but, a +/-30° and, as can be noticed in picture 4.5, on one of the side of the tube there are the remains of the fibres that were meant to slide and cover the tube, but didn't because of the high friction.

By working with natural fibers, additional problems arouse due to their nature, like the relaxation of the fiber yarns after applying a small tension to keep them straight throughout the whole length of the tube and the variability of the diameter of the fiber yarn which made it difficult to make them slide inside the holes, leading, from time to time, to the breaking of the fiber and the need to replace it.

4.3 Second experience

4.3.1 Description of the improvement proposed in this attempt

Taking into account all the observations made after the first trial, some modifications were proposed starting from the way to apply the external layer of fibers.

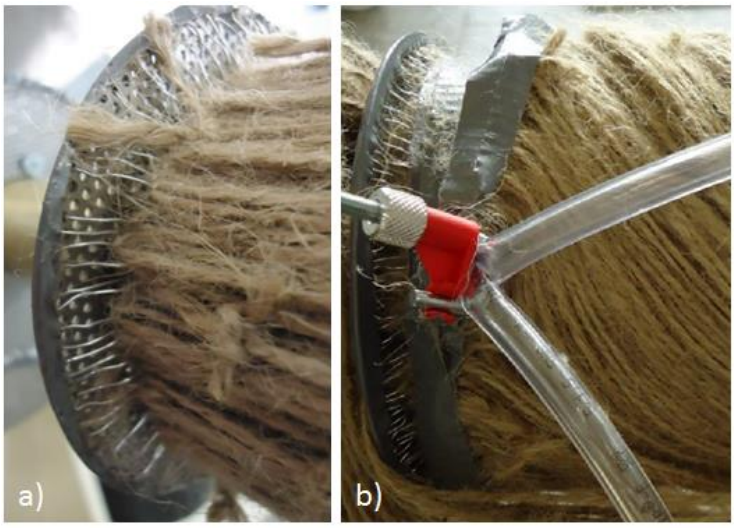


Fig. 4.6. Some details of the adjustments made for the second attempt.

Instead of placing them on the external disk, we acquired a small diameter plastic tube through which we arranged the winding of a metal wire, the aim was to obtain a flexible support that could be placed directly on the underlying layers at the internal extremities of the tube. The loose ends of the plastic tube would be used as a handle to hold on to in order to apply the torque, for a clearer understanding it possible to see picture 4.6 b).

The second important improvement proposed was the replacement of the perforated disk with a grid self-produced, using the same disk as before with metal wire going in a radial direction, see figure 4.6 a). This expedient, that allowed to use directly a needle, made it possible to reduce the time for the placing of the fibers from 5 hours per layer to 2 hours per layer. The realization of such device took 1 hour per disk but, once realized, it could be reused for the production of other tubes. Another effect of this change was a higher number of fibers placed for each layer.

For the production of the second tube, four 0° layers of fibers were applied to test if we could still obtain a good impregnation even with a higher thickness of the tube. A brush was acquired to help obtain a more homogeneous distribution on the surface and it was decided to proceed with the impregnation after having placed the fibers sufficiently close to 45°, to reach it with the appliance of the torque. To solve the problem of the slipping of the fastening rings, two planes on them were cut to facilitate the grip of the clamping tool. To collect the dripping resin, a plastic sheet, hold by a metal frame, with a hole in the center and a container beneath it, was placed under the structure.



Fig. 4.7. Set-up of the second attempt for the production of the tube, everything ready for the impregnation of the fibres.

In this second experience, pigments for the epoxy resin were used in order to obtain a red colour that could potentially be useful in the image processing phase. Taking into account all these modifications, the second tube was produced following the same main steps described in the previous paragraph.

4.3.2 Evaluation of aspects that need improvement

The main problem from this second experience was the way the torque on the final layer was applied. The set-up with the ringed tube was proven to be not a good choice for the following reasons:

1. The tubes were not fixed to the extremities so, by turning them, they tended to move toward the center of the structure and they resulted not able to keep the fibers in tension, a necessary condition to provide the desired compaction.

2. A round of tape was applied between the +45 degree fibers and the -45 one in order to avoid a displacement of the underneath fibers but the movement of the ringed tubes resulted characterized by a high friction.

3. The tubes were too flexible and the attempt to turn them resulted more in a pulling that made the turning even harder.

4. The handles obtained with the edges of the ringed tubes resulted very slippery, especially with the gloves wet by the resin.

5. The fibers presented an uneven angle throughout the whole length of the tube, with a higher angle close to its edges to underline the fact that the torque's effect was just at the very edges of the tube instead of being propagated throughout the whole length.

6. The placing of the fibres on the ringed tubes resulted to be more difficult than on the disks due to is not fixed nature and also the preparation of the ringed tubes required a longer time, double than the wrapping of metal wire around the disks.

7. Instead of the metal wire, in this second experience, zip ties were used to better hold in position the fibres from the final layer and help to compact them.

The countercheck of the unsuitableness of this set-up was given by evidences observed during the final part of the impregnation process, the applying of the peel ply :

- The fibers showed a wave tendency at the advancing of the applied peel ply, this was a clear sign that the fibers were not either squeezed nor in tension.
- Another evidence was the dripping of the resin only after the applying of the peel ply.

These reasons lead to a clear understanding than an improvement of the torque application was in order again.

This wrapping of metal wire around the disks allow an higher density of fibers, compared to the one obtained in the first experience, which for the ones placed at 0° is a good thing and will be proposed again, also for time saving reasons. As far as it concerns the fibers that have to be rotated, underlining an observation is in order, a lower linear density of the fibers, but enough to cover the whole surface, could result in a:

- Less attachment of the fibers, resulting in less friction between them and as a consequence a better positioning during torsion.
- Less fibers but better position might reasonably give a better “squeezing” with the same force.

The structure created under the tube was proven to be useful to avoid an excessive spreading of the resin and it will be proposed also in the next experience with just small modifications.



Fig. 4.8. Result of the second attempt.

As can be noticed in figure 4.8, the surface of the tube looks much more homogeneous and with less superficial defects compared to the first tube. The scratches that can be observed on the inner surface are due to the releasing part of the process because the edges of the tool used to push the mold out touched the surface, hence a more accurate care is in order. A characteristic that can be underlined is that the impregnation looks good also with a higher thickness of the tube.

4.4 Third Experience

4.4.1 New changes introduced

The changes introduced in the third design of the process consist in the following points:

1. The dimensions of the inner and the middle disks were reduced to a value smaller than the outer diameter of the plastic tube used as mold for the composite tube, as it is possible to see in figure 4.9. This way it is easier to keep the fibers closer to the surface of the mold and in position. There was no more the need for zip ties as before.



Fig. 4.9. Picture of the modified inner and middle disks.

2. The outer disk was changed with a wooden ring with nails around it. This choice was made for two reasons. The first one was to speed up the process of placing the fibers, which took only thirty minutes, and the second one was to have access to the fastening ring of the middle layer.

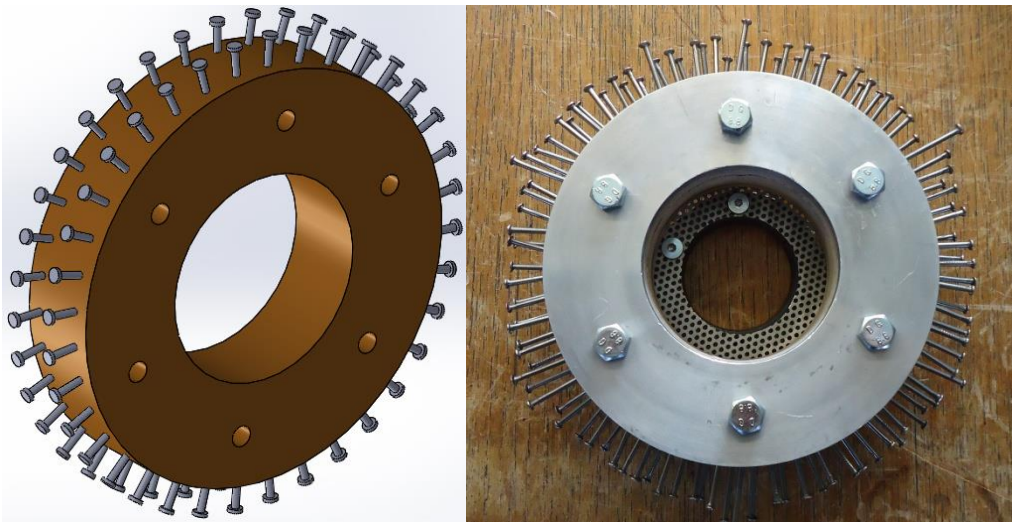


Fig. 4.10. External wooden ring.

3. Another change was made in the process, the final layer was placed after the impregnation of the rest of the fibers. The idea of placing the fibers on the wooden ring before, which could be separated in 2 parts, and then just place the whole structure came up, but the problem with this sequence was that the fibers tend to intertwine and it was becoming really hard and time taking to straighten them into parallel lines. This improvement was possible thanks to the introduction of the wooden ring.

4. Impregnating only the middle part of the structure would keep the edges free from the resin, this would allow to save time in the moment of releasing the tube from the mold. Before it was necessary to cut with a saw the edges to free the screws that connect the mold to the plugs. It was a difficult part because a lot of care was put into not cutting the underlying mold for the simple reason of reusing it. This way an easy access to the screws was possible. Of course the resulting tube was a bit shorter but it was not a problem because there were no constraints on the tube's length.

5. Less mix resin + hardener was necessary, due to the less impregnated fibers and to the structure created under the tube that was able to collect the dripping resin and allow its reuse for the fibers of the final layer.

6. Once elaborated a final design, at least for this project, the choice of not applying the peel ply on the final surface of the composite tube was made because in the previous trials it was clear that it was introducing a further compression of the fibers and it would have been better to evaluate just the effect of the applied torque.

The production was carried out taking into consideration both the previous steps and the modifications proposed above.



Fig. 4.11. Pictures representing the middle layer (above) and the external layer (under) according to the modifications of the third attempt.

This third attempt in the making of the tube was the one that satisfied the expectations the most from the point of view of the appliance of the torque. It was possible to clearly see the resin coming out of the fibers in the moment the torque was applied. This last design was therefore the one chosen to make three tubes with the same characteristics for the mechanical tests.

4.4.2 Final considerations of the third experience

Even though the design was quite satisfying, there are still some problems that need to be solved or at least are worth taking into consideration:

1. Due to their nature, natural fibers tend to stick together, this peculiarity makes it hard and time taking to place them correctly especially for the ones in the 45 degree layers. Some time was spent brushing them and there was still a portion of them, even if small, that was not perfectly in line with the others.

2. Due to the friction between the layer on the move and the one underlying, it was possible to observe that the angle at which the fibers were placed was not constant throughout the longitudinal axis of the tube.

