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Enhancing the Durability of Composite Systems

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2022

Dissertation submitted to the Faculty of Engineering of the University of Porto for the Master Program in Manufacturing

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Abstract

The aim of this thesis is to address the increase in demand of composite materials and their usage in structural applications with ways to enhance the long-term durability of these systems, which is their main limitation at the moment. To accomplish this, natural age-regulating mechanisms in animals and plants were closely studied, to infer how they function and how they can eventually be applied to extend the durability of composites. The main theory that was used to support these conclusions was the free radical theory of ageing. Finally, an investigation methodology is suggested that may serve as basis for future works, and possibly help conceive new composite systems with enhanced durability. This methodology would consist of exploring two suggested ideas based on the reviewed bibliography, one referring to stomata in plants and the other one to the characteristics existent in rocks that reduce creep behaviour.

Sumário

O objetivo desta tese passa por abordar o aumento da procura em materiais compósitos e a sua utilização em aplicações estruturais com formas de aumentar a durabilidade a longo prazo desses sistemas, que é sua principal limitação no momento. Para isso, os mecanismos existentes de regulação do envelhecimento de animas e plantas foram estudados, de modo a perceber o seu funcionamento e de que maneira podem ser aplicados para aumentar a durabilidade dos compósitos. A principal teoria de envelhecimento que foi usada como apoio para as conclusões retiradas foi a teoria dos radicais livres. Por fim, sugere-se uma metodologia de investigação que possa servir de base para futuros trabalhos, e possivelmente ajudar a desenvolver um novo sistema compósito que exiba maior durabilidade. Esta metodologia consistiria em explorar duas ideias retiradas da revisão bibliográfica, onde uma refere-se ao estómato presente em plantas e uma outra que se fundamenta na capacidade das pedras de reduzir o comportamento à fluência.

Acknowledgements

I would like to express my deepest appreciation to Prof. António Torres Marques for his valuable patience and feedback throughout the development of this paper. His worthy guidance and expertise were very appreciated in completing this dissertation. I am also thankful to Prof. Albertino Arteiro for his criticism and help on the last few days making sure that the thesis is well presented, objective and structured. A special appreciation message to my good friend Bernardo Picão that during these last few days has taken is time and kindness to review my paper.

I am thankful for my classmates during the course of my bachelor and master’s degree in the Faculty of Engineering of the University of Porto. Their help and intellectual challenge made me grow personally to achieve what I have and to prepare me for the future.

Finally, I’d like to acknowledge the importance of my friends and family. Their support and belief in me were my biggest motivation during this process. I cannot express how lucky I am to have them in my life, and I hope this paper makes them proud. Couldn’t finish the acknowledgements without thanking my dog and cat for all the emotional support.

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List of Abbreviations

AO	**Atomic Oxygen**
ATM	**Accelerated Testing Methodology**
CFRP	**Carbon Fibre Reinforced Polymers**
CMC	**Ceramic Matrix Composite**
FRP	**Fibre Reinforced Polymers**
LCD	**Linear Cumulative Damage Law**
LEO	**Low Earth Orbit**
MMC	**Metal Matrix Composite**
PECVD	**Plasma-Enhanced Chemical Vapor Deposition**
PMC	**Polymer Matrix Composite**
RoM	**Rule of Mixture**
ROS	**Reactive Oxygen Species**
RVE	**Representative Volume Element**
TTSP	**Time-Temperature Superposition Principle**

List of Symbols

T_g	**Glass Transition Temperature**
E_{11}	**Effective Modulus in the Longitudinal Direction**
E_{11f}	**Elastic Modulus of the Fibre in the Longitudinal Direction**
E_m	**Elastic Modulus of the matrix**
V_f	**Volume Fraction of the Fibre**
V_m	**Volume Fraction of the Matrix**
E_{22}	**Effective Modulus in the Transverse Direction**
E_{22f}	**Elastic Modulus of the Fibre in the Transverse Direction**
E_2	**Effective Modulus in the Transverse Direction**
G_{12}	**Effective Shear Modulus**
G_m	**Shear Modulus of the Matrix**
T	**Temperature**
σ_i	**Stress in the *i* Direction**
ε	**Strain**
C_m	**Stiffness Tensor of the Matrix**
S_f	**Compliance Tensor of the Fibre**
F_i	**Allowable Strength in the *i* Direction**
D_c	**Creep Compliance**
η	**Viscosity**
E	**Youngs' Elastic Modulus**
t	**Time**
$a_{T0}(T)$	**Time-Temperature Shift Factor**
T_0	**Reference Temperature**
T_m	**Melting Temperature**
n	**Stress Exponent**
b	**Burguers Vector**
d	**Grain Size**
p	**Stress Component**
k	**Boltzmann's Constant**
D	**Diffusion Coefficient**
A	**Dimensionless Constant**

1. Introduction

For centuries, humanity has strived to improve its quality of life. This process of modernization has profoundly affected the way people leave nowadays. The continuous search for the frantic impetus of progress has consequently pushed science forward, and innovation in the last century has reached an all-time high. The pace of innovation has been so exciting that it is difficult to pinpoint which advances have had the biggest impact. Society around us is changing and so are its demands on existing structures.

Advanced composite materials have been in the spotlight in a lot of industries that are looking to produce high-strength and lightweight materials. The demand from end-use industries such as construction aerospace, and automotive industries is rising, along with manufacturers and suppliers making it a 72.12B $ market size back in 2020 (Globe Newswire, 2021). The race to efficiently produce high performance materials has brought expanded interest in composite materials, and it is expected that its market size will nearly double by 2028. The continuous use of composite systems especially in structural applications that require high reliability for long durations causes some concerning regarding the resistance of the material through time. Therefore, there is a critical need to study the durability of composite systems, to evaluate and forecast the long-term resilience of these materials.

To solve problems and to explore new ideas, nature has always been a major source of inspiration. Biomimetics or biomimicry is used in many sectors, such as design, material development, technology and others. Therefore, closely exploring animals and plants may be a fruitful way to find templates for new and unique technology.

The overall aim of this thesis is to investigate Fibre Reinforced Polymers (FRPs) structures for the purpose of proposing a more reliable life performance. The durability of advanced materials is a paramount characteristic, and the focus of the present work is on investigating the influence of time, temperature and other environmental conditions on the material properties. In general, the idea is to explore ageing behaviour in materials and in living organisms, and draw a parallel with both mechanisms.

Our discussion will follow three major parts. The first one is a complex and informative bibliographic review of the durability of composites and ageing mechanisms of living organisms, followed by a chapter where we summarize and explore the prior information in order to suggest a resolution to the problem. And, finally, a more direct response to the dissertation in hand with a proposal of an investigation methodology.

2. State of the Art

2.1 Composite Systems

Composite systems are materials that are very desired by their capability to exhibit high-strength and lightweight. The possibility of mixing two or more different materials together is a prolific strategy that enables the development of a product with enhanced mechanical properties. Their applicability is vast but, for now, prices for advanced materials are very high compared to their traditional counterparts due to the higher complexity and difficulty in manufacturing.

For many years composites have been all around us, in object with which we interact every day, in fact it can even be found in nature, for example wood with long cellulose fibres that hold together a substance called lignin. The first human-made composite dates back to 1500 BC, when a mixture of mud and straw was used to create strong and durable buildings for Egyptians (Ngo, 2020). Nowadays, composites have spread into our everyday lives such as products that are used in medical applications, oil and gas, sports, transportation, construction and many more. In all these applications, composites managed to bring enormous benefits, and in some cases, namely in spacecrafts it is critical for them to function properly.

The progress made in the development of these advanced materials over the years (such as reducing cycle time) has created an increase in demand. This makes nowadays Carbon Fibre Reinforced Polymers (CFRP) and other composites an emerging solution for the automotive industry, as well as retaining a crucial role in the aerospace industry.

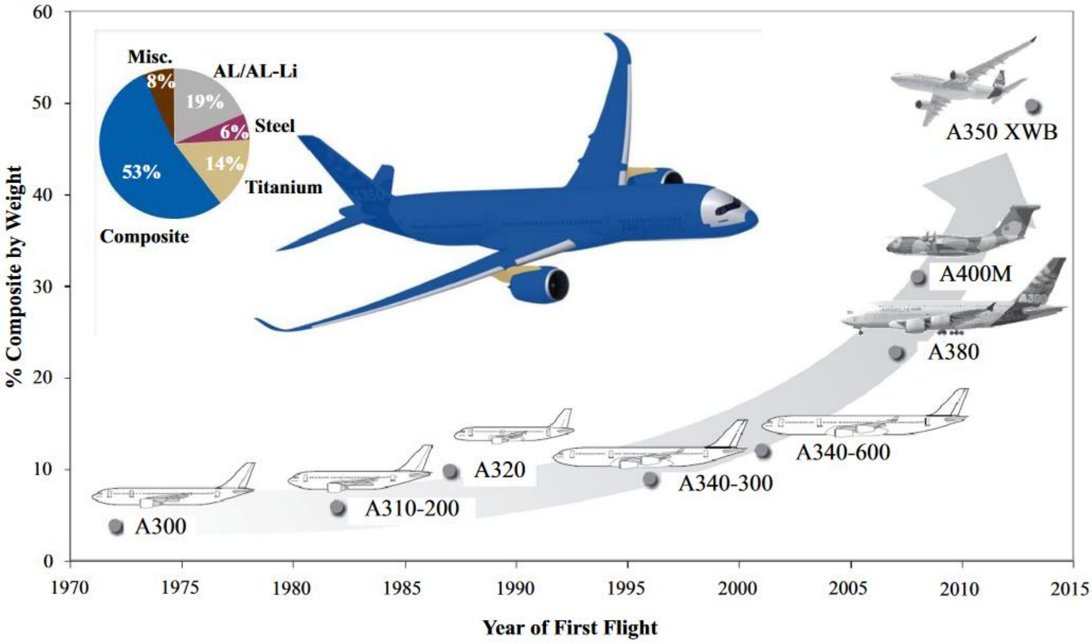

Figure 1: Evolution through time of the presence of composites by weight in commercial aircrafts (Xu, et al., 2018)

The ability to design materials with unique properties not found in existing natural materials attracts a lot of interest. One of the biggest and most important markets for these materials is the aerospace industry. In fact, composite systems are continuously establishing themselves in civilian aircrafts such as the Boeing 787 Dreamliner and Airbus A350 XWB. As seen in Figure 1, the use of composites in commercial airplanes has been evolving very rapidly, with airplanes having nowadays around 80% composites by volume and about 50% by weight (Giurgiutiu, 2019).

The demand for composites continues to increase worldwide, and it is expected to reach 144,5 billion U.S. dollars in 2028 which means more than double the demand than in 2015 (Statista, 2022). These materials have been used in endless applications because of their enhanced mechanical, physical and versatility.

2.1.1 The Role of Constituents of Composite Materials

Composites are made through a combination of two or more materials. The materials used have distinct physical and chemical properties in comparison to each other. Upon modification, composite materials gain several qualities such as mentioned before.

This optimization in mechanical properties is a product of the combination of the matrix with a reinforcement material, with intentional alignments or randomly oriented filaments. This system combines the good properties of the matrix and the fibre, as shown on Figure 2. Therefore, when designing a composite, it is important to take into consideration the different properties both the matrix and fibre have. Composites, most of the time, are anisotropic, which means that their properties change when measured in different directions.

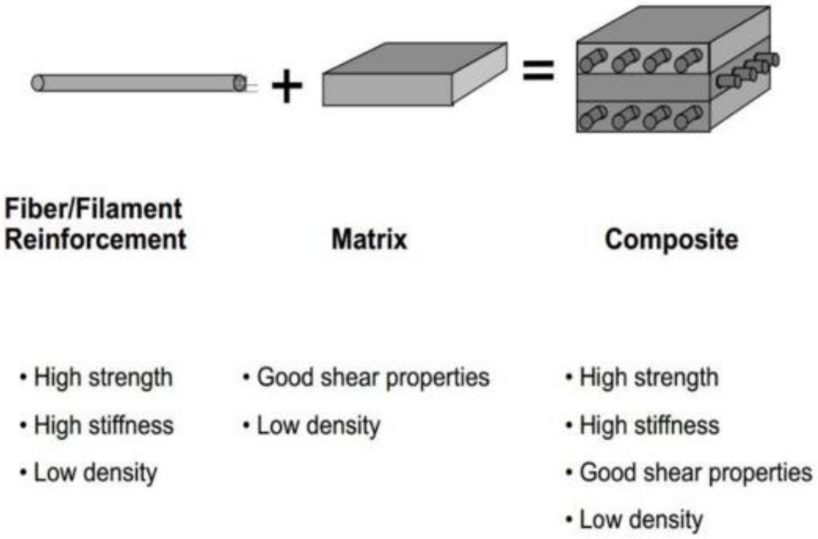

Figure 2: Schematic representation of a composite system (Kumar & Lohchab, 2016)

The reinforcement increases the mechanical properties of the composite, while the matrix ensures load transfer to the reinforcement, protects the reinforcement surface, and adds the necessary toughness to the system. In general, fibres provide more strength and stiffness to the composite system. Some examples of fibre materials are glass, carbon, aramid, boron, basalt, and natural fibres.

The matrix can be a polymer (thermoset or thermoplastic), a ceramic or a metallic material. Its function is to protect and transfer the loads between fibres, to make sure the fibres help the composite resist cracks and fractures (Sharma, Bhandari, Aherwar, & Rimašauskiene, 2020). The matrix selected to use in a composite system depends on the properties required by the application of that same system. Examples of polymeric matrices are the thermosets: unsaturated polyester, epoxy, vinyl ester, etc; and thermoplastics: polypropylene – PP polyester, polyamide – PA, polytetrafluoroethylene – PTFE, poly ether ketone - PEEK, etc.

Composite systems present numerous advantages that makes it a very attractive material, such as design flexibility, high specific stiffness and strength, damping capacity, low thermal expansion coefficient and durability. Unfortunately, since it is a relatively recent material, there is still much to uncover, and scientist are working on trying to better their understanding of its properties (long-term behaviour, added-value of the systems, etc.). (Marques A. T., 2019)

In Figure 3, we can observe the different types of composites. They are differentiated according to the nature of the matrix and the fibres. The most commonly used composites are the polymer matrices, also known as Polymer Matrix Composites (PMCs), which normally provide a good surface finish quality to the composites and protect reinforcing fibres against chemical attacks. There are two unique characteristics of polymeric solids that distinguish them, the ambient temperature and the loading rate. They have a huge influence on the mechanical properties of polymeric solids (Mallick, 2007).

A polymeric matrix can be classified in two different types according to their polymer category: thermosetting resins and thermoplastic resins. Thermoplastic polymers are characterized by the ability to be softened and melted with the increase of temperature, and then hardened when cooled. On the other hand, thermosets polymers are irreversible, well defined and, through the process of curing, the chemical networks get bigger and stronger. The curing process can be caused by heating or using a curing agent in the process. In general, thermoset polymers are more used for structural applications, as they appear to be more resistant to solvents and corrosive environments than thermoplastics (Tanzi, Farè, & Candiani, 2019).

A thermal property that is relevant for amorphous polymeric materials and that will be mentioned in the following chapters is the glass transition temperature (T_g). This temperature range defines the transition from rigid material to softened material. In fact, this temperature dictates the intensity of molecular mobility within the polymer, that results on the product being in the glassy state or the rubbery state (Campo, 2008). In addition, this temperature is not the same as the melting temperature, as when there is an increase in the molecular mobility the material gets soft but never reaching the melted state.

Ceramic Matrix Composites (CMCs) are a mixture of fibres (ceramic or not) and a ceramic matrix, making them very resistant to high temperatures and efficient in corrosive environments (Sharma, Bhandari, Aherwar, & Rimašauskiene, 2020). Therefore, it is common to use ceramic matrices for high-temperature applications such as pistons or rotors in gas-turbine parts. These materials were developed to overcome some problems that can be found when using ceramics, namely the intrinsic brittleness and mechanical unreliability of monolithic ceramics (Cho, Boccaccini, & Shaffer, 2009).

Finally, there are the Metal Matrix Composites (MMCs) where the use of reinforcement improves the strength and wear of the material matrix. Compared to traditional materials, MMCs can bring better strength-to-weight ratios, stiffness, and ductility (Sharma, Bhandari, Aherwar, & Rimašauskiene, 2020). Despite being found in aircraft components and space systems their applicability is still very limited by manufacturing costs. The most common matrix metals are Aluminium, Copper, Iron, Magnesium, Nickel and Titanium (Sharma, Bhandari, Aherwar, & Rimašauskiene, 2020).

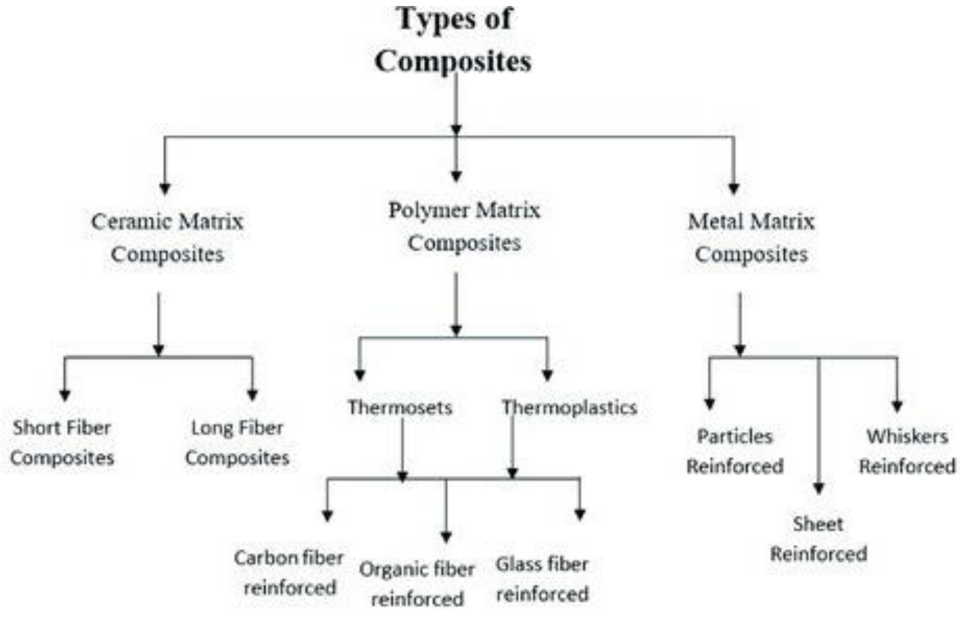

Figure 3: Types of composites (Singh, Kumar, & Chohan, 2020)

On the other hand, one can classify the composite system with respect to the fibres that act as reinforcement. In fact, the role of the reinforcement is fundamentally to ameliorate the mechanical properties of the neat resin system. Depending on the fibre being used, the end composite system is going to have a different mechanical behaviour due to the variety of properties each type of fibre has.

There exist different types of reinforcements but the most important one are fibres. In theory, the strength of a fibre is defined by how strong the atomic bonds that hold the fibre material are; therefore, strong chemical bonds are the best suited (Jr. & Rethwisch, 2018). For this reason, the structures of the strongest composite fibres are composed of elements with strong covalent bonds, such as Boron, Carbon and Silicon.

In general, fibres are classified in two major categories: Organic fibres and Inorganic fibres. And the most common fibre reinforcements incorporated in polymer matrices are Glass, Carbon, and Aramids. The focus will be primarily towards fibres used in PMCs since it is the most common combination, and the information is more expansive.

Glass Fibre-Reinforced Polymer Composites are produced in large quantities, mostly because they are cheaper than carbon reinforcements. Glass is used as reinforcement for several reasons: it is easily drawn into high-strength fibres from the molten state, more economically accessible, relatively strong and when combined with a polymer matrix it produces a composite having a very high specific strength, and chemical inertness that allows the composite system to be used in a variety of corrosive environments (Jr. & Rethwisch, 2018). Nevertheless, this group of material presents some limitations, especially regarding stiffness and service temperature.

In advanced polymer composites, the most commonly used reinforcement is Carbon, as it is a high-performance fibre material. When compared to other fibre materials, carbon fibres have the highest specific modulus and specific strength of all; they also retain high tensile modulus and high strength at elevated temperatures. Besides that, Carbon exhibits a variety of physical and mechanical traits allowing composite systems to have specific engineered properties, and CFRPs now have manufacturing processes that are relatively inexpensive and cost effective. Despite the only concern with the material is its high temperature oxidation that may cause a problem to the composite system for now (Jr. & Rethwisch, 2018).

		Tensile Strength GPa	**Specific Strength GPa**	**Modulus of Elasticity GPa**	**Specific Modulus GPa**
Whiskers	Graphite	20	9.1	700	318
	Silicon nitride	5-7	1.56-2.2	350-380	109-118
	Aluminium oxide	10-20	2.5-5.0	700-1500	175-375
	Silicon Carbide	20	6.25	480	150
Fibres	Aluminium oxide	1.38	0.35	379	96
	Aramid	3.6-4.1	2.5-2.85	131	91
	Carbon	1.5-4.8	0.7-2.7	228-724	106-407
	E-glass	3.45	1.34	72.5	28.1
	Boron	3.6	1.4	400	156
	Silicon carbide	3.9	1.3	400	133
	UHMWPE (Spectra 900™)	2.6	2.68	117	121
Metallic Wires	High-strength steel	2.39	0.3	210	26.6
	Molybdenum	2.2	0.22	324	31.8
	Tungsten	2.89	0.15	407	

Table 1: Characteristics of several reinforcement materials (Jr. & Rethwisch, 2018)

Aramid fibres are very attractive for their very good strength-to-weight ratios. They present one of the highest longitudinal tensile strengths and tensile modulus of all polymeric fibre materials, but they are relatively weak in compression (Jr. & Rethwisch, 2018). This type of fibre is also known for its toughness, impact resistance, and resistance to creep and fatigue failure. Although they are vulnerable to degradation by strong acids and bases, they are somewhat inert in other solvents and chemicals (Jr. & Rethwisch, 2018).

Even though the composition of the fibres is relevant, it is not the only factor to affect the reinforcements' performance. Length, shape, orientation, and the mechanical properties of the matrix are all important factors to determine the performance of the fibre. The orientation of the fibre, the fibre concentration, and their distribution all have a considerable impact on the strength and other properties of fibre-reinforced composites (Thomason, Vluga, Schipper, & Krikort, 1996) (Fu & Lauke, 1996). The strength of the composite is greatest along the longitudinal directional of fibre, so optimum performance is obtained if the load is applied along its direction (Jr. & Rethwisch, 2018). This also means that the slightest shift in the angle of loading may drastically reduce the strength of the composite in some specific directions.

In fact, it is possible to assume two main possibilities in respect to the orientation of the fibres: a parallel alignment of the longitudinal axis of the fibres in a single direction, and another random alignment. In general, continuous fibres are normally aligned, while discontinuous fibres may be aligned, randomly or partially oriented. As expected, when we have fibre distribution uniform the composite presents better properties. The different orientation and concentration of fibres can be seen in Figure 4. Also, the orientation is significant for the composite system, but so are other factors for example, the geometry and structure of the fibres that affect the composite properties in a more specific way, such as thermal resistance and porosity (Liu, de Araujo, & Hu, 2016).

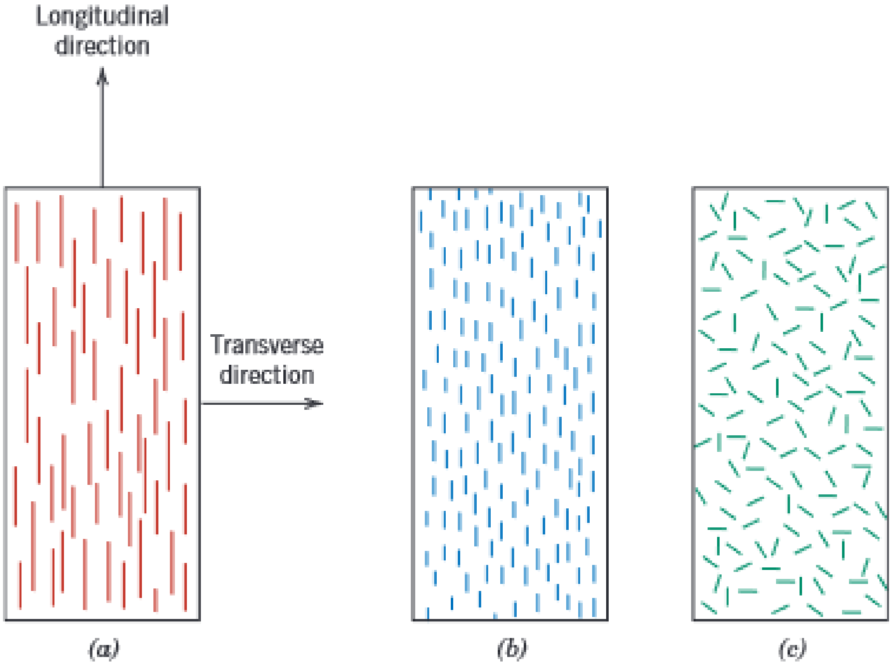

Figure 4: Schematic representation of fibres: (a) continuous and aligned, (b) discontinuous and aligned, (c) discontinuous and randomly oriented (Jr. & Rethwisch, 2018)

2.1.2 Mechanical Behaviour of PMCs

The vast field of applications of fibre-reinforced PMCs creates an important requirement in the evaluation of their mechanical properties in order to design and manufacture lightweight durable structures. There is the need to define the composite system by determining the strength and toughness of the material, by characterizing the fibres and resins, by characterizing the elastic behaviour, and others.

In this matter, there is a difference between the fibre and the matrix influence. Regarding the elastic properties that difference is evident, especially regarding the orientation of those fibres since the fibres enhance the elastic properties of the composite through their orientation.

The elastic constants of plain fibre-reinforced composites depend greatly on fibre orientation with notable anisotropic characteristics (Miranda, Soeiro, & Silva, 2022).

An evaluation of the elastic performance of a composite system must be done to better understand the behaviour of the material in question. This evaluation is done most efficiently through the modelling of composite systems at different scales. For instance, it may be done at the macro-scale, which deals with the whole structure: its shape and boundary effects when subjected to loads. Or it can focus on the laminate or ply of the composite, also known as mesoscale. However, the micro-scale presents itself as the one with the most importance (Raju, Hiremath, & Mahapatra, 2018).

In addition, the modelling scale needs to be properly defined because the in-plane dimensions of composite laminates go beyond the length scale at which failure events occur, such as delamination, fibre/matrix debonding and matrix cracking. For instance, at lower structural scales, composite system idealisations have higher resolution and higher kinematic freedom, and they are able to recreate all kinds of damage mechanisms, each of which captured with separate damage laws (Arteiro, Catalanotti, Melro, Linde, & Camanho, 2014).

The micro-scale evaluation obtained through micro-mechanical scale models (based at the constituent level) is essential to successfully design a durable structure. The process of incorporation of the elastic properties of the composite and related optimization in relation to the constituent materials and geometric parameters is crucial. In turn, the geometric parameters depend on the length scales, starting from the fibre and inclusions in the order of 10^{-6}, to the fabric and plies with thicknesses with amplitude of 10^{-3}, to the laminates having dimensions of the order of 10^{-2}, and all the way up to the composite system having dimensions of more than a few meters. This scale difference is observable in Figure 5.

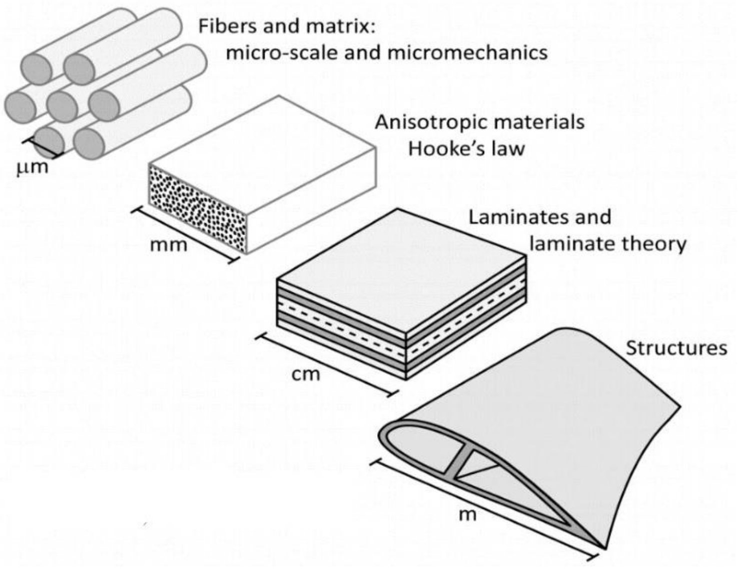

Figure 5: Multiscale methods (Vedvik, 2017)

In addition, it is also important to note that a suitable micro-mechanical homogenisation technique provides the effective properties of the composite system based on the properties of the constituents (Raju, Hiremath, & Mahapatra, 2018).

Models based on a self-consistent scheme are the more well-established micromechanics-based models. There are many models capable of analysing and predicting the mechanical behaviour of PMCs. Different models incorporate different information in order to predict effective mechanical properties. However, only some of the models are going to be reviewed here.

- Rule of Mixtures (RoM)

The Rule of Mixtures (RoM) is ubiquitous in the calculation of the elastic properties of composite materials, and it is characterized by its simpleness. In general, it incorporates two models, the Voigt model for longitudinal loading and the Reuss model for transverse loading (Raju, Hiremath, & Mahapatra, 2018).

- **Voigt model**

This model assumes that, in longitudinal loading conditions, the strain in the fibre and the matrix are assumed to be the same as the strain in the effective medium, as seen on Figure 6 (a). The effective elastic modulus based on the RoM in the case of longitudinal loading is calculated using the following equation (Raju, Hiremath, & Mahapatra, 2018):

$$\boldsymbol{E_{11} = E_{11f}V_f + E_mV_m = E_{11f}V_f + E_m(1 - V_f)} \qquad (1)$$

With:

E_{11} - effective modulus in the longitudinal;
E_{11f}- elastic modulus of the fibre (assumed transversely isotropic);
E_m - elastic modulus of the matrix (assumed isotropic);
V_f - volume fraction of the fibre;
V_m –volume fraction of the matrix.

- **Reuss model**

In this model the iso-stress condition is assumed for the transverse loading, as seen on Figure 6 (b), that means that the stress in the fibre is transmitted to the matrix and is equivalent to that of the stress in the effective representative volume element (RVE). For transverse loading condition the following equation is used (Raju, Hiremath, & Mahapatra, 2018):

$$\frac{1}{E_{22}} = \frac{V_f}{E_{22f}} + \frac{1 - V_f}{E_m} \qquad (2)$$

With:

E_{22} - effective modulus in the transverse direction;

E_{22f} - transverse elastic modulus of the fibre (assumed transversely isotropic);

The RoM model is also capable of predicting the effective Poisson’s ratio and the effective shear modulus for iso-shear stress and strain conditions.