3. The fibers were not completely fixed at the edges but they are held in position by making them pass through a metal ring, this means that they can actually slide a bit and this happens specially while brushing them. The result was that, since some fibers were longer than others, the "squeezing" was given only by the shorter ones. It is reasonable to suppose that if able to fix them at a certain length, in a way that would not take too much time, the effect of compacting of the fibers would be greater.

4. Another problem that is constant in working with natural fibers is the fact that they tend to relax, with carbon or glass fibers we would be able to apply a greater value of force and possibly we could maximize the squeezing effect. These two reasons lead to the consideration that natural fibers may not be the most appropriate for this technique, at least in the way that has been designed. Glass and carbon fibers may be better suited for this technique.

5. The choice of not using the peel ply affects the final surface and it's possible to observe a wavy final surface with fibers that are impregnated but not completely surrounded by the resin. With this surface it's more difficult to evaluate an outer diameter of the composite tube, useful for further calculation, in particular the one about the fiber volume content.

The choice of keeping the impregnated fibers spinning for the whole curing time was proposed again both for the second and the third experience.

Three tubes with this setting and procedure were produced, see picture 4.12, with the aim of evaluating the repeatability of the process along with the characteristics from a mechanical and fibre volume content point of view.

The presence in the laboratory of the winding machine allowed the possibility to compare the results of the three tubes, obtained with the squeeze-winding technique, with the ones obtained from tubes manufactured with the filament winding technique, one of the most used in the industry and described in the previous chapter. In the following chapter, the results of the mechanical test from both types of tubes will be presented.



Fig. 4.12 Picture representing the three tubes made with the final manufacturing process developed called “squeeze-winding”.

Chapter 5.
Characterization of the Squeeze-Winding Process

The production technique developed and described in the previous chapter, called Squeeze-winding, is in need of a characterization in terms of the reproducibility and in terms of the actual performances that the products display. For this purpose three tubes were produced trying to maintain the same settings and characteristics in order to evaluate the above-mentioned aspects of the technique. It is important to underline that the whole process was carried out manually, both in terms of the placing of the fibres and the appliance of the resin and the final torque. This aspect of the production inevitably introduces a higher degree of variability compared to an automatized process. In order to evaluate the effectiveness of the process developed from a performance point of view, samples of the as-produced tubes will be compared in compression with the ones obtained from a tube produced with the well-developed filament winding technique. The production of the filament wound tube was possible thanks to the presence and availability of the winding machine in the laboratory where this experimental activity was carried out, the Institute of Polymer Material and Plastics Engineering of Clausthal Technical University, sited in the Harz region in Lower-Saxony, Germany. Another aspect that has been analysed and described in this chapter is the fibre volume content obtainable with the new process and it will be compared to the one obtained from the filament winding technique. Such value is an important parameter that needs to be taken into consideration to partially account for the mechanical performances of the produced structure.

5.1 Natural Fibre Tubes

5.1.1 Components

The type of fibre chosen for the evaluation of the technique is Jute. Natural fibres present the advantage of being much cheaper than artificial ones and this was the main driver for this choice. The matrix used is a mixture of Epoxy Resin and a slow hardener, in a relative mass ratio of 100:32 respectively of resin and hardener. The properties of such constituents are presented below in table 5.1 .

Element	Material	
Matrix	Epoxy Resin	RIMR 135
Matrix	Slow Hardener	RIMH 137
Reinforcement	Fibre	Jute

Table 5.1. List of components and their properties.

In the picture below, fig. 5.1, it is possible to see a representation of both a thread of natural fibre and epoxy resin.

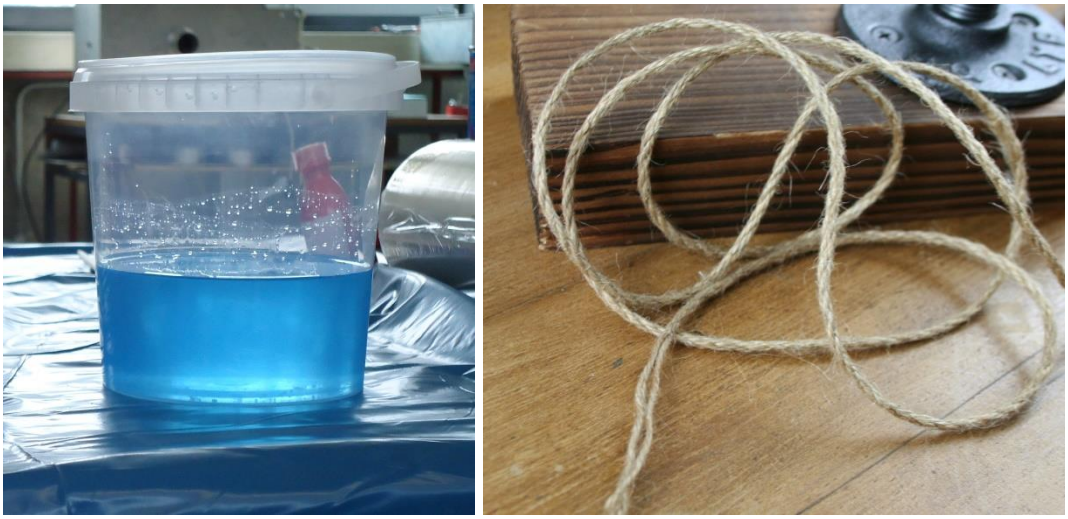


Fig. 5.1.Mixture of epoxy resin and hardener and a jute thread.

5.1.2 Production times

The time necessary for the production of each tube was calculated considering all the steps that characterize the process, which briefly are the placing of the fibre, the appliance of the uncured matrix and the successive torque, the curing of the mixture and the demoulding of the produced tube. In table 5.2, it is possible to see the time associated to each step that characterizes the squeeze winding technique. It is meaningful to underline that the times presented are the final ones, longer times were necessary before the optimization of the process, occurred through the attempts described in the previous chapter. It is worth mentioning that the winding machine allows the user to choose and set the desired speed with which it performs the programmed steps, yet some restrictions presented during its use. Such constrains were due to the need of keeping the fibres in tension, especially for ones in the two external layers placed at +/- 45°, they had the important task of compacting the underlying fibres. The non-negligible tension of the fibres and a lively speed of the guide of the machine led, for time to time, to the breaking of the fibre itself, the need to stop the process and replace the thread in order to continue with the production. However the time required for the production of the desired tube with the filament winding technique resulted around 3 hours, less compared to the other process, plus of course the curing and the demoulding time.

Process & Time required to perform it		
Phase 1	**Placing of the fibres**	**6 [h]**
Phase 2	**Impregnation and appliance of the torque**	**2 [h]**
Phase 3	**Curing process**	**24 [h]**
Phase 4	**Demoulding**	**1 [h]**

Table 5.2. Times associated to the different production phases.

5.1.3 Visual description of the produced tubes

It might be useful to recall that the internal arrangement of the fibres chosen for the production of the three tubes, with the aim of evaluating the production process, presents three layers of fibres at 0°, with respect to the axis of the tube, starting from the inner part of the composite tube and 2 external layers placed at +/- 45° also with respect its axis. All of the final tubes present a rough external surface due to the choice of not using the peel ply, a texture generally used to obtain a smooth final surface.

The internal surface of the tube presents some tiny bubbles remained entrapped of few millimetres of diameter. By observing the section of the tube it is possible to appreciate the impregnation of the natural fibres, even though a more precise evaluation will be done thanks to the observation of some samples with the optic microscope.

5.2 Production of the comparative natural fibre tube

The tube, produced with filament winding and used as a comparison for the characterization of the developed technique, was obtained after a series of three attempts. In order to start with the production, it is necessary to program the machine by providing details on the desired product, like the number and order of layers, the angle of each layer, the thickness of the fibres, the desired length of the tube and the diameter of the mold. Such informations will allow the program to calculate the movements of the machine along the different axis.

5.2.1 Brief description of the attempts

The first try was characterized by the challenge to place the fibres of the first three layers at 0°, like for the tubes previously produced with the new process, this is usually not done with the filament winding technique. The placing of the fibres occurred almost with the desired angle, yet some other problems emerged that led to the understanding that an increase in the angle of the fibre was in order.



Fig. 5.2. Result of the first attempt in making a 0° fibres tube with filament winding technique.

The problems remarked consisted in a non-uniform placing of the fibres on the surface of the mold, resulting in some areas with a lot of fibres piled one over the other and other parts completely empty, it was due to the fact that the pins, placed at the extremities of the tube to fasten the fibres to the structure, were too distant one from the other and yet it was quite unavoidable because otherwise the eye of the machine would not have been able to differentiate in the positioning of one pin from the other. Proceeding with the placing of the external layer at +/-45°, the fibres were so distant from the surface of the mold, that by compacting them, they broke.

The second attempt was performed with an angle of the fibres of 5° instead of the previous 0°, the positioning and distribution of the fibres resulted visually much better in comparison with the previous case. The problem associated with this try was that the quantity of the mixture, resin plus hardener, that the fibres absorbed in the bath, before positioning, resulted not enough to fill the spaces in between the fibres.



Fig. 5.3. Result of the second attempt in making a tube with the filament winding technique.

The third attempt resulted quite satisfactory and was carried out like the second one, with the addition of a final step that consisted in spreading extra resin in the empty holes with the help of a paint brush. A representation of the different phases during the production and a picture of the final tube is provided in figure 5.4 and in 5.5.

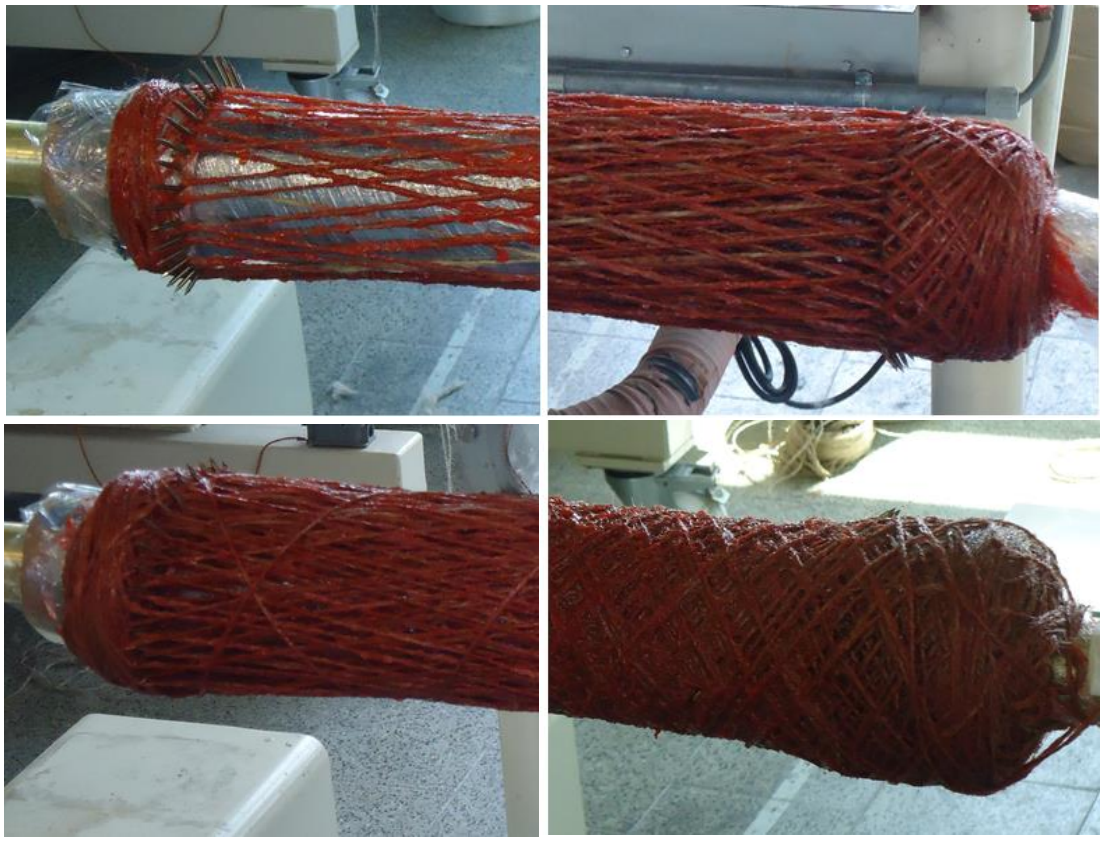


Fig. 5.4. Representation of the different stages of the placing of the fibres in the filament winding technique.