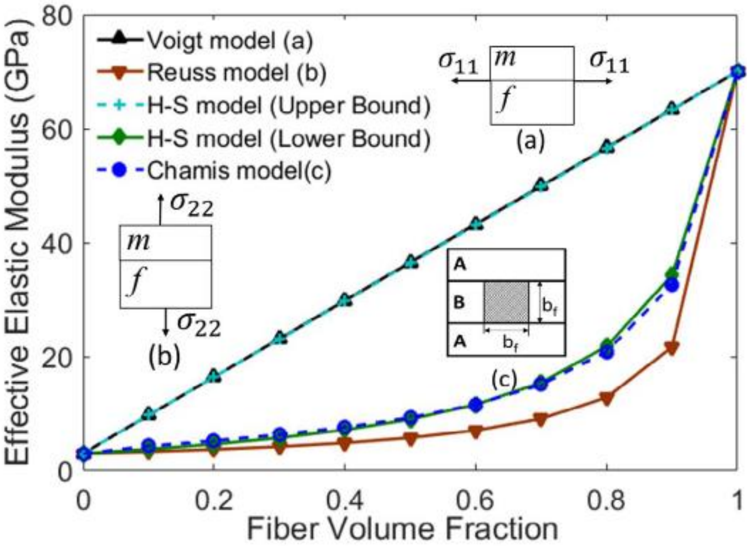

Figure 6: Effective elastic modulus in function of fibre volume fraction calculated from different models. Inset (a) shows Iso-strain condition for longitudinal loading, inset (b) shows Iso-stress condition for transverse loading, and inset (c) shows Chamis model with square packing, where m represents the matrix and f the fibre (Raju, Hiremath, & Mahapatra, 2018)

It is important to refer that the modulus projected according to the Voigt model gives the upper bound of the effective elastic modulus and provides reasonable predictions for the effective longitudinal stiffness of a unidirectional lamina. Nevertheless, the RoM is incapable of predicting values for the effective transverse and shear elastic properties.

- Halpin-Tsai model

Based on the geometry and orientation of the fibres and the elastic properties of both the fibres and the matrix, this model relies on the self-consistent field method even if often considered to be empirical. Halpin and Tsai (and Kardos) established generalized equations with empirical terms that provide a much-improved estimate of the effective properties. The Halpin-Tsai model is very precise when obtaining results for the transverse Young’s modulus and the shear modulus through curve fitting of the outcomes obtained from elasticity models, and presents a better estimative of the effective properties compared to RoM. These properties may be determined through the following equations (Raju, Hiremath, & Mahapatra, 2018):

$$E_2 = \frac{1+\xi_2\eta_2V_f}{1-\eta_2V_f}E_m, \text{ where } \eta_2 = \frac{\gamma_2-1}{\gamma_2+\xi_2} \text{ and } \gamma_2 = \frac{E_{2f}}{E_m} \tag{3}$$

$$G_{12} = \frac{1+\xi_{12}\eta_{12}V_f}{1-\eta_{12}V_f}G_m, \text{ where } \eta_{12} = \frac{\gamma_{12}-1}{\gamma_{12}+\xi_{12}} \text{ and } \gamma_{12} = \frac{G_{2f}}{G_m} \tag{4}$$

With:

E_2 - effective modulus in the transverse direction;
G_{12} – effective shear modulus;
G_m - shear modulus of the matrix;

It is important to note that the factor ξ is an empirical parameter that measures the geometry of the inclusion. It is determined by curve fitting of experimental data for a range of geometric parameters of interest. It is assumed that ξ_2= 1.52 and ξ_{12}= $1+40V_f^{10}$, results that are valid for at least $0{,}20 \leq V_f \leq 0{,}55$ and $V_f \leq 0{,}70$ respectively. Also, the function η may be interpreted as a ratio between the fibre phase property and the matrix property (Raju, Hiremath, & Mahapatra, 2018).

The main restriction of this model is the estimation of factor ξ that is acquired from curve fitting, which requires experimental and finite element simulation data.

- Mori-Tanaka model

This model allows to obtain elastic properties of the composite systems while taking into consideration that the composite is transversely isotropic. Mori and Tanaka's work was important to determine a method to calculate the homogenized internal stress on a composite material, based on Eigen-strain theory. According to this theory the concentration tensor relates (T) the strains (ε) in the fibre with the ones in the matrix, and the stresses (σ) are related by the Mori-Tanaka tensor that is defined as below (Raju, Hiremath, & Mahapatra, 2018):

$$A = C^mT^{-1}S^f \tag{5}$$

where C_m is the stiffness tensor of the matrix, and S_f is the compliance tensor of the fibre. A full review on Mori-Tanaka scheme is properly elaborated in Raju, Hiremath and Mahapatra's review on micromechanics models for PMCs (Raju, Hiremath, & Mahapatra, 2018).

In that same paper, a comparison between the previous presented models is made and some considerable differences were noted. The RoM models are good to predict the effective moduli values in the respective direction, transversely (Reuss) and longitudinal (Voigt). These models are very basic and simple, they take some considerations that minimizes some important effects, such as the fibre packing geometry.

On the other hand, Mori-Tanaka's model is very accurate for lower fibre volume fractions, because it assumes only one inclusion in an infinite medium. Although this could be avoided by assuming that the matrix has the properties of the desired composite system and then placing a single inclusion into this matrix. This way, the impact of several inclusions is modelled, making the Mori-Tanaka's model more refine. (Raju, Hiremath, & Mahapatra, 2018)

These theoretical methods very often serve as a basis to study the mechanical behaviour of composite materials using robust finite element simulation models which are very precise and reliable in predicting mechanical performances.

2.1.3 Failure in Composite Systems

Composite materials are used for many applications and in most of them the conditions are severe, either by the extreme loads applied in the structure or by the environmental surroundings that take the material to the extreme. Failure criteria defines the limit in terms of values in stress or strain that divide "failed" states from "non-failed" states. Its definition is very important to be use in the analysis, calculation of safety factors and design of composite systems (Camanho, Arteiro, Catalanotti, Melro, & Vogler, 2015).

Since the early 2000's, a big number of different failure criteria were developed and employed to solve practical problems, and through the years the accuracy of each theory has been tested out and compared to others to determine those that approximate the better to reality (Hinton, Kaddour, & Soden, 2004). In the international activity, known as World-Wide Failure Exercise, that was organized to define which failure criteria theories are better suited for composites. The theoretical predictions that exhibit the best performance were Puck, Cuntze, Tsai, Zinoviev, and Bogetti (Hinton, Kaddour, & Soden, 2004).

Even though those were the most accurate theories, until 1996 the most used theories were as shown in Figure 7. With several theories to predict failure in composite materials there is still the need to separate them into two different groups. The non-phenomenological failure criteria are the simplest group, were operating it is easy and instantly susceptible to computational procedures but does not identify failure modes. In contrast, there is phenomenological failure criteria group that provides information on the damage of the composites and the regarding type of failure (Camanho, Arteiro, Catalanotti, Melro, & Vogler, 2015).

Failure modes in composites represent the numerous ways that the material might fail. These damage mechanisms usually take place at the microscopic level and are the consequence of damage accumulation, which then evolves until reaching macroscopic level (McCarthy & Vaughan, 2015). Some examples of failure mechanisms of composite systems are micro-cracks in matrix, fibre fractures, debonding between fibres and matrix, delamination (for laminate composites), etc. Depending on the loading conditions some failure modes will occur easily than others, for example for longitudinal tension it is typically the fibre that will fail first just like in Figure 8 because it has the lowest ultimate strain (Daniel & Ishai, 1994).

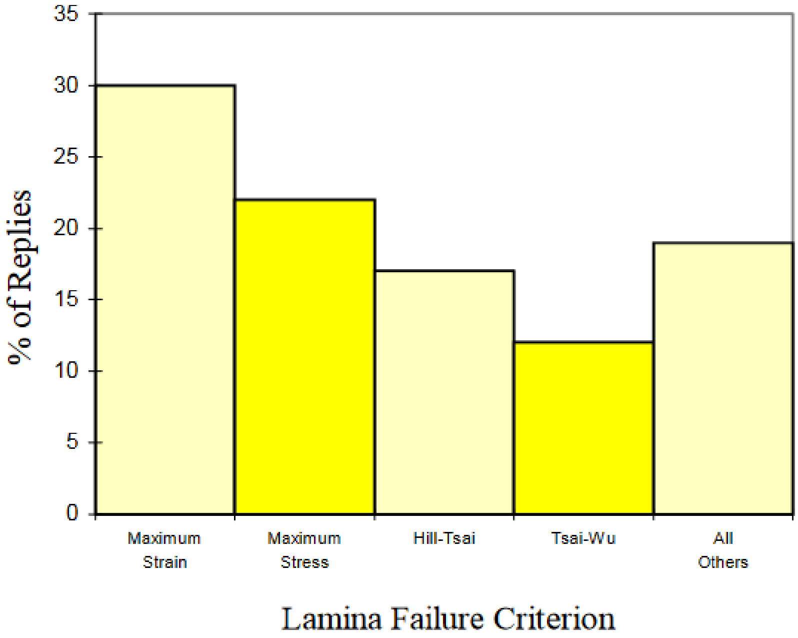

Figure 7: Failure criteria distribution after survey (Sun, Quinn, Tao, & Oplinger, 1996)

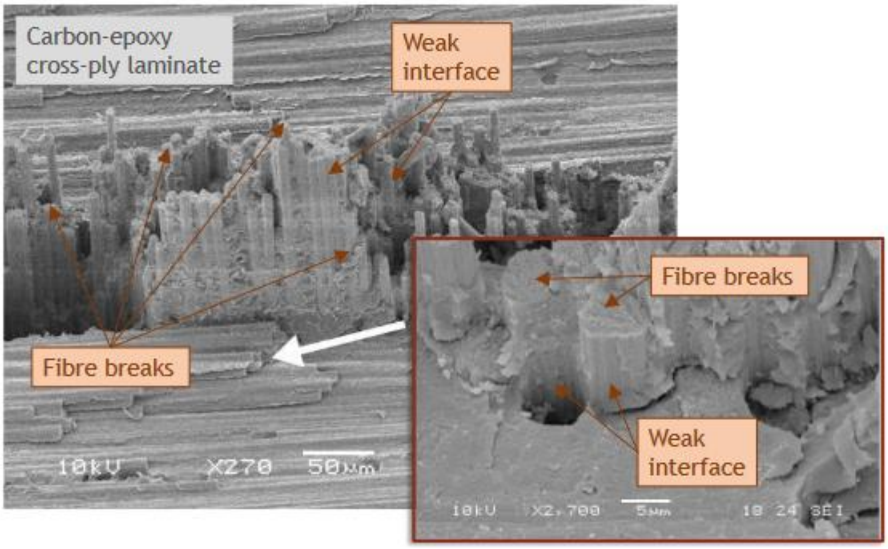

Figure 8: Microscopic view of laminate surface with broken fibres (Laffan, Pinho, Robinson, & Iannucci, 2010)

Looking further to the failure criteria the developed theories are the product of an extension and adaption of isotropic failure criteria for anisotropy in composites (Camanho, Arteiro, Catalanotti, Melro, & Vogler, 2015). Micro-mechanical damage mechanisms are the consequence of failure in composites, this is why there is an increased need to develop accurate and physically based failure theories.

The most recognised failure criteria for composites laminates are the maximum stress criterion and the maximum strain criterion, both being based on defining limits that cannot be exceed otherwise failure will occur. Their popularity in engineering applications today is desirable for their simplicity and the identification of failure modes, although there is a lot of controversy towards the accuracy of them (Li, 2020). The difference between them lays on the strength components that are being analysed, being that for the maximum stress criterion the limits are characterized in terms of stress components, while for the other one in terms of strain components (Aboudi, Arnold, & Bednarcyk, 2021).

Another phenomenological failure criteria that is also very common is Tsai-Hill failure criterion that is a modification of von Mises criterion use in ductile metals with anisotropy and adapted to unidirectional composite laminates (Daniel & Ishai, 1994). The equation that defines this criterion allows for considerable interaction among stress components even though tensile and compressive strengths must be specified according to the signs of the normal stresses, as it is shown in the following equation (Daniel & Ishai, 1994):

$$\boldsymbol{\frac{{\sigma_1}^2}{{F_1}^2}+\frac{{\sigma_2}^2}{{F_2}^2}+\frac{{\tau_6}^2}{{F_6}^2}-\frac{\sigma_1\sigma_2}{{F_1}^2}\geq 1} \tag{6}$$

An anisotropic composite material according to the Tsai-Hill failure criterion exhibits failure if the equation equals or surpasses 1. The directions of the respective strengths analysed are defined by the numbers in index, where 1 and 2 represent the longitudinal and transversal direction, respectively. While the index 6 represents the ply between the transversal and longitudinal directions.

In contrast, the Tsai-Wu failure criterion makes the distinction between tensile and compressive strengths, and it considers the normal stresses interaction. However, with this failure criteria it is impossible to detect failure modes. Unlike the other criteria, the Tsai-Wu criterion is defined by a polynomial function, as following (Aboudi, Arnold, & Bednarcyk, 2021):

$$\boldsymbol{F_i\sigma_i+F_{ij}\sigma_i\sigma_j=1} \tag{7}$$

Just like the Tsai-Hill equation, failure occurs when the equation equals 1. One advantage of this failure criteria is that it can be applied to a 3D state of stress, although for plane stress application the equation gets already complex.

In Figure 9, there is an example of the failure envelope of each of the failure criteria that were just now reviewed. The values inside the failure envelope are the ones considered to be safe, while outside stress values would go above the given failure criterion (Aboudi, Arnold, & Bednarcyk, 2021). This schematic comparison for $\sigma_1 - \sigma_2$ stress space shows the significant difference between failure theories, and even from interactive and non-interactive criteria, being that interactive failure criteria are the Tsai-Hill and Tsai-Wu. Another interesting aspect regarding this representation, is that the four different failure envelopes cross themselves in the same point located in the stress axes, which corresponds to the uniaxial failure values that are given for the stress-based criteria (Aboudi, Arnold, & Bednarcyk, 2021).

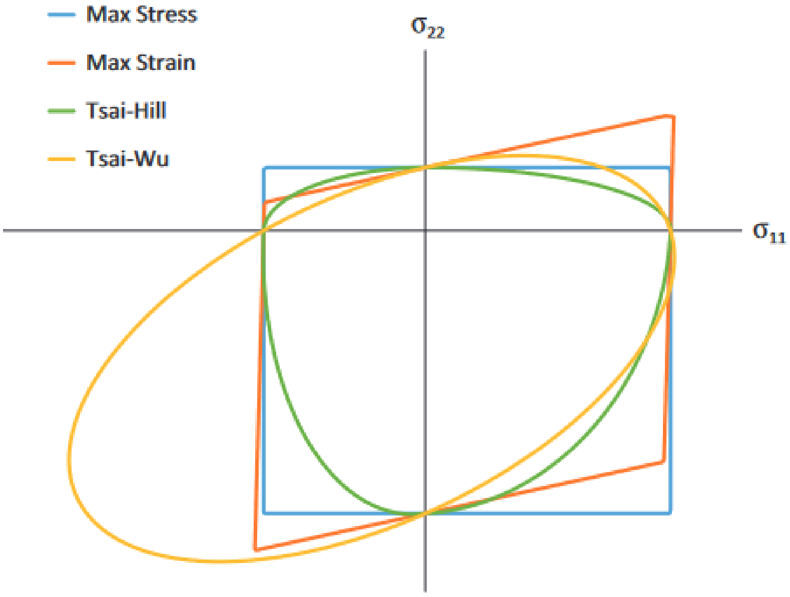

Figure 9: Failure envelopes of the four failure criteria presented (Aboudi, Arnold, & Bednarcyk, 2021)

2.2 Composites: Durability and Degradation

In composites, there are two ways for the advanced material to lose properties over time, either by ageing or by environmental degradation. Both are particularly important when studying the utility of a composite system in any given application, although the focus of this thesis is towards enhancing the durability damaged by ageing. Nevertheless, a brief introduction in degradation of composites may give a distinct perspective to the problem.

Contrary to ageing, degradation has been studied for years and the information provided is clearer and more thorough. Composites are widely used in many different applications and are submitted to distinct environments, which brings susceptibility to specific types of degradation. High and low temperatures, high humidity, space environments, are just some of the examples of conditions that could cause substantial degradation (Jacob, 2002). In fact, these environmental conditions directly affect bonding between the fibre and the matrix, causing a loss in mechanical properties (Drzal, 1985).

In composites, there are two distinct terms for “boundary”, the first one being “interface”, a two-dimensional zone, that represents the adhesion between two layers of different chemistry and/or microstructure (Drzal, 1985). In addition, another term named “interphase” that describes a three-dimensional zone that represents the interface and the occurring chemical interactions, being the volume of material affected by the interaction at the interface (Jesson & Watts, 2012). The existing interface/interphase between fibre and matrix has great importance for the behaviour and performance of a composite system (Guigon & Klinklin, 1994). For example, with high humidity the presence of moisture in the interface can affect and/or modify the interfacial adhesion causing a change in the mechanical performance of the composite.

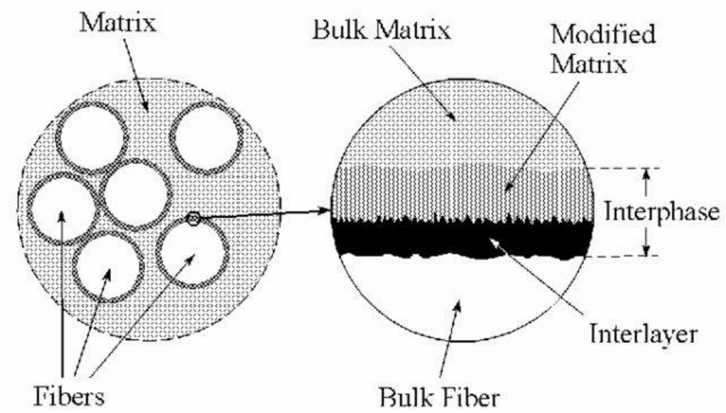

Figure 10: A schematic illustration of a composite interphase (Cech, Babik, Knob, & Palesh, 2012)

The fibre/matrix interface is a crucial aspect for composites, and it is one of those characterizing the durability of the advanced material. The mechanical performance of the composite can be translated on how good the load transmits from the matrix to the fibres; therefore, the response of fibre/matrix interface within the composite plays an important role (Sethi, 2014).

When focusing on ageing the issues differ, the interface/interphase relationship with performance may lose some relevance and the focus turns towards viscoelasticity. Ageing is a phenomenon that has a progressive and slow evolution, which affects mostly the structure and morphology of the material (Bathias, 2005). Consequently, causing a slow and irreversible evolution in its mechanical properties in service.

In reinforced polymers, the matrix is the most affected by time and temperature, which affects mechanical performance, this phenomenon is called viscoelastic behaviour (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018). Viscoelastic phenomena occurs when there is a mechanical change, that is, mechanical properties of the material suffer alterations with time or by rising temperatures. These changes happen due to the combination of viscous and elastic behaviour of the material when under stress.

Due to this time-dependent deformation a phenomenon of particular interest can be associated, creep. When referring to the relationship between viscoelastic behaviour with polymers, a slight differentiation needs to be considered, as the type of polymer and temperature play an important role, because this behaviour is mostly associated to thermoplastic polymers at temperatures above T_g (Vernon, 1992). So, overall, a polymeric resin that presents viscoelastic behaviour has the elastic capacity to recover after being deformed due to loadings applied, but, on the other hand, exhibits a viscous phenomenon called creep (Gargallo & Radic, 2009). Creep refers to non-reversible deformations under the effect of continuous mechanical stresses, which means that deformation increases with time when a material is loaded at constant stress (Altenbach & Eisenträger, 2020).

Although physical and chemical ageing also have a big influence in FRP's durability, they can be prevented by pre-ageing treatments and by stabilization of molecular structures respectively (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018). On the other hand, fibres are very stable chemically and physically, and so, they do not exhibit any type of degradation. Therefore, the viscoelasticity of the matrix is the main characteristic to affect the durability of the composite.

Even though, in general, fibres do not present an important role in the composite system viscoelastic behaviour, there are some exceptions. One example is that of aramid fibres, that show low creep and small viscoelastic behaviour, but it can be significant when making long-term predictions for structural applications (Giannopoulos & Burgoyne, 2012). Because organic fibres present viscoelastic effects, through the following text it will be considered that the composite reinforcement is glass or carbon fibres, because they do not present any viscoelastic behaviour.

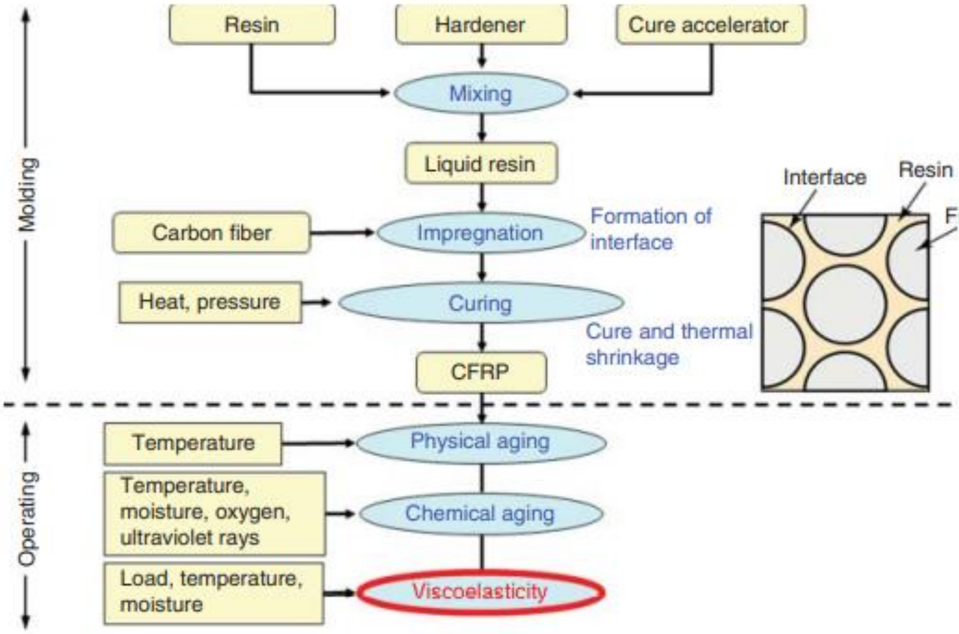

Figure 11: Role of matrix resin on CFRP (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018)

Physical and chemical ageing are mostly affected by moisture and temperature. Some interactions between the materials and the environment may induce irreversible effects, such as water where the hygrothermal ageing causes permanent damage (hydrolysis, oxidation, or leaching) (Krauklis, Akulichev, Gagani, & Echtermeyer, 2019). This damage is considered chemical and the effects although irreversible have not significant damage. This can be avoided or reduced the effect by stabilizing the molecular structures (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018).

Moreover, thermal exposure for long times can lead composites to chain fragmentation and volatilization of the resulting small molecules causing a weight loss in the resin (Hinkey & Connell, 2012). The presence of oxygen can as well cause weight loss in the matrix, consequently causing some damage such as embrittlement, shrinkage, and cracking. A proposed solution for damage tolerant composites has been a “self-healing” matrix, where physical damage such as cracks can be reverse by healing them (O'Brien, 2009). The self-repair function releases an encapsulated adhesive (healing agent) into small cracks to seal them or slow their growth, consequently restoring mechanical properties (Pang & Bond, 2005).

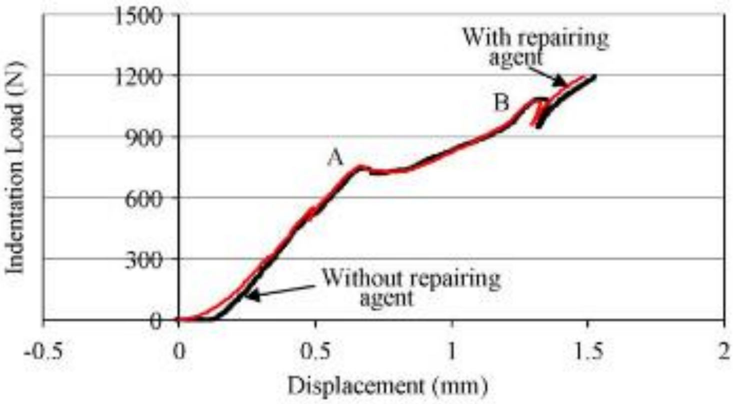

Figure 12: Load-displacement curves for indentation on infiltrated and uninfiltrated specimens (Pang & Bond, 2005)

Viscoelasticity is a property present in materials that exhibit both viscous and elastic behaviours. Viscous materials are characterized by heat energy formation caused by the manipulation of the material, while elastic materials are able to store internally the energy and then release it with minimal loss (Dunn, 2019). This implies that the laws of either solids or fluids cannot treat the matrix properties.

The viscoelastic response of the polymer-based composite is more pronounced during creep, relaxation, and dynamic mechanical loading, where the two first situation refer primarily to time dependent characteristics (Papanicolaou & Zaoutsos, 2011). Nevertheless, temperature is also very relevant in polymers' performance, which affects the viscoelastic behaviour of the composite as shown in Figure 13. In fact, temperature has a greater influence on creep behaviour for FRPs then the use of different materials, which results from generated molecular transitions caused by thermal conditions, just as glassy and rubbery phases (Monticeli, et al., 2020).

In that same chart, the viscoelasticity of the material is visible where a linear elastic behaviour is firstly identified for small deformations, and then a nonlinear behaviour is prominent for larger distortions (Papanicolaou & Zaoutsos, 2011). However, for polymer matrix composites the behaviour generally is nonlinear and rate-dependent (Kontou, 2011).

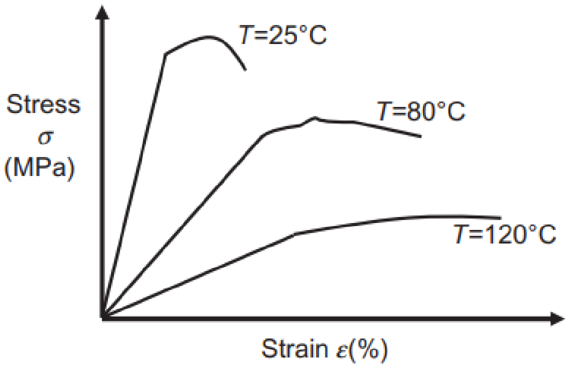

Figure 13: Stress-strain curve of polymers at different temperatures (Papanicolaou & Zaoutsos, 2011)

Thus, there are two extreme types of behaviour of the materials that need to be considered. The perfectly elastic or Hookean material, in which the stress is directly proportional to the deformation with the modulus of elasticity of the material having the proportionality constant. Moreover, the perfect or Newtonian fluid, where the shear stress is directly proportional to the rate of change of shear deformation, although in this relation the viscosity of the fluid is the proportionality constant. Both types of behaviour can be observed simultaneously at each point in time of the material (Papanicolaou & Zaoutsos, 2011).

In general, polymers have the tendency to behave more like an elastic material or like a viscous fluid according to the characteristics of usage. For example, at elevated temperatures a polymeric material performs more as a viscous fluid. The same happens to medium and large loads applied in the materials, which means that in these situations the material exhibits a nonlinear behaviour (Betten, 2005). In fact, it is known that mechanical loading, deformation, applied strain/stress rate, temperature, humidity, and time have a strong influence in the behaviour of polymers and polymer-based composites (Papanicolaou & Zaoutsos, 2011). This particularly response characteristic is the so-called viscoelastic behaviour.

To better understand the durability of composite systems and its evolution with time, there is the need to do some testing regarding the creep, fatigue, and stress relaxation of FRPs. These are all time-temperature dependent behaviours and help describe the viscoelastic behaviour of polymers. Looking at what is being explored in this thesis, of all three time-dependent behaviours creep is going to be the one under the spotlight, as it is the type of behaviour in materials that better compares to ageing in animals and plants.

Fatigue is a phenomenon that occurs with time, but with cyclical loads in contrast to creep. It is actually a very important process, as it is one of the most important events that causes cracking or failure in materials. The viscoelasticity characteristic present in the matrix of the composite system has an influence on fatigue behaviour. This means that with cyclical loadings in composite systems it would behave differently than in other materials because of the viscoelasticity of the matrix. Miyano and Nakada back in 2018 proved that in their book, where they defined some performances for cyclical loadings considering the nature of the resin. For example, in that study, the tensile strength of the composite system for the transversal direction increases with loading cycles (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018).

On the other hand, stress relaxation is the phenomenon of relaxation, that is observable by applying a continuous deformation at a steady temperature and humidity consequently causing the need for less stress to be applied to maintain constant deformation with time (Papanicolaou & Zaoutsos, 2011).

Coming back to focus on creep, there are numerous ways to improve the creep resistance and consequently increase the materials life. In thermoset polymers, where a cross-linking reaction occurs to form new chemical bonds between macromolecular chains, the creep resistance is increased by intensifying the number of cross-linking between the chains. Therefore, the glass transition temperature is increased, which then raises the creep resistance. So, a fully cured thermoset polymer is very desired (Mouritz, 2012). This method of increasing the melting temperatures is very common not only in polymers, but in all sorts of materials.

However, thermoplastics are commonly less resistant to creep compared to thermoset polymers since their creep behaviour is determined by the arrangement of the network structure of chains (Mouritz, 2012). For that reason, amorphous polymers exhibit less resistance to creep compared to other thermoplastic polymers.

So, generally thermoset exhibit better creep behaviour than thermoplastics, although that is not always the case. For instance, there exists nowadays some thermoplastic polymers with enhance temperature resistance making them more resistant to creep.

On the other hand, there is also the need to consider mechanical stresses applied in the material, as thermoset plastics are more fragile and under higher magnitude when compared to thermoplastics. This is an important feature to take into consideration when selecting the material, since the application it has will play an crucial role.

Another method to improve the effectiveness of creep resistance of composite materials is to focus on fibres. Here, the fibre alignment and length/weight can play an important role. In a study conducted by Chevali and Janowski a strong reliance of fibre alignment on creep resistance was observed in radiographs, and the positive impact that long-fibre reinforcements can have on the resin to reduce creep compliance (Chevali & Janowski, 2010). As expected, the fibre alignment is more efficient when it is aligned with the load direction.

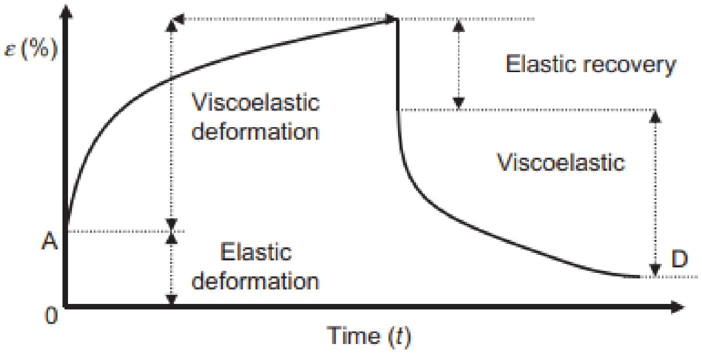

Figure 14: Strain vs time curve showing during creep phenomenon and after (Papanicolaou & Zaoutsos, 2011)

In Figure 14, it is shown the viscoelastic behaviour of a polymeric material while submitted to deformation, as there is only a small elastic deformation causing the deformation to be non-linear. The same happens when talking about the material recovery, where a time-dependent reduction in deformation characterizes the sudden removal of the constant loads (Papanicolaou & Zaoutsos, 2011). It is also visible in the figure that when a polymer or PMC is under load the immediate response is elastic, caused by the stretching of bonds in the polymer chains, and only after a point of time it starts to suffer viscous deformation caused by creep. (Mouritz, 2012)

The rate of deformation and of loading in polymers has a serious impact on some mechanical properties, such as stiffness, strength, and ductility. Thus, to predict the long-term behaviour of composite materials, creep tests are carried out, which implies that a non-linear viscoelastic analysis must be done to predict the long-term creep behaviour (Koeneman & Kicher, 1971). Although, it is important to emphasize that environmental conditions and different loads applied have an impact on how intense the creep is (Papanicolaou & Zaoutsos, 2011).