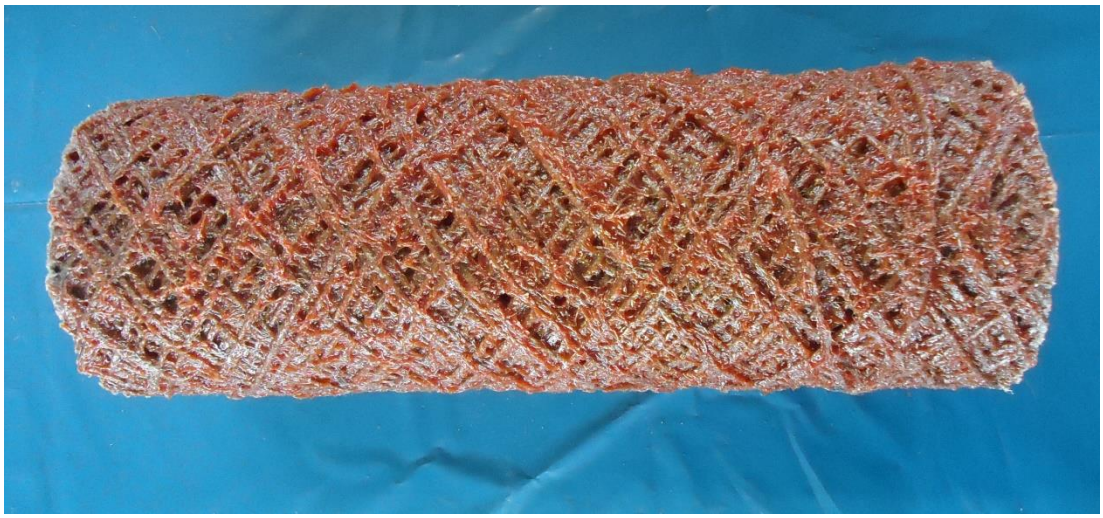


Fig. 5.5. Result of the third attempt in making a tube with the filament winding process.

5.2.2 Considerations

For all these attempts, the curing process of the resin, necessary to give rigidity to the matrix, was carried out at normal pressure and ambient temperature. Another aspect that it is worth mentioning is that the winding machine used is about 30 years old and it is reasonable to believe that newer winding machines would present a better positioning system and therefore better final products. Due to the availability of the machine, this is an aspect that cannot be changed and so it will not be considered further.

5.3 Mechanical characterization

Considering the initial requirements for a tube that could serve as a component for a bigger structure and that, as a loading condition, it would be subjected mainly to compression, it was decided to test the tubes just in compression where it would have been possible to appreciate the effect of the presence of 0° fibres. The two accessible machines for the compression tests, available in the laboratory of the Polymer Department of the Technical University of Clausthal and in the Mechanical Department of Politecnico, had a maximum load of 100kN and 250kN, respectively. To evaluate the load necessary to lead to the breaking of the tube , an attempt of a compression test was first made with one of the tubes produced during the development of the process with the machine from the Polymer Department of TU Clausthal. The maximum load was not enough even to obtain a significant strain in the tube. This led to the belief that also a 250 kN load would not have been able to break the tube. A testing of samples obtained from the tube itself was in order.

5.3.1 Preparation of the samples and settings

After a search for the right compression test standards, the lack of standards that would refer specifically to samples obtained from tubes and hence samples with a curved shaped surface, lead to the decision of not following exactly a specific standard. However the standard ISO 604, Plastics – Determination of compressive properties, was taken into consideration to maintain at least the same proportions between the upper and lower bases and the height of the samples as presented in such standard for parallelepiped specimens. A total number of 20 specimens, for each tube, were cut with the help of a band saw, the average dimensions were 15 mm of height and an average angle opening of 18°.

Each sample was successively measured with a calibre to calculate the area of the surface, an important value to determine the stresses once the test had provided the user with the strengths. Such calculation was carried out considering the curved sides as flat and as the major and minor basis of a trapezoid. In the figure 5.6, it is possible to see a picture of same samples cut from one of the natural fibre tubes. To recognize the three tubes, a label was placed on each one of them with a different colour, blue, green and red.



Fig. 5.6. Some specimens from the natural fibre tube with the red label.

The tests were carried out with a speed of testing of 5 mm/min and they did not require any tabs to keep the specimens in position since the upper and lower bases were cut with a lot of care in order to obtain parallel surfaces so that they could stand on the steel plate by themselves.

5.3.2 Compression data

In the figure 5.7 and 5.6, there are represented the Stress-Strain curves for the samples obtained from the squeeze winding tube, the one with the red label, and the curves of the samples from the filament winding tube. The curves of the samples from the other two produced tubes are not reported for the sake of brevity because they show the same trend of the one presented.

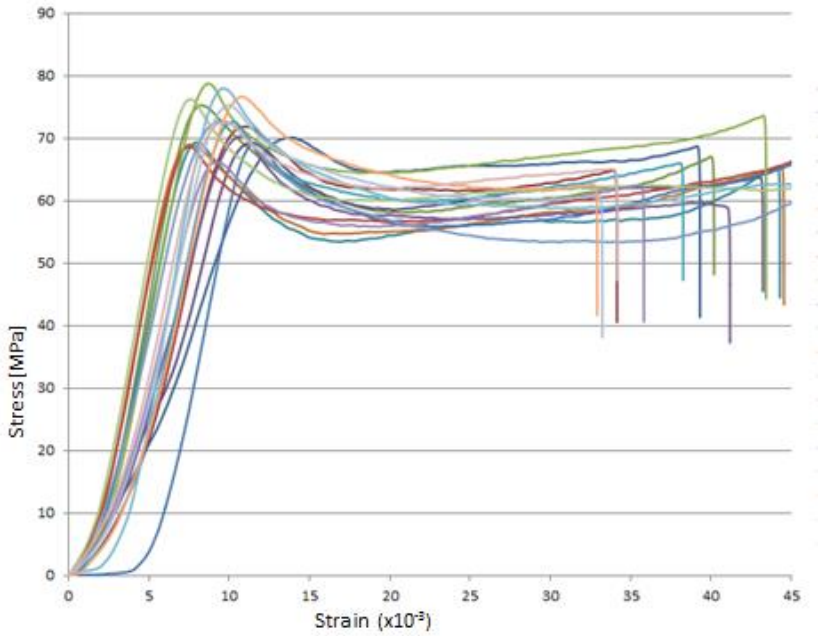


Fig. 5.7. Graphic representation of the stress-strain curve of the samples of the "red" tube.

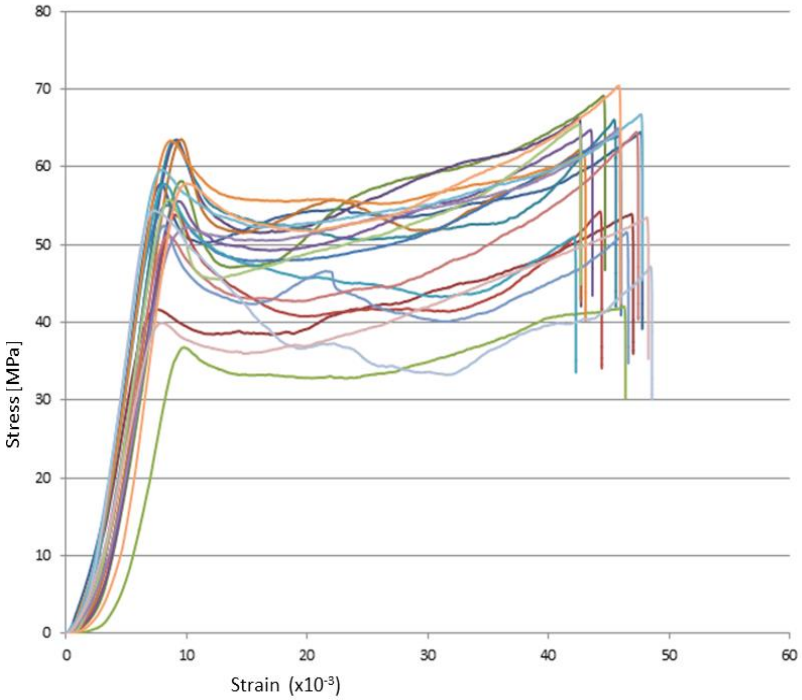


Fig. 5.8. Graphic representation of the stress-strain curve of the samples of the "winding" tube.

As it is possible to notice from the provided curves, the range that describes the elastic behaviour of the material, that embodies the left part of the diagram before the main peak, is quite homogenous, while the part on the right of the peak is more disorganized, it is strictly related to the breaking behaviour of the single specimen. In table 5.3 it is possible to see the elastic modulus and maximum stresses averaged on all the specimens tested for each tube.

	Average Max Stress [MPa]	Standard Deviation σ_{max} [MPa]	Average Elastic Modulus [MPa]	Standard Deviation E [Mpa]
Winding tube	**61,1**	**7,16**	**9858,3**	**1164,2**
Red Tube	**73,05**	**4,14**	**12173,7**	**1880,4**
BlueTube	**69,5**	**7,6**	**12017,1**	**1141,1**
Green Tube	**67,46**	**3,5**	**11502,1**	**1117,3**

Table 5.3. Average values of the mechanical behaviour of the material of the natural fibre tubes. blue, green and red colours refer to three tubes produced exactly the same way.

From the data of the compression tests, is it possible to observe that the values calculated for each tube differ no more than 5% between each other, both concerning the maximum stress and the average elastic modulus.