To understand creep behaviour let's take a glance at a typical curve until rupture, as shown in Figure 15. This curve can be applied for polymers, ceramics, and metals, although for these last two their melting point defines the beginning of their creep (Betten, 2005). The curve is divided into three stages of progress, and the beginning is characterized by ε_0, that represents the end of the initial elastic response of the material. The first creep stage, also known as primary creep, is characterized by a very rapid initial rate of deformation until it the rate decreases as the materials starts to create more resistance by strain hardening (Mouritz, 2012).

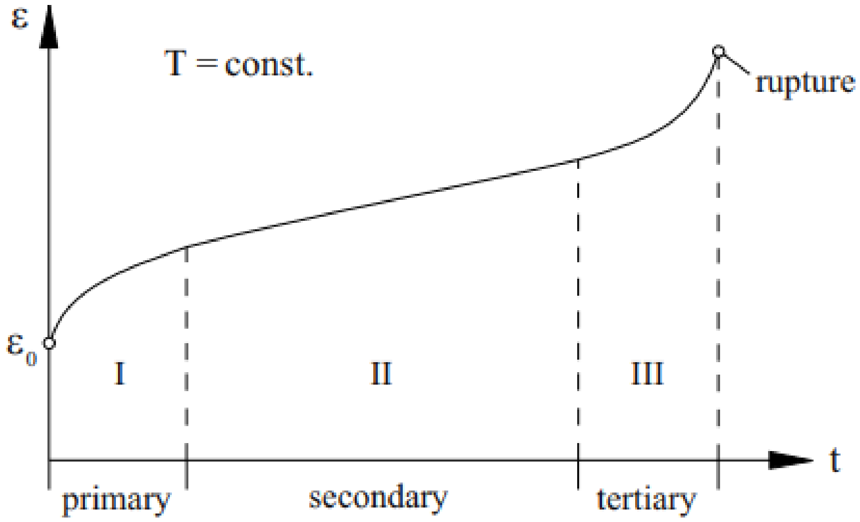

Figure 15: Creep curve (Betten, 2005)

Furthermore, with the stabilisation of the creep rate the secondary stage begins with the so-called period named steady-rate creep. The minimum creep rate is extended for as long as strain hardening keeps holding back plastic deformation, making this the longest creep phase in the materials lifetime (Mouritz, 2012).

Finally, when creep damage really starts represents the beginning of the tertiary creep phase, and it goes until final rupture of the material. Once again, the creep rate increases as the resistance to plastic deformation is not sufficient and the material starts giving up by developing internal voids and forming microscopic cracks (Mouritz, 2012).

While in mechanical characterization of metals and other materials it is typical to do a simple tensile experiment, when talking about polymers that is not the case. In fact, it is only used as a quality control element rather than an absolute data. So, creep control is used because it is a more accurate method that focus on studying the viscoelastic behaviour of materials (Papanicolaou & Zaoutsos, 2011). A deformation-time (ε- t) diagram is collected after measuring the deformation of the material at a constant load and temperature. According to the correspondent creep diagram, a polymer can be categorized as having a linear viscoelastic behaviour or a nonlinear viscoelastic behaviour. The linearity description of the viscoelastic material is relevant to define the equations that characterized the material.

In Figure 16, there is a set of creep curves ε- t with each one corresponding to a different fixed value of applied stress. From this set of curves, another set of diagrams were attained that are useful for taking some conclusions regarding the viscoelastic behaviour of the material. As a matter of fact, the isochronous curve is better compared to a simple tensile experiment because it is obtained through the isometric curve that takes into account the creep phenomenon (Papanicolaou & Zaoutsos, 2011). If a straight line is drawn in the isochronous curve, it means that the material is a linear viscoelastic material (Marques A. T., 1981). However, a material’s behaviour can shift from linear to nonlinear when the applied creep stress exceeds the linear-nonlinear viscoelastic threshold.

Therefore, the next step consists of treating the data and obtaining a master curve that predicts the viscoelastic material behaviour. There exist mainly two problems with testing viscoelastic materials. The first one, is the specific characteristics that these types of material have, because they exhibit strong time and temperature dependencies, which complicates the conversion of any method that predicts the behaviour for metals to perform on composite materials (Miyano, Tsai, Christensen, & Kuraishi, 2001). On the other hand, it is convenient that the material's behaviour is predicted for the long-term using short-term tests to establish the lifetime of materials.

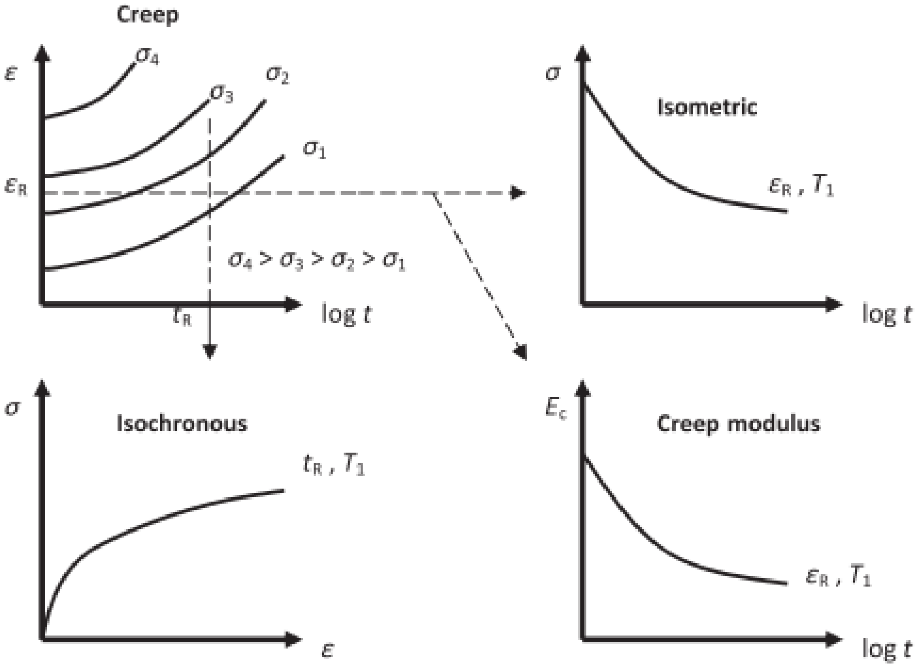

Figure 16: Three different curves obtained through the creep curves in first diagram (Papanicolaou & Zaoutsos, 2011)

For the last 50 or so years, numerous lifetime models have been proposed for viscoelastic materials, including empirical models, statistical models, and theoretical models (Guedes, 2006). There have been established numerous accelerated testing methods (ATMs) that are chosen according to the mechanical properties studied and the application of the material. Even though, there are some recent studies suggesting that ATM for some specific conditions are not reliable previsions for the long-term behaviour of polymers (Frigione & Rodríguez-Prieto, 2021).

Miyano et al. created a process where reduced variables are used, the time-temperature superposition principle (TTSP). This method dictates that the viscoelastic response at higher temperatures is the same as the response at the low temperature for a longer time. The TTSP, as the name says, can only be used to describe temperature dependent mechanical properties of viscoelastic materials and is based on the classical linear cumulative damage law (LCD) (Guedes, 2006). Although this method was developed for non-destructive material properties, it can also be applied to failure properties. Wide ranges of loading and environmental conditions are covered in the master curves obtained by the ATM using the TTSP (Miyano, Cai, & Nakada, 2008).

The ATM procedure is illustrated in Figure 17 and explained in (Miyano, Cai, & Nakada, 2008). After applying in the composite material various loading conditions for several different tests, the creep and fatigue strengths are obtained through master curves for any stress ratio, temperature, and frequency (Miyano, Cai, & Nakada, 2008).

In more detail, the ATM has three important conditions to consider that are enunciated in Figure 17. The "A" condition is applicable for all material that exhibit viscoelastic behaviour, therefore being composites and matrix resin. The condition states that the TTSP is valid for all strengths, non-destructive and destructive, meaning that it is applicable to all the types of testing. In addition, the 2nd condition dictates that the LCD law is for the monotonic loading, that means that LCD law is only suitable for the strength by monotonic loading, which is a static load that increases continuously. This condition is crucial to make the creep strength master curve possible to obtain. Finally, the condition "C" says that there is a linear dependence between the stress ratio of the cyclic loadings and the fatigue strengths. (Miyano, Cai, & Nakada, 2008)

Looking at the procedure of ATM, there is a path to follow until reaching to the desired results, so, a more detailed information about this methodology is needed. The first step consists of measuring through time, at the same temperature, the variation in modulus of the viscoelastic matrix. This test must be done for various high temperatures, so that at the end there are several modulus curves. Afterwards, there is the construction of the master curve of the modulus at a reference temperature that results from shifting the viscoelastic curves of the numerous temperatures obtained previously. This transition is possible with the time-temperature shift factor that is the measure of the acceleration of the life of the resin by means of the high temperatures. (Miyano, Cai, & Nakada, 2008)

Then, the following step is to attain the creep strength master curves, which is divided in two parts. The first one being to determine the constant strain rate strength master curve of the composites by taking into consideration the condition "A" and use it in the constant strain rate loading tests realised previously. The second part uses the condition "B" to convert the constant strain rate strength master curve to the desired one, the creep master curve of the composite. (Miyano, Cai, & Nakada, 2008)

To obtain the fatigue strength master curve, several fatigue test must be done at several stress levels, a zero-stress ratio, various temperatures, and a single frequency, also by using the first condition. At the end, from the master curves of both creep and fatigue strengths and considering the last condition, it is possible to obtain the creep and fatigue strength at any given frequency, stress ratio and temperature. (Miyano, Cai, & Nakada, 2008)

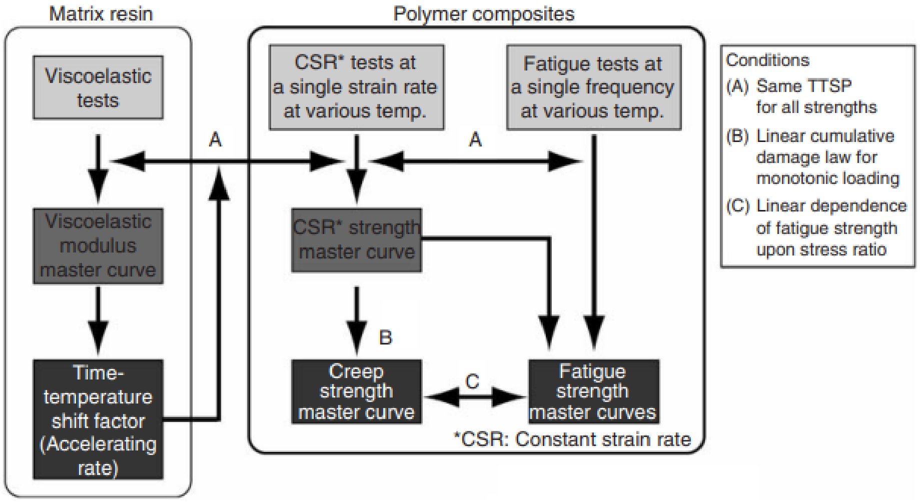

Figure 17: Procedure of ATM (Miyano, Cai, & Nakada, 2008)

The viscoelastic behaviour for TTSP is explained and analysed through a spring in Maxwell model, Figure 18, where the variation of temperature is represented by the viscosity of the dashpot that declines with increasing temperature (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018).

$$D_c(t) = \frac{\varepsilon(t)}{\sigma_0} = \frac{\varepsilon_E}{\sigma_0} + \frac{\varepsilon_\eta}{\sigma_0} = \frac{1}{E} + \frac{t}{\eta} \qquad (8)$$

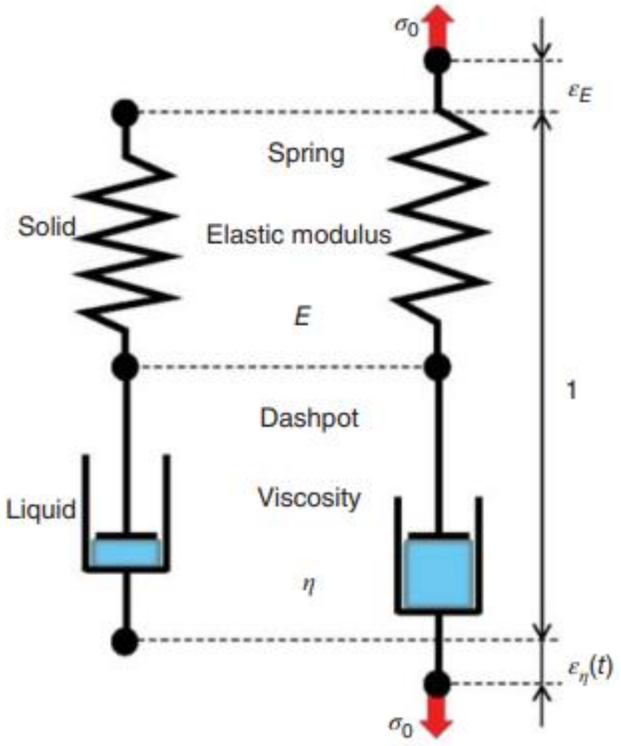

Figure 18: Maxwell model (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018)

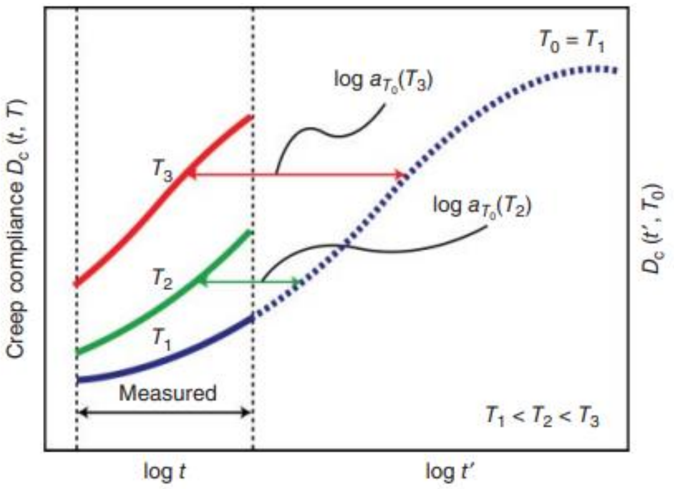

Figure 19: Example of creep compliance at various temperatures and the master curve for matrix resin (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018)

Looking at an example now from what was learned previously. From what was learned previously, to build a master curve of creep compliance (D_c) of the resin, different creep analyses are performed at several temperatures forming several curves, one for each test, with different temperature, as it is shown on the left side of Figure 19. With these different curves, the master curve is then formed by shifting each creep compliance curve horizontally, as shown on the right side of Figure 19 (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018).