Another important observation that can be extracted from these data is a comparison of the values reported for the samples of tubes produced with the squeeze-winding process and the samples of the tube obtained with the filament winding technique. There is a clear improvement of the mechanical performances, indicated by an increase of both the maximum stress and the elastic modulus of around 20% which underlines the potential of the squeeze winding process.

5.3.3 Fracture behaviour of the samples

The tested specimens presented different fracture behaviour that affected their state after the test. This might be related to the fact that it is a manual process and there might be some differences in the fibre distribution or some extra defects of which it is hard to be aware of. In the following pictures it is possible to see some broken specimens.



Fig. 5.9 Example of a sample after the compression test.

5.4 Fibre Volume Content

As anticipated at the beginning of the chapter, the fibre volume content is an important parameter to account for the mechanical properties of the material produced. It is not a simple task to evaluate such a parameter, especially in the case of natural fibres where, for example, it is not possible to burn only the resin to obtain the fibres contained in it, as it can be done for glass fibres reinforced composites. The way chosen to accomplish such a task, is through the processing of images taken with a microscope (Keyence Corporation, Type: VHX-500K) of samples extracted from the tube. Even though this may seem the solution of the problem of evaluating the desired value, there are many possibilities that could be elaborated to reach a final range of values and each one of them enclosures some difficulties. A couple methods have been approached and, in the following of the chapter, a description will be presented along with the results obtained and the problems

encountered. Before describing such methods, it might be useful to say that the number of fibres placed in the structure has been counted after the placing, such value can be used to evaluate the desired quantity and it will be compared to the fibre volume content obtained with the image processing.

5.4.1 Black&White method

By considering that the composite material is mainly composed of two separate phases, the matrix and the fibre, the first method taken into consideration consists in processing the image, with the goal of obtaining a black colour for one phase of the material and the white colour for the other one. Once the picture is obtained, it can be easily analysed with a simple Matlab code that counts 1 for each white pixel and 0 for every black one. Dividing the number of one type of pixels for the total number of pixels contained in the image, it is possible to have the fraction of the phase that particular type of pixels was referring to.

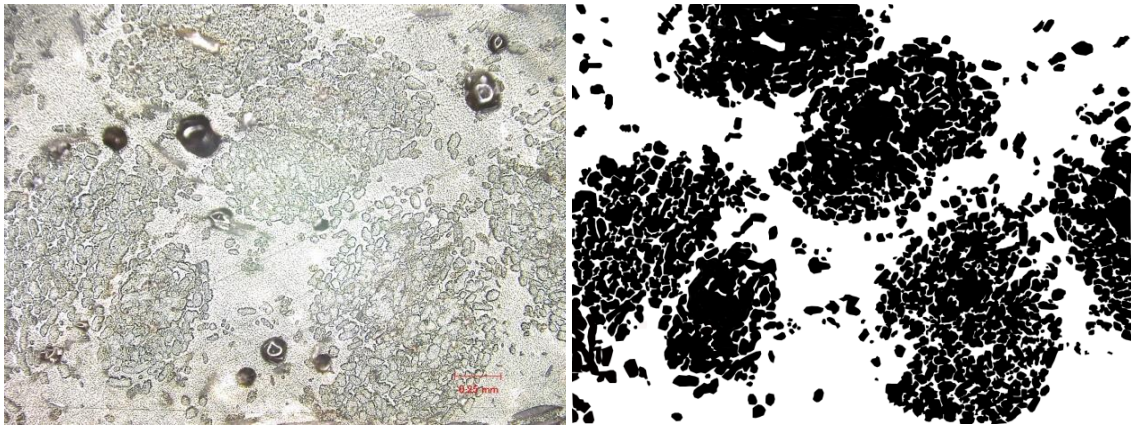


Fig. 5.10. Optic microscope picture and the elaborated image with GIMP.

The hard part of the method is obtaining such a black and white picture. The potential ways covered were both with Matlab, that allows you to select parts of a picture on a colour or shape base, or with an image elaboration program called GIMP, that presents several different tools that allow you to select particular areas. The main problem linked to this technique was that the pictures of the samples taken with the microscope lacked contrast, they generally looked like the same colour with slightly different shades that vary also through the picture. Natural fibres also present the further difficulty of being characterized by an irregular shape. It was not possible to obtain a fast and automatic method to

separate the two phases with an acceptable degree of tolerance. An attempt was made in selecting manually the fibre regions but it resulted too much time consuming so it was put aside. In figure 5.10, it is possible to see an example of how the picture was transformed into a binary one, really easy to process with Matlab, but it took around 8 hours to obtain it.

5.4.2 Grid Method

The grid method consists in applying to the image to analyse a grid with a dense mesh and count the number of points where the lines meet over the fibre. By dividing the number of points on the fibres for the number of total points in the image, it is possible to obtain a good estimation of the fibre volume content.

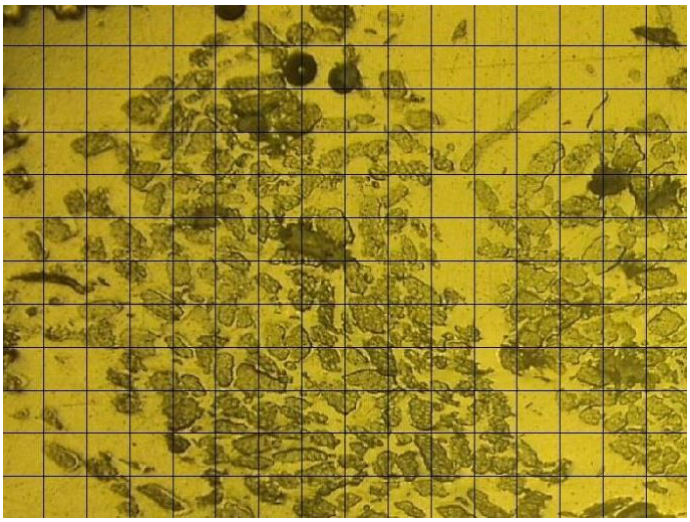


Fig. 5.11. Example of picture analysed with the grid method.

Since it often happens that a point is visually placed exactly in between the fibre and the matrix, which makes it difficult to establish whether it belongs to one phase or the other, and also counting the points manually it is quite time taking and easily subjected to error, it was attempted to program Matlab to automatically choose to which phase the point belonged on a colour base and to turn that point into a specific colour if it was matrix and another if it was resin, this would have given the user the possibility to understand how reliable the code was. The result was not successful because some areas were totally mistaken for the other phase. The poor contrast of the image was at the base of this problem and attempts, still not successful, were made with the microscope settings and the use of different types of lens.

5.4.3 Results of the image processing

What was adopted, in the end, to evaluate the fibre volume content, was to consider 2 values in counting the points, a maximum and a minimum value, obtained counted 1 for the maximum value and 0 for a minimum value in the eventuality of meeting a not clear point of the mesh. These two values would give a reasonable range within which the fibre volume content had to be contained. Each picture analysed was divided in 4 parts to be able to increase the number of total nod of the mesh. The dimension of each single portion of image was 600x800 pixel and a nod was placed every 50 pixel, considering four part each picture, it makes a total of 768 nods per picture. 10 pictures were analysed for every tube.

	Calculated with the number of fibre %	Range obtained with Image Processing %	
Winding tube	/	27,5	33
Red Tube	47,37	45,5	52,5
BlueTube	46,82	43,5	50
Green Tube	42,14	41	49,5

Table 5.4. Presentation of the data obtained in the two methods of the fibre volume content.

As it is possible to notice from Table 5.4, the values calculated for the squeeze winding tubes fall in the range of the values obtained with the grid method which gives it a good reliability. Even considering the highest value of the fibre volume content for the tube produced with the filament winding technique, an increase higher than 30% of the quantity of fibres is acknowledged.

5.5 Main characteristics of the developed technique

Starting from the negative aspects of the process at the state of art, they mainly are the long fibre positioning times and the presence of small bubbles on the inside of the tube and on both surfaces. A switch to an automatized method both for the first phase and the appliance of the torque would be advisable. The squeeze winding process developed in the first part of the project allowed us to obtain tubes made with a material that presented a fibre volume content of 45% average, this value resulted higher than the one obtained from the material of the filament wound tube, as comparative, of about 30%.

The mechanical properties of both the materials were tested in compression and an increase both of the load at breaking and of the elastic modulus was registered, of respectively 15% and 20%. Such improvements of the mechanical properties can be explained both because of the fibre volume content and the presence of 0° fibre that this technique allows. Another aspect that is worth mentioning is that the impregnation of the fibres is quite good and can be observed in the pictures obtained with the optic microscope, and example is given in figure 5.12, along with the presence of some bubbles. A good reproducibility of the process was observed which allowed to obtain values of the mechanical properties and of the fibre volume content within a close range. A final aspect to underline is that the desired value of fibre volume content was around 60%, a possible reason why it was not possible to obtain it, it is link to the nature of natural fibres that tend to relax and elongate when put under tension. It is reasonable to think that artificial fibres would be better suited for the task.

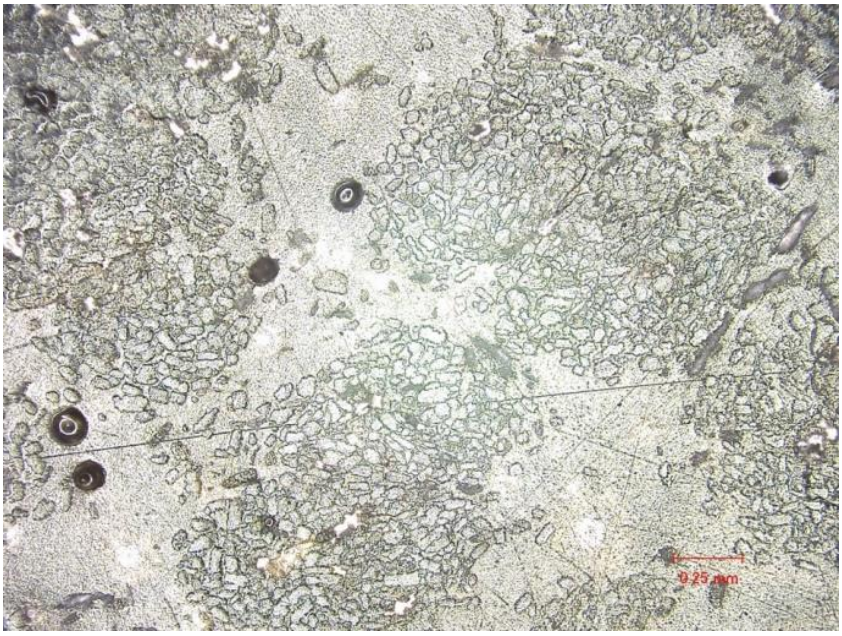


Fig. 5.12. Optic microscope picture of a natural fibre sample.

Chapter 6.
Design and Production of the Bioinspired Composite Tube

The previous part of this thesis' work concerned with the developing of a manufacturing technique that would be suitable to obtain a tubular composite with a good level of fibre volume content and fibres placed longitudinally to the axis of the tube. The Squeeze-Winding process has been successively put to the test with a different type of fibres and a specific internal design. It is important to underline that the research activity, carried out during these eight months in the Institute of Polymer Materials and Plastic Engineering of Clausthal, takes its roots from previous works characterized by the attempt to reproduce some of the toughening mechanisms of cortical bone. In order to do that, they focused on the production of laminates with an arrangement of fibres aimed at recreating the osteonic structure of bone, described in the Chapter 2, at a bigger scale due to technology limitations. This project takes the concepts understood from the process of optimization of the design of the internal structure and reapplies them to the production, with the developed process, of a tubular composite with the such structured internal arrangement. The final goal is to obtain a component that could provide interesting performance from a compression point of view, being bone mainly subjected to compression. Throughout this chapter, a detailed description of the design and production phases will be presented and analysed, taking into account positive and negative aspects of both the choice of arrangement of the fibres and the limits and potentialities of the technique.

6.1 Framework of the project

The concept behind the osteons is that they lead to interesting toughening mechanisms in the cortical bone for example by deviating the crack around their cylindrical surface, thus increasing the length of the crack path and hence the energy necessary for the crack to proceed. This is just one mechanism that can be cited and they are described in a more complete way in the chapter of the Introduction to Bone. By extracting the design ideas that nature evolved and applying them to the production of a composite material, an increase in the material's performance could be pursued. It is important to underline that long bones, like for example the femur, are subjected to a load that is mainly characterized by compression and bending, this is another reason why such natural material is taken as a source of inspiration for the development of the tubular component with the desired properties. Bone evelved through evolution to fulfil at its best the requirements imposed by the external loads to which it is subjected. In the picture below, figure 6.1, it is possible to notice a repetitive circular structure, constituted of an external sleeve of +/-45° carbon fibres, filled with unidirectional glass fibre, of few millimetres of diameter representing the osteonic structure described in chapter 2. Such structure is wrapped around a glass fibre fabric, observed to increase the transversal mechanical properties of the material and favour the compaction of the layers. This internal structure is the arrival point of the research projects that proceeded this work and constitutes its starting point for the design of the internal fibre structure of the composite tube to produce.



Fig. 6.1. Representation of the bioinspired laminate produced by previous research works [15].

6.2 Design ad hoc for the tubular structure

Considering the desired shape, a development of a design for the arrangement for the fibres was in order. The requirements taken into consideration were the placing at 0° of the structures representing the osteons and the positioning of a single layer of fabric that would wind around the layers, alternating from one osteon to the other, with the aim of increasing the transversal properties of the material and ease the compaction of the several layers. The number of layers chosen in the first part of the project, with natural fibres, will be re-proposed in this design phase. The drawing of the section of the tube presented in figure 6.2, has the goal of clarifying the previous description indicating all the components used inside the bioinspired composite material.

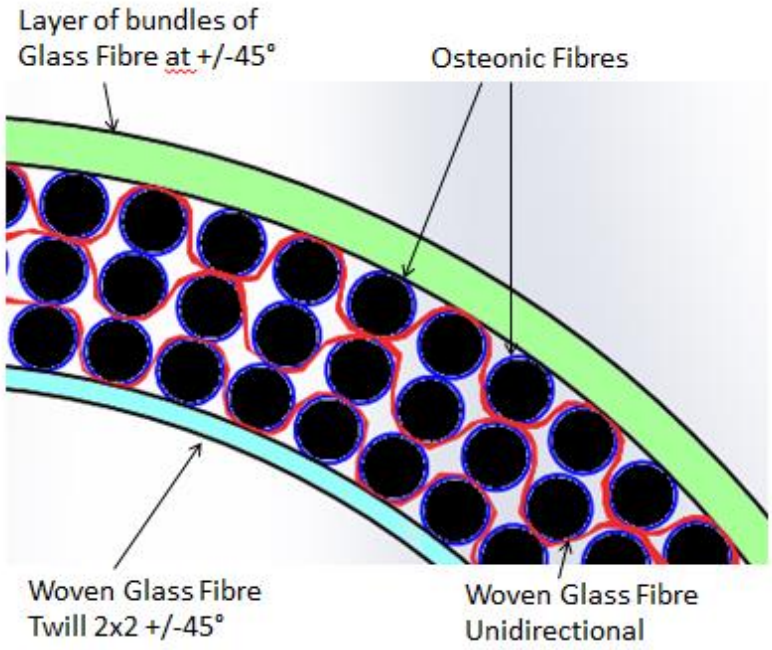


Fig. 6.2. Drawing of the design of a part of the section of the bioinspired tube to produce with indicated all the reinforcement components listed below.

There will also be two external layers for the appliance of the torque, where there are placed bundles of glass fibres, they are presented in the external layer of the drawing in figure 6.2.

6.3 Description of the components

Once decided how to arrange the structure it becomes necessary to define the components chosen for that arrangement. The main element that constitutes the bioinspired composite tube, the same one used in the previous research project [15], is the sleeve, of 2,5 mm of diameter, constituted of +/-45° carbon fibres filled with unidirectional glass fibres. In picture 6.3, it is possible to see a representation of what will be called from now on the osteonic fibre.



Fig. 6.3. Osteonic fibre: the rowing of Glass Fibre and the sleeve of Twill 2x2 Carbon Fibre[15].

The fibre fabric used to roll up the osteonic fibre is made of unidirectional glass fibres placed at 90° with respect to the axis of the tube in the making. A picture of such component is resented in figure 6.4. Another woven of glass fibre oriented +/-45°, with respect to the axis of the tube, was used to cover the surface of the mold in order to obtain a better finishing surface and increase the quantity of non 0° fibre for the transversal properties.

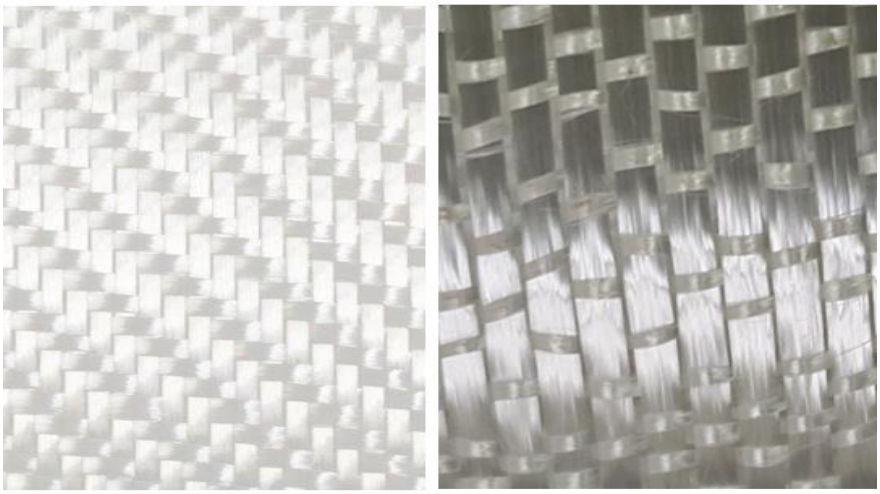


Fig. 6.4. Representation of Twill 2x2 Glass Fibre Woven, on the left, and Unidirectional Glass Fibre Woven of the right.

As far as it concerns the two external layers of fibres, necessary for the chosen technique to provide the desired compaction of the whole structure and the resulting "squeezing" effect, bundles of glass fibres were chosen. To the disk to which they would be fastened, a torque would be applied and the fibres would compact the underlying ones by been wrapped around them. In table 6.1 it is possible to find a summary of all the components with their specific characteristics.

Osteonic Fibre	**Glass Fibre**	**Unidirectional**	**1,2 [g/m]**
Osteonic Fibre	**Carbon Fibre**	**Twill 2x2 +/-45°**	**3,4 [g/m]**
Bundles +/-45°	**Glass Fibre**	**Unidirectional**	**2,4 [g/m]**
Internal woven	**Glass Fibre**	**Unidirectional**	**220 [g/m^2]**
Internal surface woven	**Glass Fibre**	**Twill 2x2 +/-45°**	**300 [g/m^2]**

Table 6.1. Characteristics of the reinforcement components.

The matrix used for the production of this set of tubes is the same one used for the previous natural tubes, epoxy resin RIMR 135 and the hardener RIMH 137.

6.4 Production phase

As anticipated at the beginning of the chapter, the method used to carry out the production of the bioinspired composite tubes is the same one described in the two previous chapters, the squeeze-winding process.

6.4.1 Modifications and first try

The placing of the bundles of fibres through the metal rings resulted quite problematic from the very beginning. Such bundles, unlike a thread of Jute, easily tend to open up and their sliding inside the rings accentuated it. It was clear that a change in the way the bundles were placed was in order. For the three internal layers a solution was provided by using the pins, the ones used in the filament winding technique, at the edges of the tube. This idea not only bypassed the problem of the breaking of the glass fibre for those three layers but also allowed to save a lot of time for the placing, in the previous case it was about 1 hour and a half for each layer while this way is was only 1 hour for all the three layers. A picture is provided to account for the change proposed, fig. 6.5.



Fig. 6.5. Pins placed on the plugs to hold the osteonic fibres at 0°.

The fibre fabric was placed during the positioning of the osteonic ones, being careful to alternate the fibre up and down of the fabric to give the desired effect. In figure 6.6, it is possible to see the fibre after positioning.



Fig. 6.6. The osteonic fibres alternating up and down the glass fibre fabric.

The picture represents one end of the tube and, of course, the edges have been cut after the production. The layer whose fibre resulted at +45° at the end of the process, the ones attached to the middle disk, were placed with the original method, so extra care was put into their positioning and it took around 2 hours to complete it. The last layer at -45° was laid down after the impregnation of the structure with the resin and it took around 45 minutes to complete it. The rest of the process was carried out as described previously in the chapter about the technique.

6.4.2 Observations and second try

The first tube produced, clearly showed in the section that it was harder than expected to obtain the required wavy trend of the fabric around he osteonic fibres. This was due to the fact that they could oscillate from their original position a little bit and that the fibres from one layer could easily get together with the ones of the layer above or below. It resulted in an alternation of the fabric of 3 or 4 per wave. The second tube was characterized by a careful fixing of the osteonic fibres at the edges with a round of tape per layer. The rest of the process was performed in the same manner as the previous tube. In the picture below, figure 6.7, it is possible to see a portion of the section of the first tube while the second one is not reported because it looks exactly as the first one.