The number of horizontal shifts is called the time-temperature shift factor $a_{T_0}(T)$, knowing that T_0 is the reference temperature and equals T_1. This shift factor is described by the following equation (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018):

$$a_{T_0}(T_i) = \frac{t_i}{t_0}, \ \log a_{T_0}(T_i) = \log t_i - \log t_0 \tag{9}$$

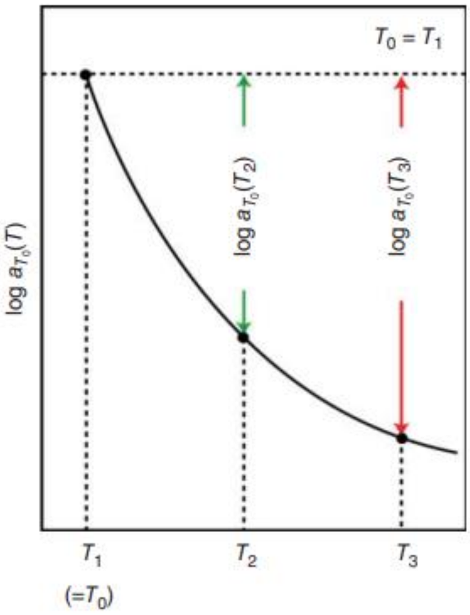

Figure 20: Time-temperature shift factor (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018)

Moreover, to assemble the master curve of creep strength of FRPs the procedure is similar to the one just explained, although instead of measuring creep compliance this time is creep strength σ_c. The shift factor is identical used for the master curve of the matrix resin. With this master curve exemplified in Figure 21, the long-term creep strength of FRP is predicted. (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018)

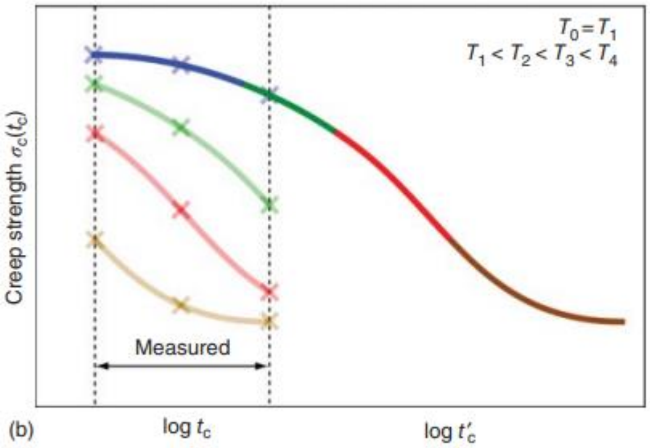

Figure 21: Master curve of creep strength in detail (Miyano & Nakada, Durability of Fiber-Reinforced Polymers, 2018)

Gathering all this information collected in this chapter, it is possible to predict some behaviours from the composite systems regarding its mechanical properties, and what type of effects it may shift with. For instance, the presence of fibres in FRPs should allow the composite system to have a stronger behaviour towards creep, since the viscoelastic behaviour comes from the matrix resin. So, if compared a graphic representing the master curve of creep compliance between a FRP and a polymeric matrix (the same used in the composite material), it is expected that the FRP's master curve shows a better resistance to creep through time. The quality of interphase between the matrix and the fibres, and the percentage of fibres would presumably have a big impact on how good the composite system behaves towards creep.

To verify this logical argument, a study from J. Raghavan and M. Meshii was used as support (Raghavan & Meshii, 1997). In this article, a unidirectional continuous carbon fibre reinforced polymer composite (AS4|3501-6) was the material analysed, with a viscoelastic epoxy matrix (3501-6). The results obtained in the study are partially illustrated in Figure 22 and Figure 23.

In both figures, it is shown that the epoxy matrix with the reinforcement of fibres reduces the magnitude of creep. A consequence of the reinforcing carbon fibres is the increase of the epoxy stiffness, therefore making the composite stronger. This verifies the previous assumption, that the addition of fibres in the matrix significantly alters the creep behaviour of that same matrix by reducing the creep magnitude and creep rate (Raghavan & Meshii, 1997).

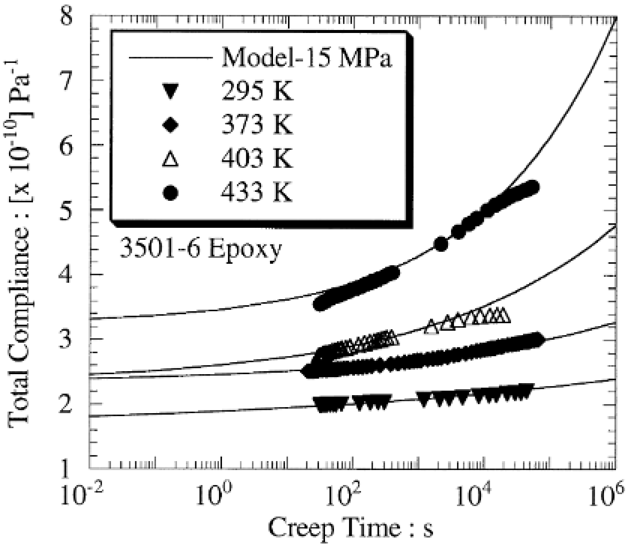

Figure 22: Experimental and model fit compliance of epoxy at a creep stress of 15 MPa (Raghavan & Meshii, 1997)

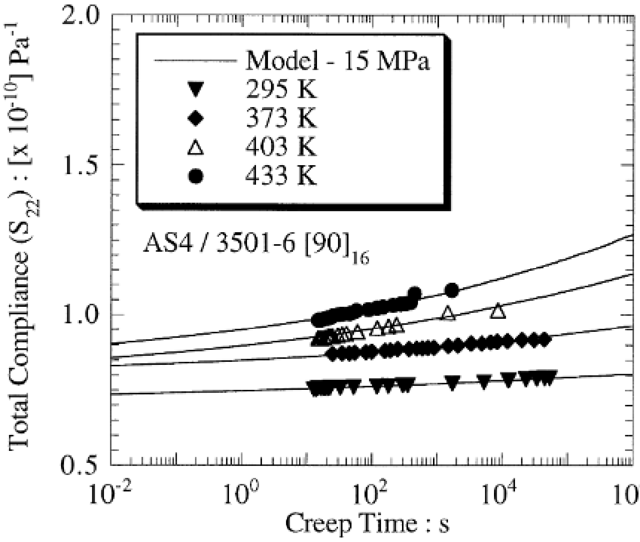

Figure 23: Experimental and model fit compliance of the composite at a creep stress of 15 MPa (Raghavan & Meshii, 1997)

2.3 Learning from Nature

In this chapter, a parallel is done between nature and advanced materials, as learning from living beings can serve as an inspiration for innovation. In fact, the relationship between synthetic materials and biology and chemistry, is called biomimetic, which according to the dictionary refers to "imitating nature or a natural process". Even though, the term is relatively recent, it has been of great use throughout history, which makes sense when we consider that it allows to mimic biology or nature that has been gone through evolution over 3.8 billion years, since life is estimated to appear on the Earth (Marshall, 2009).

Studying biological functions, structures, and principles of various living beings in nature has given the insights needed for the design and fabrication of various materials and devices of commercial interest (Bhushan, 2009). From molecular to macroscale level, there are numerous examples of mechanisms found in nature that are of great interest for human evolution, such as energy conversion and conservation, aerodynamic lift, self-cleaning, high adhesion, and others (Bhushan, 2009). These mechanisms in fact help finding solutions for more sustainable design, which due to global warming concerns is a major focus. One way to achieve it is with a lightweight structure where the materials strengths are optimal, consequently it reduces waste.

One famous example of this is "The Gherkin" building in the city of London, otherwise known as 30 St Mary Axe. The Gherkin with its external lattice structure shape makes the building stiffer and more efficient compared to traditional high-rise (Srisuwan, 2019). This building, located in the heart of London's financial centre shown in Figure 24, is inspired by the organism form and function of the Venus flower basket (Figure 25), a sea creature that feeds by directing water to flow through its body. Taking advantage of this, architect Norman Foster searched to manage the air flow around the outside of the building to use it to power a natural ventilation system, reducing the need for air conditioning by 50%. In addition, the lattice design and round shape of the Venus' flower basket sponge has the ability to disperse stresses caused by strong waters, which is also used in the Gherkin Tower to reduce wind deflections (du Plessis, et al., 2020).

Figure 24: Photo of Gerkin Tower in London (Mutuli, 2022)

Figure 25: Photo of Venus' flower basket (Euplectella aspergillum) (asknature, 2020)

For the thesis in hand, a parallel can be made between durability of the composite materials and ageing of living creatures. Nature offers a huge variety of species that present different lifespans and that live in different environments and conditions. In fact, even in the same group of animals there are some big differences in the life expectancy caused by evolution itself. Therefore, the research in biodiversity on planet earth is of extreme importance, the diverse forms of ageing existent are subjected to a lot of research, especially on how these compare to human ageing.

Ageing can be defined as the "progressive loss of physiological integrity, leading to impaired function and increased vulnerability to death" (López-Otín, Blasco, Partridge, Serrano, & Kroemer, 2013). Another term related to ageing that is commonly used in biology is senescence, which refers to the process by which a "cell ages and permanently stops dividing but does not die", definition retrieved from dictionary. Although, when talking about materials, senescence is not much relevant since it occurs at a cellular level because it is specific to living beings, a parallel may still be done with senescence and material degradation.

This topic is one of the most complex biological processes known. The multi-factorial issue has huge complexity and diversity, because across the tree of life the mechanism of ageing evolves and expresses in different ways making it very different from what is known in humans (Cohen, 2017). In fact, back into 1990, Medvedev estimated that there are more than 300 theories of ageing, although since then many of them have been refuted (Medvedev, 1990).

In the following subchapters, the focus will be on studying living organisms and try to understand characteristics that enhance their longevity. Firstly, the attention will go to Animals of various species, and then plants.

2.3.1 Animals

When looking at ageing, it is important to look at different types of animals, since their organisms fight ageing in different ways, conducting to different ageing theories. From mammals to invertebrates, animals present different lifespan. Slow growth, late maturity and late fecundity are some of the life history traits that long-lived species share (Edwards, et al., 2019). Consequently, evolution has rewarded them now with long lasting lives. The paper's objective is to focus on these evolutions in different species and try to understand this multi-factorial and very complex function of organism that is ageing (López-Otín, Blasco, Partridge, Serrano, & Kroemer, 2013). Although, when talking about the process of ageing the evidence is conflicting, whether it is similar across all organisms or particular to each species (Cohen, 2017). There are still many challenges and contradictions facing the ageing process, therefore, the topic has still a lot to move forward and many questions have still to be answered.

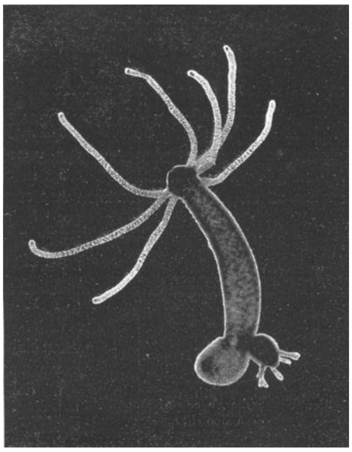

Figure 26: Photo of Hydra (Hydridae) (Flatters, 1912)

A very interesting case study is hydra (*Hydridae*), a very small freshwater creature from the phylum Cnidaria class of invertebrates. This unusual animal has the ability to regenerate and exhibit very low senescence, making it a very attractive model for ageing studies. In fact, asexual hydra apparently displays negligible senescence that is a consequence of the constant self-renewal of the stem cells (Tomczyk, Fischer, Austad, & Galliot, 2014), which are cells that generate other cells with specialized functions. This capacity of replacing the "outworn" cells of the organism gives hydra a sense of immortality, with its characteristics being unchanged for an almost unlimited time (Khokhlov, 2014). When comparing to materials, this would be the same as changing the whole material as soon as it starts to lose some properties/characteristics. Unfortunately, materials are not an organism and do not have the capacity to renew parts as desired.

Regenerative mechanisms are not that unusual in living organisms, from the fascinating example that is hydra, to mammalians, they all show some sort of regeneration of tissues and organs (Iismaa, et al., 2018). These key molecular mechanisms can be divided in two different types of reparative mechanism. The first one is epimorphosis, where the increase of the process of cell division occurring in tissues, also known as proliferation, precedes the development of new tissues (Agata, Saito, & Nakajima, 2007). In contrast, there is morphallaxis that is typically observed in invertebrates, and it consists of very low proliferation going instead through the re-patterning of pre-existing cells, like the example of hydra (Iismaa, et al., 2018).

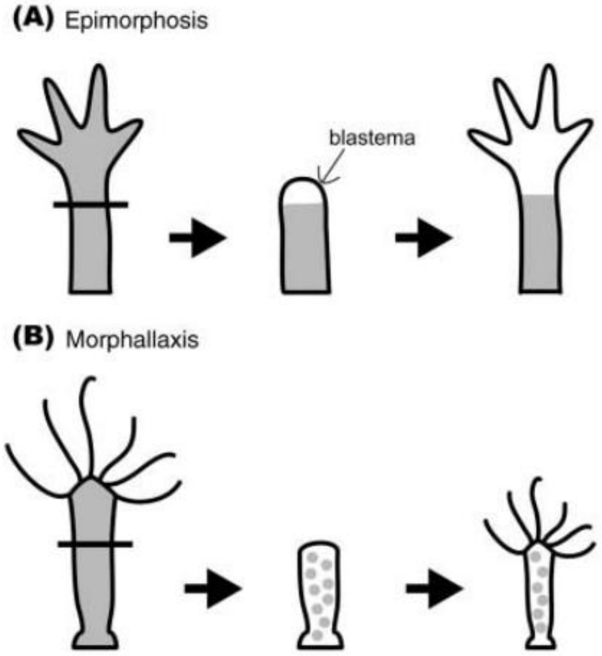

Figure 27: Types of reparative mechanisms, the grey area represents the old part and the white are the new ones (Agata, Saito, & Nakajima, 2007)

As mentioned, regenerative abilities differ from animal to animal as well as their mechanism. Some have the capability to renew all the tissues and organs, others are limited to specific body parts. In most animals, age affects regenerative repair this is why hydra example is good, because it is not affected by age and it has a regeneration without proliferation, like the one represented in Figure 27 (B).

Ageing is a very complex biological process and can have many factors including genetic/molecular aspects and external influences. In living beings, the production of reactive oxygen species (ROS) leads to oxidative stress, which contributes to accelerate the ageing process (Getoff, 2007). ROS are a range of by-products of molecular oxygen that occur as a normal attribute of aerobic life (Krumova & Cosa, 2016). Oxygen stress is caused by the production of reactive oxygen transients, which can be produced by sunlight, ionizing, microwave and ultrasonic radiation (Getoff, 2007). This leads to the formation of antioxidant to defend and slow damage to cells caused by ROS. Oxidative stress because of the imbalance between ROS and antioxidant is related to numerous disorders, such as cancer and cardiovascular disorders (Krumova & Cosa, 2016). These types of consequences have severe effects on cells therefore have a direct impact on the longevity of animals.

In fact, when talking about ageing there is a theory of ageing called the free radical theory of ageing defending that oxidative stress produced by the free radical (OH, peroxyl, ozone and other oxidizing species) is toxic, which is any molecular species that has an unpaired electron including ROS (Liochev, 2013). Increase damage to important cellular targets and growth inhibition are some of the negative consequences of oxidative stress, although it can also have some positive impacts, for instance decreasing metabolic rate and increasing cellular generation time (Liochev, 2013).

The free radical theory of ageing focuses on damage caused by free radicals derived from oxygen, consequently this theory makes part of a big classification of theories called the damage theory of ageing, focusing on theories that have an impact on cellular waste accumulation (Viña, Borrás, & Miquel, 2007).