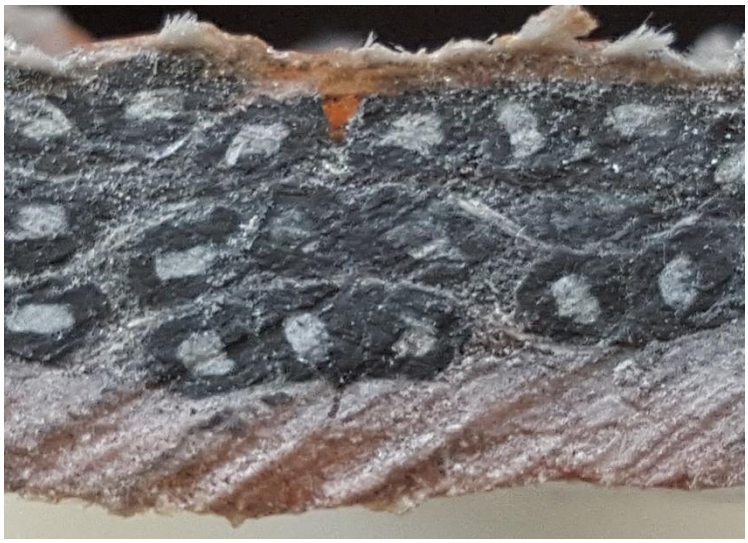


Fig. 6.7. Portion of the section of the first bioinspired composite tube.

6.5 Comparative tubes

Comparative tubes are necessary to evaluate the effectiveness of the bioinspired internal structure. In order to be comparable they have to present the same type of fibres, carbon and glass, and in the same quantities.

6.5.1 Design of the comparative structure

A careful design was in order to guarantee the conditions above-mentioned. The carbon fibre sleeves have been replaced with the same quantity of carbon fibre but in the form of carbon plain woven with the fibre placed at +/-45°. The unidirectional glass fibres contained in the carbon sleeves have been replaced with bundles of fibre in the same quantity. In the table below it is possible to have a summary of the components of the comparative structure.

Bundles 0°	**Glass Fibre**	**Unidirectional**	**1,2 [g/m]**
Carbor Fibre woven	**Carbon Fibre**	**Plain +/-45°**	**600 [g/m^2]**
Bundels +/-45°	**Glass Fibre**	**Unidirectional**	**2,4 [g/m]**
Internal woven	**Glass Fibre**	**Unidirectional**	**220 [g/m^2]**
Internal surface woven	**Glass Fibre**	**Twill 2x2 +/-45°**	**300 [g/m^2]**

Table 6.2 Schematic summary of the comparative structure components.

6.5.2 Production of the comparative tube

Once that all the fibres were placed, it was proceeded to the impregnation phase of the process. An important observation emerged, at the appliance of the torque, with the compressing by the external layer of the inner fibres, the woven within the structure started to show a wavy behaviour that resulted in an inhomogeneity of the section of the tube. This effect can be noticed both on the surface of the comparative tube and on its section, see figure 6.8 and 6.9 for a better visual understanding. For the production of the second comparative tube it was attempted to wrap the two fabrics more towards the inner part of the tube in order to see if it was sufficient to avoid the wavy trend by having more fibre compressing it. Unfortunately the same surface resulted on the second tube. The problem could be noticed also on the bioinspired tube, but on a much smaller scale because of the fact that there was less fabric and it was only the glass fibre one, which appeared to be more flexible than the carbon fibre woven.

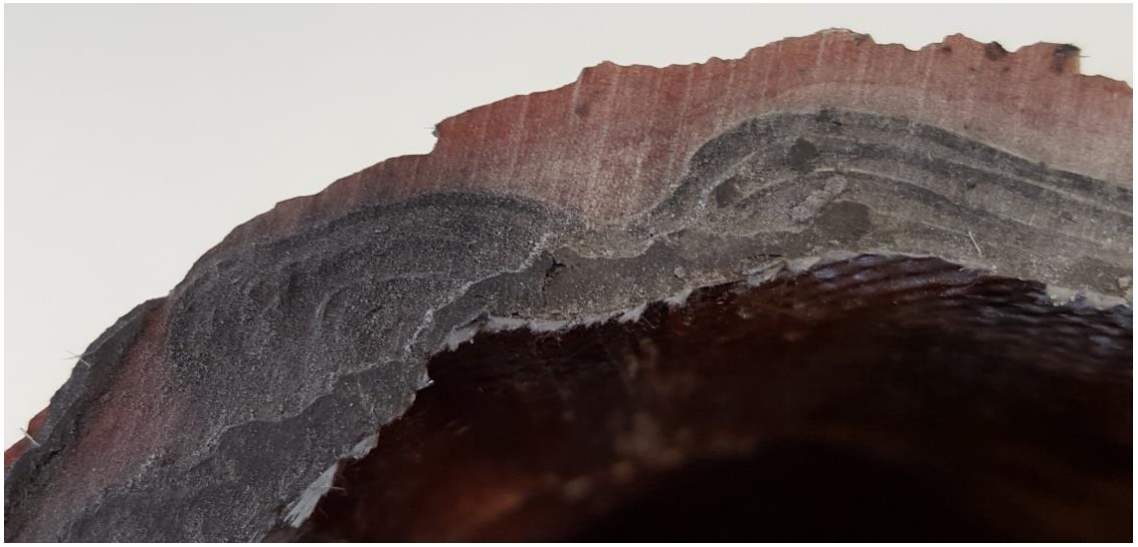


Fig. 6.8. Portion of the section of the first comparative tube produced.



Fig. 6.9. Picture of the two types of tube produced, the bioinspired on the left and the comparative of the right.

This experiment allowed to understand a limit of the technique that had not been taken into consideration before the production of such tubes. The squeeze winding process is not suitable in the presence of fabrics within the structure. This is an important aspect that needs to be taken into account in the design phase for the production of a desired piece.

6.6 Production of a last bioinspired tube

One last tube was produced to take into account the observation made about the presence of a fabric inside and therefore it was produced without the unidirectional glass fibre woven. As expected the final surface is very smooth and the section presents a distribution more regular and homogeneous as it is possible to see in figure 6.10.

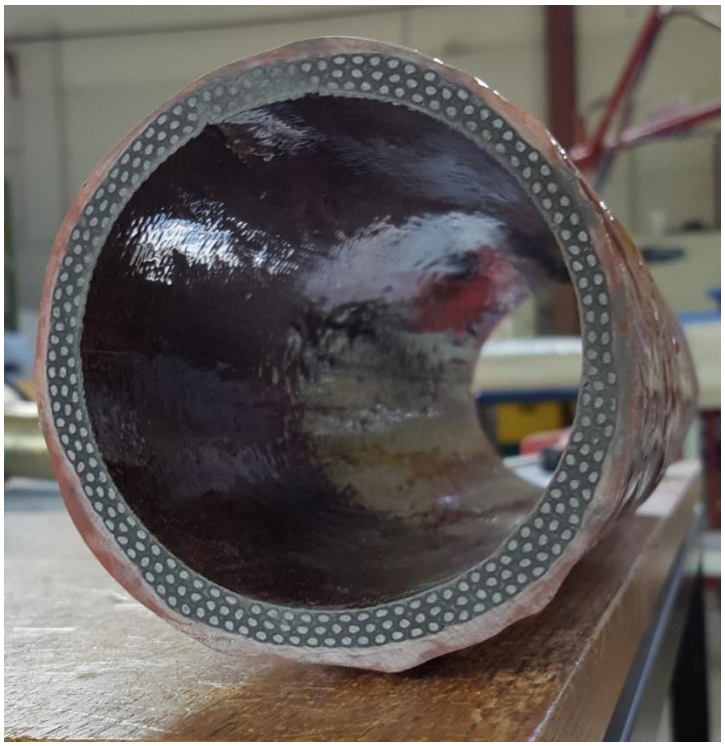


Fig. 6.10. Picture of the tube composed only of osteonic fibres.

Chapter 7.
Characterization of the Artificial Fibre Composite Materials

The production phase of the second part of the project permitted to obtain a total of 5 tubes, two of them presented the internal structure characterized by the presence of the so called Osteonic fibres and a glass fibre woven wound around them. Two other tubes, called comparative, were manufactured with simple glass fibre bundles and a carbon fabric thought to provide for carbon component in the bioinspired tube. One last tube only with Osteonic fibres and no fabric was created in order to test an observation that presented during the production of the other four tubes. Once produced, a characterization of the second set of tubes was in order. The tests were performed with the testing machine available at the Institute of Polymer Materials and Plastic Engineering. A further investigation was carried out evaluating the fibre volume content of all the samples extracted from the tubes, this gave the possibility to propose more accurate considerations basing them on the two parameters and comparisons between the values of the different tubes, especially the ones with a different internal structure. This part of the process which constitute the last practical experience is fundamental to recollect of the concepts and deliver the final outcomes of the project and it will be described throughout this chapter.

7.1 Mechanical Tests

The type of mechanical test selected to characterize the material of which the tube is made is compression. Such choice was led by the requirement given for the goal of the project, which consists in the realization of a tubular component with good performances in compression. Such a tube would successively become part of a bigger structure, for example a first idea was a C-Frame structure used as frame for many tools present in a workshop.

7.1.1 Specimens

The mechanical tests were carried out on samples obtained from the tube due to the loading limits imposed by the machine availability. The specimens were cut with the use of a saw band and were obtained from a ring coming from the tube. A total number of 16 specimens were tested with average dimensions of 20 mm in height, a thickness of 9 mm and an average angular opening of 22.5°. Due to the shape of the samples, it was not possible to follow in details a specific standard but the normative ISO 604 was taken into account as a reference for the proportions of the bases of the specimens and the height. The specimens were cut with the most care to obtain parallel basis so that they could stand by themselves. In the picture below it is possible to see some of the specimen from the three types of tube, ready to be tested.



Fig. 7.1. Representation of the specimens, one for each type of tube respectively, bioinspired, comparative and bioinspired without fabric.

7.1.2 Testing Procedure

The specimens were tested with a speed of testing of 5 mm/min until breaking. The curves of stress and strain are reported in figure 7.2, 7.3, 7.4, 7.5, 7.6 below.

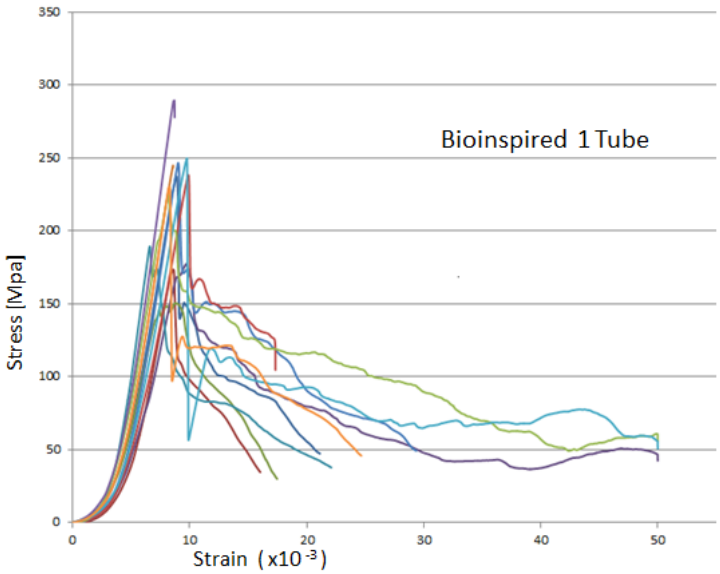


Fig. 7.2 Stress-Strain Curve of the specimens from the first Bioinspired tube.

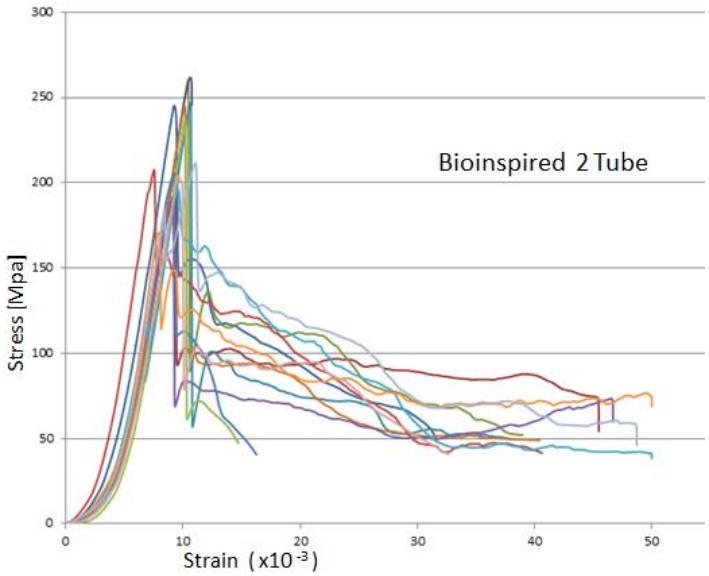


Fig. 7.3 Stress-Strain Curve of the specimens from the second Bioinspired tube.

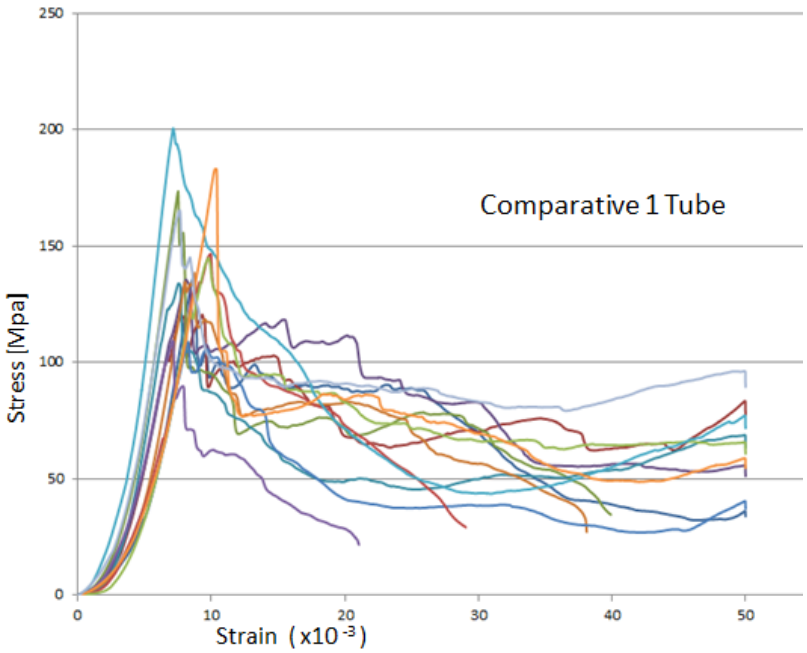


Fig. 7.4 Stress-Strain Curve of the specimens from the first Comparative tube.

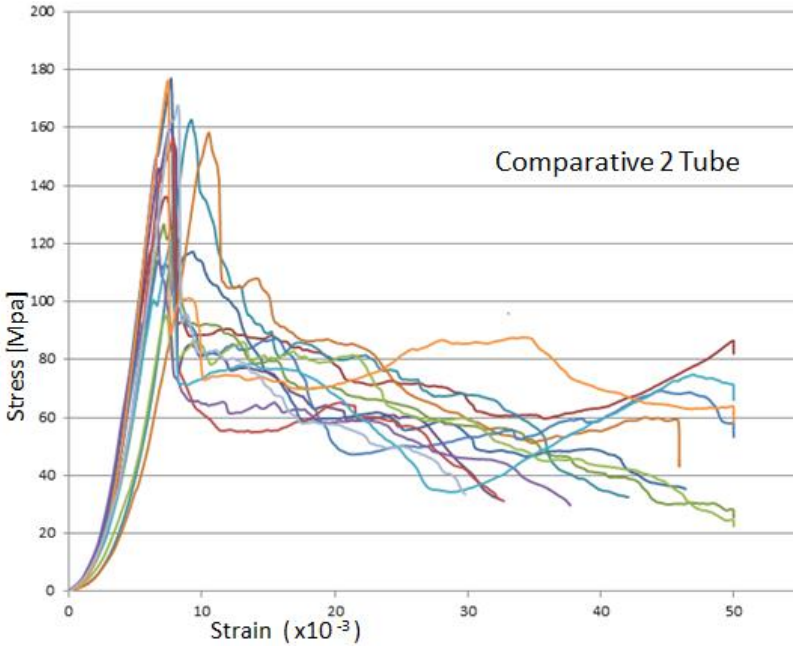


Fig. 7.5 Stress-Strain Curve of the specimens from the second Comparative tube.

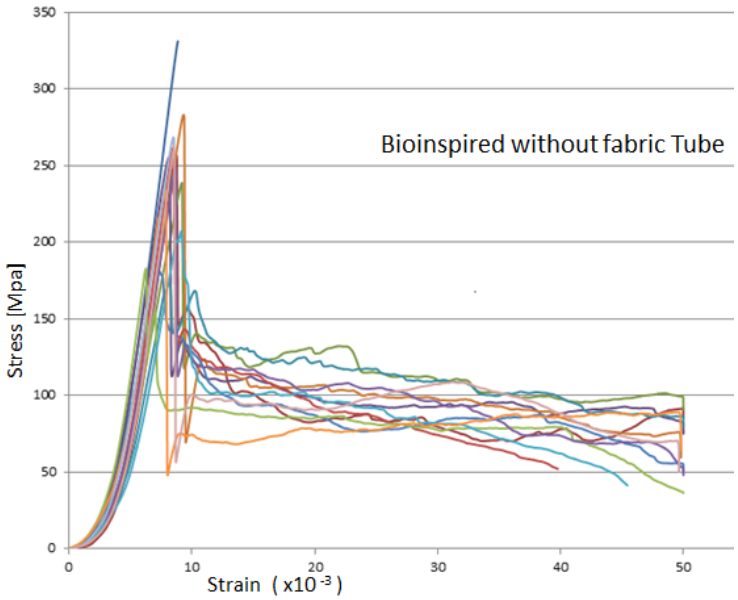


Fig. 7.6. Stress-Strain Curve of the specimens from the Bioinspired without fabric tube.

7.1.3 Data Analysis

From the mechanical test data, it is possible to extract some important parameters, which will help understanding the compression performances of the materials that constitute the tubes. A first value is the maximum stress at yield, after which an increase in strain occurs without an increase in stress. The other important parameter that can be extracted from such data is the modulus of elasticity, E, which can be defined as a measure of the resistance of a material to being elastically deformed. The values obtained for each specimen were averaged over all the 16 samples per tube. Few values extremely out of the range given by the standard deviation were not taken into account for the evaluation of the Elastic Modulus and the Maximum Stress at Yield. In the following table 7.1, it is possible to find a summary of the above –mentioned values and their standard deviation associated to each type of produced tube. As it is possible to notice, the material that presents the higher value of both the maximum stress at strain and elastic modulus is the one associated to the tube produced without the internal fabric around the osteonic fibres.

Tube's Material	σ max [MPa]	StanDev σmax	E [MPa]	StanDev E
Bioinspired 1	**222,92**	**44,88**	**43082,12**	**8591,63**
Bioinspired 2	**213,18**	**32,95**	**41111,7**	**3908,74**
Comparative 1	**143,25**	**40,4**	**30844,15**	**5925**
Comparative 2	**137,31**	**30,68**	**29501,73**	**6000,69**
Bioinspired noFabric	**234,42**	**45,22**	**50066,14**	**7447,34**

Table 7.1 Summary of the Compression Properties associated to the materials composing the produced tubes.

Some pictures of the samples after the compression tests can be provided and it is possible to observe that they broke in different manners.



Fig. 7.7. Some of the specimens after the compression test.

7.2 Image processing

The image processing carried out with this type of fibres is based on the idea that there is a macro flux of the resin, that flows around the bundles of fibre, and a micro flux which allows the resin to penetrate within the bundles and surround the single glass or carbon fibre.

7.2.1 Micro Flux of the resin

This part of the image processing phase had the goal of determining the percentage of resin and fibre within the bundles, both for carbon and glass fibre. Microscopic pictures were taken with an optic magnification of 200x and it was possible to observe both types of fibres. It is worth mentioning that glass and carbon fibres are easily recognized between themselves because of the dimensions mainly and because glass fibres presented a section almost circular while the carbon fibre showed a more oval shape. In all cases, both glass and carbon fibre are much easier to select in comparison with natural fibres where this method was not applied due to the difficulties encountered in separating the natural fibre phase from the matrix one. In picture 7.8, it is possible to see one of the 20 pictures taken with the optical microscope and processed in order to obtain the Black&White image in the right part of the same picture.

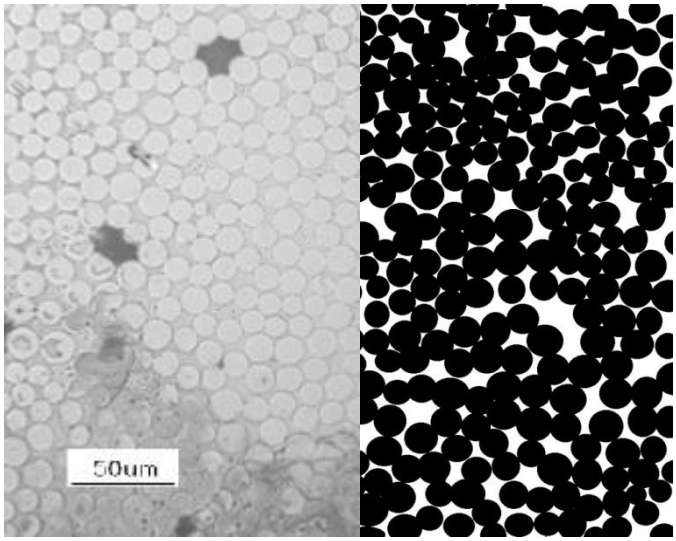


Fig. 7.8. Optic microscopic picture of glass fibres and the B&W equivalent within a sample of the produced tube.

As it can easily be noticed, the white parts corresponds to the matrix and the black one to the fibres, such has been achieved, selecting on the originals, the parts where the fibres are present. Such a picture is suitable to be processed with matlab because a code can program matlab to count 1 for a white pixel and 0 for a black one. What can be obtained is the percentage of matrix flown in the bundles which will be used to acquire the fibre volume content.