Within this theory of ageing, it is possible to see many examples of animals that control in different ways the oxidative stress. One example is the ocean quahog clam (*Arctica islandica*) that is claimed to be the oldest recorded non-colonial animal with a remarkable 405 lifespan years registered (Bodnar A. G., 2009). This bivalve clam when comparing to other shorter-lived bivalves has the capacity of forming low ROS causing a low standard metabolic rate, presenting strong arguments for the free radical theory of ageing (Strahl, 2011). Although, there are more factors in play such as the lifesaving lifestyle and high cellular defence and repair mechanisms that aid the *Arctica islandica* to achieve high lifespans (Strahl, 2011). Studies have shown that environmental factors can have a great impact in the production of ROS and the accumulation of damage in animals from different locations (Lesser, 2006).

Figure 28: Photo of Ocean Quahog Clam (Arctica islandica) (Bay-Nouailhat, 2008)

Other long-lived invertebrate species includes the sea urchins (*Echinoidea*), another specie that appears not to age. Such as the ocean quahog clam, this specie is also referred to having negligible senescence (Finch, 1990). The undefined growth and continued reproduction throughout their lifetime are very specific from sea urchins, whether short-lived or long-lived. The red sea urchin (*Strongylocentrotus franciscanus*) is amongst the longest-lived animals and can live up to 200 years (Ebert & Southon, 2003). The lifespan of sea urchins is very wide, as different species of sea urchins have very different reported lifecycle starting from 4 and going to more than 100 years. Therefore, explaining interest by scientists to understand it and explain the differences between short-lived and long-lived sea urchins.

After several conducted studies, it is still uncertain if the oxidative stress in sea urchins has much impact on ageing or not, and theories are more towards the gene regulation theory of ageing (Bodnar A. G., 2015). This theory proposes that senescence is the result of changes in gene expression (Weinert & Timiras, 2003). The idea that ageing is a programmed mechanism influenced by genes is very debated and currently discussed. In this paper, this theory won’t be furthermore explored because of its complexity and difficulty to relate it to materials since it focusses on gene expression.

Figure 29: Photo of Red Sea Urchin (Strongylocentrotus franciscanus) (McDaniel, 2016)

Another animal that shows considerable longevity is the Greenland Shark (*Somniosus microcephalus*) that was recognized as the world's longest-lived vertebrate a few years ago, with an estimated lifespan of at least 272 years (Edwards, et al., 2019). Although the interest is still relatively small, this shark specie is known about several aspects, such as physiology, ecology, and behaviour. In fact, the free radical theory of ageing was initially thought to be linked with the exceptional lifespan of the Greenland shark, but after some investigations the oxidative status did not justified the shark specie longevity and it was explained by ecological features specific to these species, such as the adaptation to cold waters and deep dives (Costantini, Smith, Killen, Nielsen, & Steffensen, 2017).

The free radical theory of ageing has been chosen to be talked about because it is a damage theory, which makes it easier to compare to materials. However, there is another class of theories that can also facilitate that comparison, such as the wear and tear theories. In this theory, the physiological work of cells is what causes ageing (Pearl, 1928). The theory is essentially studied in the fruit fly (*Drosophila melanogaster*), and it has shown that the range of temperature in which these insects can live has a correlation to their lifespan (Miquel, Lundgren, Bensch, & Atlan, 1976) (Molon, et al., 2020). Longevity increases with later maturation of the larvae, which is prolonged by colder room temperatures.

More research has been conducted since the early 20th century, and the explanation found for the relationship between longevity and room temperature has to do with the adjustment on the oxygen and energy consumed by these flies (Fleming & Miquel, 1983). This theory suggests that the metabolic rate is an important factor in determining the lifespan of an animal, as a high-rate metabolism translates in quicker ageing (Molon, et al., 2020).

Figure 30: Photo of Fruit Fly (Drosophila melanogaster) (Acharya, 2017)

2.3.2 Plants

On the other hand, the holder of all records in longevity, are plants. A lot of research is done to learn from the mechanisms of ageing in plants, specially to help fighting it in humans. As in humans and in animals, ageing in plants is multi-factorial, as it can be caused by telomere depletion, loss of proteostasis, epigenetic changes, genomic instability, and others (Popov, Syromyatnikov, Franceschi, Moskalev, & Krutovsky, 2022). Therefore, in order to fight these ageing factors, plants developed a number of mechanisms to extend their life. The following text looks at those mechanisms and tries to explain it.

Firstly, it is important to characterize ageing in plants because plant development differs from animals, which has considerable implications in the mechanisms associated with age-related changes. While an animal lifespan is defined as the survival of the whole body, in plants it is not that easy. Plant’s lifespan is defined by the indeterminate growth of vegetative meristems and ageing is seen as the deterioration process that relates probability of death with chronological age (Watson & Riha, 2011) (Van Doorn & Yoshimoto, 2010). So, the equivalent to animals’ death is not very clear, but the recommended term is plant senescence.

Meristem is a proliferating tissue found in plants that is composed of self-renewing stem cells and is responsible for continuous growth of perennial plants, which are plants that live more than 2 years and the main focus here when talking about longevity (Tsukaya, 2014). For this reason, it is difficult to clarify individual plants, as meristems can independently regenerate into a new organism.

Another distinction that must be clarified is the use of the term senescence that also differs in animals and in plants. In plants, it refers to the process that leads to death at the organ and tissue level, while in animals it is manifested by the deteriorating effects of ageing (Lim, Kim, & Nam, 2007). Therefore, during lifetime of perennial plants, leaf senescence occurs multiple times consequently creating the beautiful landscapes of trees in autumn. This process is reversible, and it is rather orchestrated, and it starts with degradation of other organs and cellular structures (Watson & Riha, 2011). So, leaf senescence looks more like a step of leaf development, causing some ambiguity to say that senescence in plants is an age-related process.

In plants, such as in animals, a lot of longevity is connected to the efficiency of defensive mechanisms. Animals in their defence against predators rely more on physical attributes such as claws, camouflage, mimicry, and teeth. Even though that some animals use other types of methods to defend themselves for example with chemical mechanisms like poison, or even by adapting to the environment around them.

In plants the defensive mechanisms differ a bit since it is made to protect them from a different type of predators and also because plants are fixed and do not move around. Two types of defences exist in plants, the one to protect from insect herbivores by obstructing them and another to call for "help". The first one, requires a more direct defence with both physical and chemical barriers that affect insect herbivore's growth, reproduction, development, etc. The other type of defence is by attracting natural enemies of the herbivores therefore making it a dangerous environment for insect herbivores (Belete, 2018).

The longest-living tree in the world is called *Methuselah*, is located in California and has been around for over 4,800 years (The Gymnosperm Database, 2007). The tree's name refers to a biblical figure that is considered to be the oldest man who ever lived with 969 years old when he died. When referring to longevity it is a very common name, for example in the fruit fly there is a gene that has shown to be correlated with the *Drosophila melanogaster's* lifespan, that gene was named methuselah (Sgrò, Belinda Van Heerwaarden, Wee, Hoffmann, & Lee, 2013).

Figure 31: Photo of Methuselah, the almost 5,000-year-old bristlecone pine (Piriya Photography, 2020)

In fact, this long living tree belongs to the Great Basin bristlecone pine tree species (*Pinus longaeva*), the longest living organism on Earth. The secret behind this longevity is still far from being unveiled, with many theories being advocated. These range from defensive mechanisms against insects to physiological traits that improve survival in dry and harsh habitats. The famous bristlecone pine has significant defences against bark beetles when compared to other pines, such as higher constitutive monoterpenes, resin ducts and wood density (Bentz, Hood, Hansen, Vandygriff, & Mock, 2017).

Another important aspect is the ecosystem that the pine is integrated, as some environmental conditions favour some pine species over others, such as the Great Basin bristlecone pine that to endure longer prefers a warmer and drier climate (Smithers, Alongi, &

North, 2021). This characteristic is in fact very promising for the upcoming future, to facilitate the liveability in severe weathers that will be occurring due to global warming.

Almost every tree species lives for many years and that is due to some key behaviours that favour a long lifespan. As mentioned by Lanner, those key behaviours are creating a structure that can be expanded, making the structure redundant, optimizing the structure, hormonal coordination of growth activities and defending the structure from enemies (Lanner, 2002). In another study, the relevance of the tree size is studied, as it was proved that optimal body size slows down senescence, therefore contributing to longer life (Dani & Kodandaramaiah, 2019).

Although trees seem immortal, they still age due to transcriptional inadequacies and genomic failure, even if their main cause of dying comes from "natural causes" such as harmful insects and environmental disaster (for example fires). As mentioned previously, the location of which the tree is also a major factor for its survival, to avoid starvation and harvest.

The free radical theory of ageing cannot be used to describe ageing in plants, because of the differences in terms of organisms. But oxidative damage is very present in plant's health, with the risk of affecting it at biochemical and physiological degree. As mentioned previously, in living organisms there is the need to protect cell structures and biomacromolecules from the oxidative harm that can be caused by ROS, which requires some type of defensive mechanisms such as enzymatic and non-enzymatic antioxidants (Iriti & Faoro, 2008).

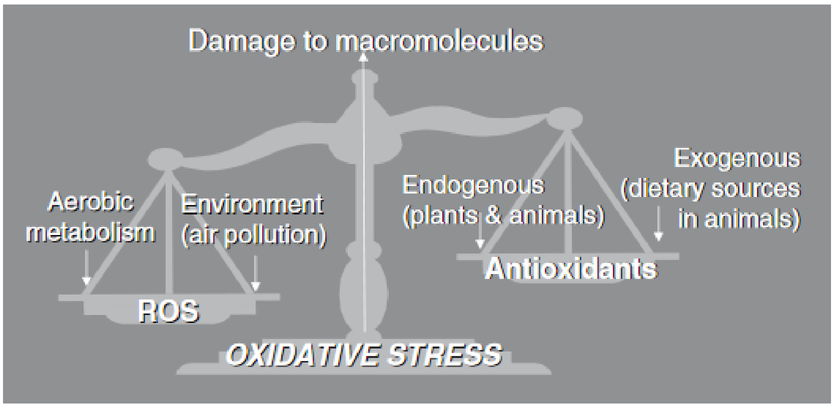

Figure 32: Prooxidant-antioxidant balance leading to oxidative stress (Iriti & Faoro, 2008)

Plants can manifest in several ways the damage caused by ozone effects. One way of manifesting it, is by so-called invisible damages, such as dysfunction of transpiration and water, decrease of photosynthetic activity, detrimental effects on flowering, and others (Black, Black, Roberts, & Stewart, 2000). Collectively, this affects the plant's growth and yield. In terms of antioxidant availability, plants are more capable than animals, as they can synthetize vitamin A, E and C, that has been proved to be effective to contradict ROS (Iriti & Faoro, 2008).

Besides the use of antioxidants, plants possess another mechanism of defence that consist of closing the stomata (small pores in plant tissue that are used for gas exchange) in order to avoid pollutant intake. Animals, on the other hand, can prevent the ozone exposure by escaping to less polluted areas.

Stomatas allow for controlling the gas exchange in plants, which consequently helps control the oxidative stress exposure. The tissue valve is composed of two guard cells, one on each side, and the opening and closure of it is managed by the turgor pressure (Marom, Shtein, & Bar-On, 2017). The turgor pressure is caused by water that accumulates and ends up pushing it against the cell wall of plants. This means that when the guard cells are turgid the stomata open, and when they are flaccid the stomata closes.

Moreover, learning from ageing in plants have allowed to produce anti-ageing products based on free radical scavengers to protect the skin against wrinkles and skin disease. The use of medicinal plants for health care is normalize and in some cases the effectiveness is high. Some of those plants utilized to fight ageing are *Aloe vera*, *Vitis vinifera*, *Camelia sinensis* and others. From their free radical scavengers are retrieved as they play an essential role as antioxidant compounds in those plants already (Sharafzadeh, 2013).

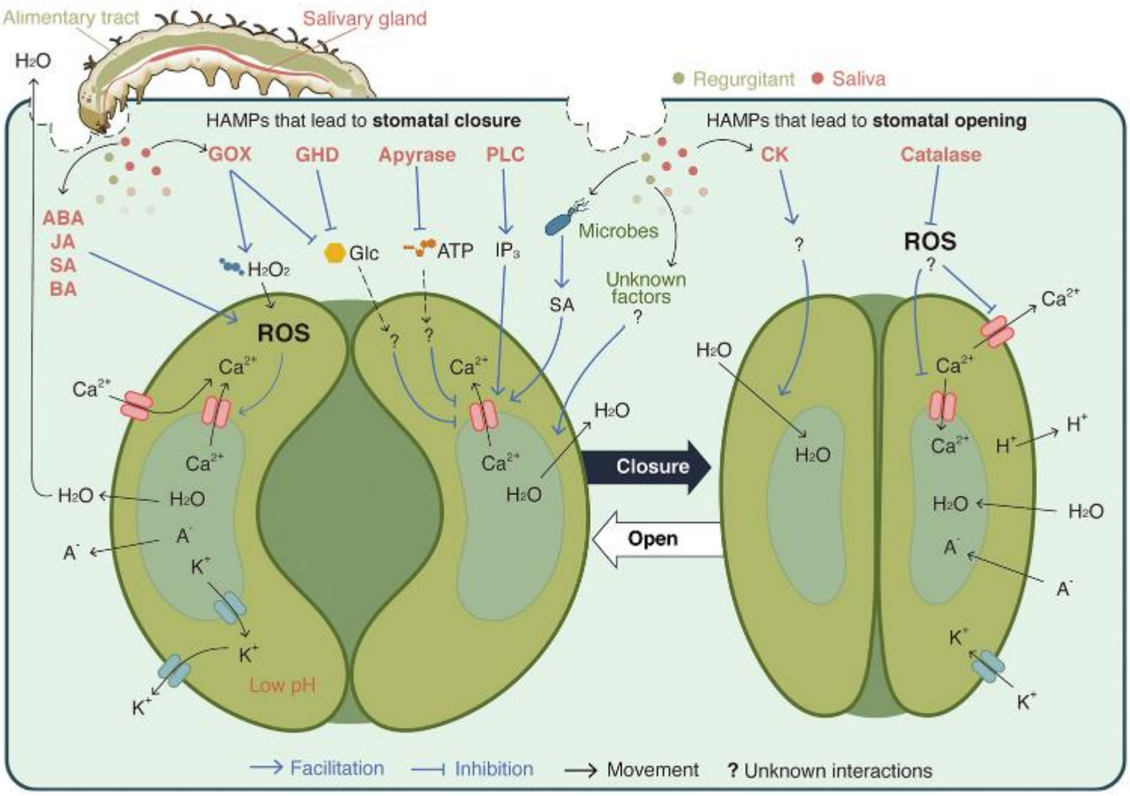

Figure 33: Potential responses of plant stomata with herbivores and the environment (Lin, et al., 2022)

2.3.3 Natural Materials

Living organisms are great to learn about ageing behaviour, but to adapt them to materials is a little bit more complex. That is not the case of natural materials such as rocks. These materials present in the planet are easier to compare, because mechanical behaviour and durability can also be studied in them. Therefore, they are a very good case study to learn from nature and maybe adapt it to composite systems, because natural mechanisms may exist to fight ageing and degradation. Although, the perspective in this situation must be wider from the ones seen with the animals and plants, as natural materials could possibly be used directly as an addition or reinforcement to the composite system.

The most common natural material is rock, that is defined in geology by the combination of minerals, and it can be classified in three main groups depending on how they were formed. The first group is sedimentary rocks, that defines those that originate from precipitation of minerals from water, or by when particles settle out of air or water. Those that are products of lava by molten rock cooling and solidification, are designated as igneous rocks. And finally, the metamorphic rocks that are a result from other rocks that were affected by heat and pressure underground.