The black spots visible in the original picture are areas where the impregnation was not successful and what is left is a void between the single fibres. The average percentage calculated of glass fibre in the bundle is 75% and this is the value used in the following for the fibre volume content. The uncertainty associated to this value is +/- 5% . It has been calculated taking into account all the values from the 20 pictures analysed and it is worth saying that it was not obtained a value lower that 70% and higher than 80%, so it can be considered a reliable interval. The same process was carried out for carbon fibres and, in figure 7.9. It is possible to see an optical microscope picture and the equivalent black and white version. The average value obtained for carbon fibre was in average 60% and it presented a wider oscillating interval, between 50% and 70%. Those two values were the one pursued in this analysis of the micro flux.

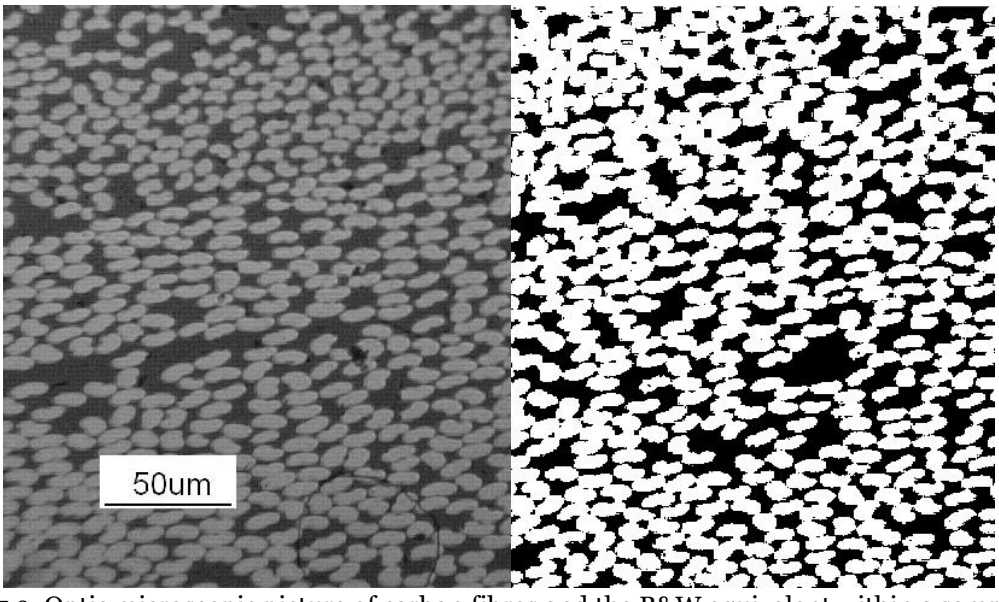


Fig. 7.9. Optic microscopic picture of carbon fibres and the B&W equivalent within a sample of the produced tube.

7.2.2 Macro Flux of the resin

In the analysis of the macro flux, the goal is to obtain the percentage of bundles for both glass and carbon. Once obtained such values, they will be multiplied respectively by the fraction of fibre calculated in the micro flux section. In order to achieve such task, the bundles of glass and carbon fibres have been isolated, separately, in each picture using the software Gimp. In figure 7.10, it is possible to see an example of the black and white pictures obtained from the last produced tube.

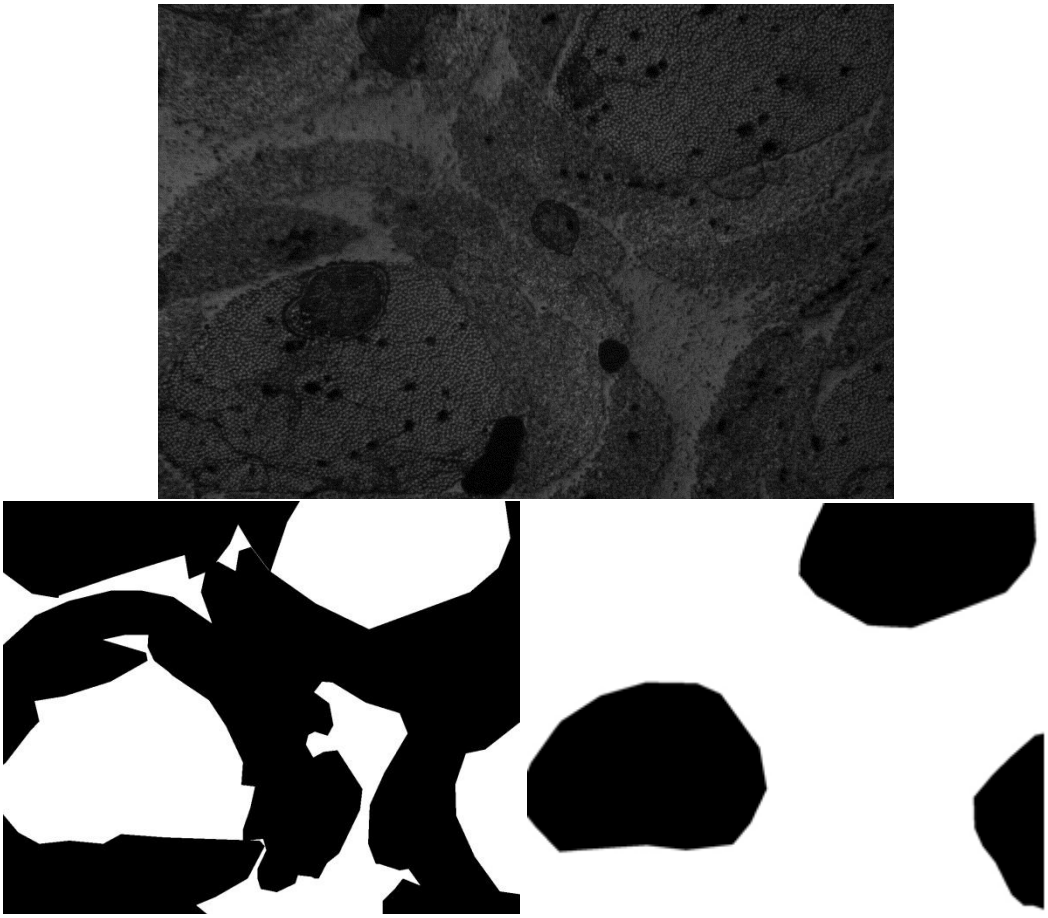


Fig. 7.10. Representation of an optic microscopic picture of a sample taken from the bioinspired without fabric tube, a B&W picture with carbon fibre in black and a B&W picture with glass fibre in black.

The results of this image processing, quite time taking, is presented in table 7.2 with the average fibre volume content associated to each produced tube. The choice of representing the final results instead of the intermediate ones is preferred for the sake of brevity. It is important to underline that the values reported in the first column represent on average of all the ones obtained for each tube, while the values in the other two columns represent the maximum and minimum registered values, rounded off, for each specific tube.

	Fibre Volume content %	Range of validity %	
Bioinspired 1st Tube	**56,12**	**51**	**60**
Bioinspired 2nd Tube	**54,86**	**49**	**56**
Comparative 1st Tube	**58,754**	**57**	**62**
Comparative 2 Tube's Material	**57,92**	**54**	**63**
Bioinspired noFabric Tube	**59,77**	**58**	**62**

Table 7.2 Summary of the values of fibre volume content obtained for the produced tubes.

7.3 Considerations

It is very interesting to notice that the values of fibre volume content evaluated for all the tubes are contained in the range between 54% and 60%, which is not wide, and yet the compression properties of the material extracted from the bioinspired tubes present an increse of more than 40% of the elastic modulus and more than 50% of the maximum stress at yield with respect to the material of the comparative tubes. Such difference can be attributed to the different internal structure that characterize the bioinspired tubes. The removal of the glass woven resulted in a good choice because it allowed to obtain a better compaction and hence a higher fibre volme fraction, therefore better mechanical performance compared to the previous produced tubes.

Chapter 8.
Conclusions and Outlook

The project started with the aim of manufacturing a composite tube that could provide good compression performances useful to be included as component for the creation of a more complex frame, common for several tools present in a workshop. This work is on a research level and constitutes a first step in the realization of such structure. The internal arrangement of fibres chosen in the design phase derives from previous projects conducted in the Mechanical Department of Politecnico di Milano. The work carried out during the eight moths spent in the Institute of Polymer Materials and Plastic Technology based in Clausthal-Zellerfeld, has been composed of two main parts: the first one was focused on developing the manufacturing technique that could permit to fulfil certain requirements; the second one has been characterized by the arrangement of the internal design and components, the production and the characterization of the material of which the tubes were constituted. Interesting results have emerged from both parts of the project. As far as it concerns the manufacturing technique, the Squeeze-Winding process, it has been possible to produce tubes that presented an increase in the fibre volume content, using natural fibres ,of more than 30 % compared to the tubes produced with the well-known Filament-Winding technique. Such difference affected the mechanical properties of the material constituting the produced tubes, which showed an increase of the maximum stress and the elastic modulus of both around 20%. The squeeze-winding technique did not allow, with natural fibres, to obtain the desired fibre volume content of 60% which is the typical value of bone material. This was attributed to the tendency of natural fibres to relax when put under tension. The manufacturing process was therefore applied to the designed bioinspired arrangement of glass and carbon fibres. This phase of the project resulted with the production of 5 tubes, two bioinspired ones, two comparative ones and one bioinspired tube without the internal glass fibre woven.

The range of fibre volume content for all the tubes produced, resulted between 54% and 60%, the one with the higher average fibre volume content was the last one produced without the fabric. This was a confirmation that the squeeze winding process gives better results if no fabric placed inside, as it appeared clear by observing the final external surface of the comparative tubes which presented a wavy trend due to the compaction of the fibre fabric. The demonstration of the effectiveness of the Osteonic internal arrangement came from the compression tests performed on specimens extracted from the tubes. The material from the Bioinspired tube with no fabric inside, showed an elastic modulus almost double than the material from the comparative tubes even though the fibre volume content resulted differing only few points percentage.

Successive steps suggested to continue this line of research may start from the testing of the tubes regarding their integrity. This might lead to a better characterization of the tubes. In fact, since only specimen from them were tested, some differences in the mechanical behaviour might, for example, be expected due to the fact that cutting the tubes longitudinally lead to an inevitable breaking of the thin fibre tubes that aimed to mimic the osteons of the cortical bone, leading to an embrittlement of the material on the sides. In addition, it can be underlined that the width was kept small, almost the same dimension of the thickness of the tube, in order to have an almost square surface. Evaluation of the K_{ic} and toughening mechanisms could be interesting. Another interesting aspect would be to find a way to combine the squeezing part of the developed technique with the appliance of vacuum, to reduce the number of air entrapments present in the final product. A future line of research should build such frame with the described tubes as components and evaluate the maximum load that it would be able to sustain.



Fig. 8.1. A comparison between a representation of the osteonic structure of cortical bone and the internal arrangement of fibre in the produced tube.

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