Type of Rocks	Some Examples
Sedimentary	Sandstone, Shale, Iron Ore, Rock Salt
Igneous	Granite, Scoria, Pumice, Obsidian
Metamorphic	Gneiss, Marble, Phyllite, Quartzite

Table 2: Classification of rocks (AMNH, 2008)

In addition, when using these rocks for structural applications there is the need to be careful towards time-dependent deformation effects such as creep and fractures in the interaction within the structure (Sijing, 1981). The study of creep behavioural on rocks is very important because in geology a lot of long-term stresses are considered in nature, such as volcanoes, landslides, rock massifs and others (Amitrano & Helmstetter, 2006).

In a study conducted on weak rocks, durability was assumed to be influenced by the composition and textural features (Martinez-Bofill, Corominas, & Soler, 2008). The textural features can be divided in two parts, with the first one referencing homogeneity and heterogeneity of samples. While the secondary feature refers to the grain size distribution, that can be sandy or muddy, and have or not a fine matrix (wacky - sand sized grains with grained clay matrix).

The performed study also revealed that the matrix has significant effect on the long-term durability and, in some cases, homogeneity showed to be positive (Martinez-Bofill, Corominas, & Soler, 2008). Therefore, the main textural feature may seem important, and for that reason it looks important to characterize homogeneous texture. Homogeneity is obtained when a grain-size and matrix distribution are uniform and regular.

To better study rocks in general, their behaviour under deformation was characterized as anelastic (Brennan, 1981). Anelasticity is defined as being a subset of linear viscoelasticity, where it presents a unique equilibrium relationship that allows anelastic materials to complete recover (Zener & Siegel, 1949). This viscoelastic behaviour of rocks is especially important to explore earth’s seismic activity and their wave dispersion, and in addition, to approximate their behaviour to polymers.

In fact, in some situations a rock can present different viscoelastic behaviours depending on the mineralogic presence in the microstructure of the material. This is the case for the partially molten granite, were with crystal content over 40% the rock shows higher internal friction (Bagdassarov & Dorfman, 1998). The same study also revealed that for granites with more than 60% melted phase, the creep resistance rises with the increase of melted phase (Bagdassarov & Dorfman, 1998).

There is an increased necessity to analyse creep behaviour of rocks, as those are crucial for assuring the stability of underground structures. So, focusing on creep, it is shown in Figure 34 that creep phenomenon is similar to polymeric materials studied previously. This resemblance translates in elastic and viscous characteristics from the rocks. A lot of factors have influence in the creep behaviour of the rocks such as temperature, moisture size and shape effects, water pressure, stress conditions, etc (Kim, 2013). The high number of physical environmental effects and forms of loading make it more difficult to obtain an accurate analysis of creep behaviour.

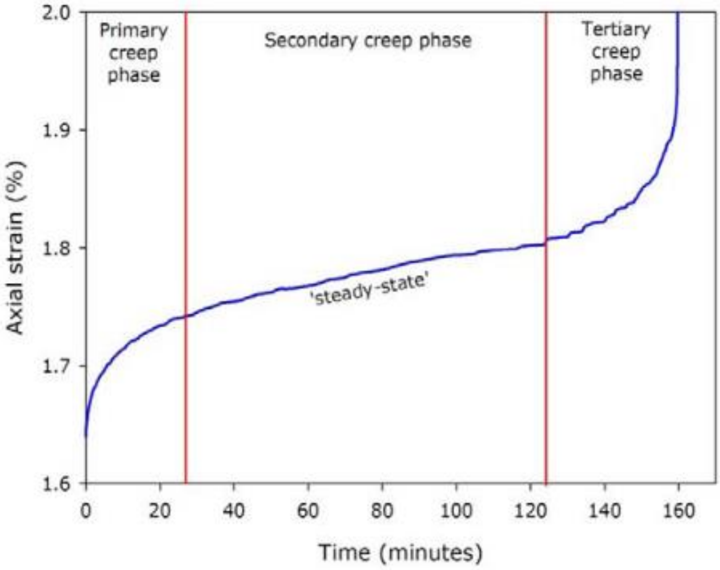

Figure 34: Typical creep curve for rocks (Heap, 2009)

Nevertheless, not all rocks follow the typical curve shown in Figure 34. In some cases, the steady state creep is not observable like for brittle rocks, and in other cases the last stage is not reached, or it is delayed for very long times, like it happens with more ductile rocks (the example of the rock salt) (Paraskevopoulou, et al., 2018).

Misra, in his Ph.D. thesis, investigated the time-dependent deformation in rocks and discovered that temperature has a huge impact. Temperature rise negatively affects the material by rising significantly the creep rate and the creep strain (Misra, 1962). In another study, the rate of water absorbed was noted to have an impact on creep behaviour, which concluded that in saturated rocks creep deformation is 2 to 7 times greater than in dry rocks (Kim, 2013).

Additionally, the creep behaviour of rocks is highly dependent on the temperature, the same happens for metals. In a study conducted by Misra and Murrell, the creep deformation for temperatures below $0.2*T_m$, T_m being the melting temperature of the rock, depends logarithmically on time and is proportional to temperature and stress, while values above that exhibit a different behaviour as creep resistance decreases significantly with stress and temperature (Murrell & Misra, 1965).

Limestone is a sedimentary rock and has been an important case study lately because of its possible use in nuclear waste repository. In a study made in 2018, this brittle rock was compared to granite and some differences were detected. Limestone appeared to exhibit better resistance to creep than granite, which was explained by the polymineralic components that are present in the granite (Paraskevopoulou, et al., 2018). This heterogeneity causes incompatible strains generated within the sample because of the different creep rates from each mineral (Paraskevopoulou, et al., 2018).

In addition, the same research found out that the limestone samples reaching failure were the ones that presented more viscosity, which relates the level of viscosity to the long-term life of brittle rocks (Paraskevopoulou, et al., 2018).

In the same way, the study of granite is also fundamental for projects of nuclear waste disposal, due to its abundance in certain regions/countries. Hong and Jeon discovered back in 2004 that the presence of water in granite negatively affects creep resistance, as it reduces the secondary stage creep and increases creep rate in that same stage (Hong & Jeon, 2004). The manifestation of water has then a great impact on creep behaviour of rocks.

Moreover, there is a natural material that is widely used in civil engineering, that is adobe. This material made from earth is a combination of sand, clay, and silt, resulting in an extremely durable material in dry environments. But just like it was seen for rocks, adobe also presents less creep resistance with the increase of water presence (Clifton & Davis, 1979).

Moreover, metals are also natural materials and their information about creep behaviour is more extensive and clearer. So, here is a quick review on metals and their creep resistance to search for ways to improve it.

Just like polymers and ceramics, the creep behaviour is divided in three stages, where the secondary creep, or steady creep rate, is described by the following equation named Mukherjee-Bird-Dorn (Mukherjee, Bird, & Dorn, 1968):

$$\dot{\varepsilon}_s = \frac{AGbD}{kT}\left(\frac{b}{d}\right)^p\left(\frac{\sigma}{G}\right)^n \tag{7}$$

In this expression the steady creep rate is function of several variables, the applied stress (σ), the temperature (T), the grain size (d), and some constants that depend on the material and creep in hand, which are the Burgers vector (b), the stress exponent (p), the shear modulus (G), the diffusion coefficient (D), the Boltzmann's constant (k), the stress exponent (n) and the dimensionless constant (A) (Malakondaiah & Rao, 1985). Two remarks can be made after analysing this equation, the first one is that the applied stress and temperature determine the creep conditions. In addition, the impact that grain size has on the creep rate, because from the equation it is possible to retrieve that increasing the grain size would reduce the creep strain rate of deformation. Consequently, designing the microstructure of a metal is one way to improve creep resistance.

When talking about alloys, the design of microstructure has also showed to be relevant in titanium alloys. A study conducted by Leyens and Peters found that a lamellar microstructure is more prone to reduce creep rate than a bimodal microstructure, because it provides more geometrical resistance for dislocation motion (Peters, Hemptenmacher, Kumpfert, & Leyens, 2003). To better understand the geometrical layout of both types of microstructures, the differences are shown in Figure 35. The lamellar microstructure looks geometrically more arranged organized, while the bimodal one seems to have no order.

In Figure 36, the creep rate is presented in function of the shear modulus (G), and it is very notable the improvement it has. In fact, the linearity of the curve really emphases on improving the elastic modulus in order to achieve better creep resistance (Kassner & Pérez-Prado, 2000). In addition, on the study conducted by Kassner and Pérez-Prado they mention that this influence also occurs when fluctuating the Young's modulus (E).

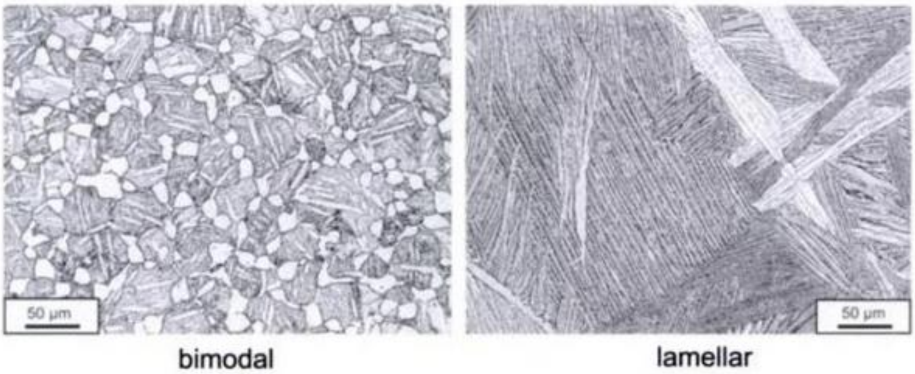

Figure 35: Types of microstructures in titanium alloys (Peters, Hemptenmacher, Kumpfert, & Leyens, 2003)

Finally, another way to improve creep resistance in metals is by doing what is done with the polymer, that being using reinforcements. Fibres would be the most used case to reinforce although the use of particles also attains improvement. In an experimental study on titanium matrix composites reinforced with silicon carbide fibres the improvement was evident, as the titanium composites exhibited substantial creep resistance compared to the titanium alloy Ti-6Al-4V (Leyens, Hausmann, & Kumpfert, 2003). Additionally, another study was made where they analysed those composites to see what can improve their creep behaviour. The study concluded that a lot of factors can have a huge impact on creep resistance, such as interfacial debonding, fibre fracture, fibre strength and distribution, fibre orientation, chemical composition and creep behaviour of the matrix, and others (Yang, 1998). Most of these factors are also relevant for the study of creep in PMCs.

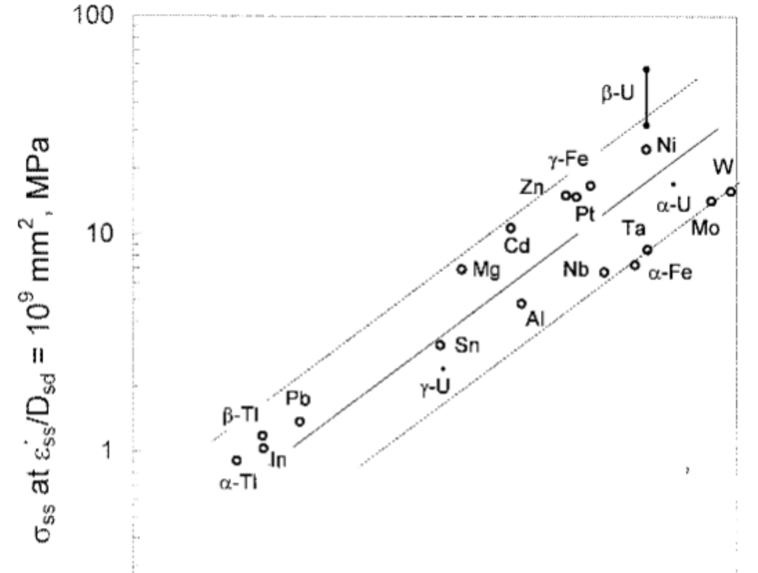

Figure 36: The effect of shear modulus on the steady-state flow stress (Kassner & Pérez-Prado, 2000)

3. Discussion

The mysteries of ageing are far from being unveiled and there is still a lot to explore. Better understanding ageing mechanisms of plants and animals is very important, not only for increasing the lifespan of humankind, but also it can give new perspective on materials ageing.

There is a recent study on “immortality” that has been very much talked regarding the rejuvenation capability of a specie of jellyfish named *Turritopsis dohrnii*. The research on this biologically immortal jellyfish was able to emphasize some genes and pathways related to telomere attrition, stem cell exhaustion, cellular senescence, genomic instability, and others (Pascual-Torner, et al., 2022). These new findings could be very important for understanding rejuvenation and proliferation, although for materials it may for now not look relevant, but in the future with more advanced materials it may be useful.

The possibility that in the future materials would behave more like living organisms, opens a new all chapter for ageing in designing materials. That would approximate to mechanisms used by animals and plants, such as intercellular communication or auto repair mechanisms.

A mechanism of ageing used by animals, humans and even in plants that could possibly be explored with the right technology is telomerase. Telomerase is an enzyme that helps repairing the end of the chromosomes. This is needed in living organisms because after each cell division (mitosis) a part of the tip of the chromosome, that does not have any DNA information, is consumed and it needs to be restored before it starts consuming DNA information in the chromosome. The end of the chromosome that is consumed and consequently restored by telomerase, is called telomeres. In fact, telomeres can be seen as a coating of chromosomes, and they are restored automatically by the telomerase enzyme (Blackburn, 2017). Having this ability in materials, where the coating of a material is automatically applied and maybe even produced, could mean serious benefits in maintaining the mechanical properties of materials through in-service life.

Focusing now on non-programmable mechanisms of ageing that can be taken into consideration nowadays, it is important to consider the free radical theory of ageing. This theory is still very controversial as there are some researchers that contradict while others suggest it remains a viable theory (Liochev, 2013). The free radical theory can be classified in two different theories of ageing: genetic mutation and cellular waste accumulation.

The effect of oxidative stress in cellular organism is undoubtedly and, in many cases, strongly related to ageing (Liochev, 2013). But when looking at materials the free radical theory of ageing is difficult to visualize, since terms such as ROS and cellular mechanisms do not exist. Therefore, the comparison can be made to a certain extent and one way to look at it is by analysing the effects that oxygen may have on composites. To do so, one can look at the degradation effects of atomic oxygen in composite materials in space applications. The reason why this topic was chosen is due to the wide use of advanced composite materials in deployable structures and spacecraft structural components.

Atomic oxygen (AO) is composed of only one oxygen atom, and it does not exist naturally for very long on Earth. It can be found in abundance in low Earth orbit (LEO), the closest orbit to Earth that goes from 160 km to less than 1000 km above the Earth's surface (ESA, 2020). One big threat that AO present is erosion and it is considered to be one of the main reasons for the degradation of the surfaces exposed (Samwel, 2014), because it causes materials to be susceptible to mass loss and surface morphology changes, especially PMCs (Reddy, 1995). The resin surface is the main affected area which consequently impacts the mechanical properties of the material and can cause significant damage if fibres become eroded too (He, Suliga, Brinkmeyer, Schenk, & Hamerton, 2019).

Recently, a solution has been found that allows for polymers and composite materials to prevent degradation in LEO. A multi-layered protection material that eases the impacts of AO and ultraviolet (UV) without losing any performance (Smith, et al., 2021). To increase adhesion to the surface of the substrate and to ensure no pinhole erosion the multi-layered protection barrier was deposited using a plasma-enhanced chemical vapor deposition (PECVD) system (Smith, et al., 2021).

From previous observations on living being organisms, it is understood that long live species have natural mechanisms to contradict the oxidative damage, either by producing more antioxidants or producing less ROS. So, there are two ways to look at this when talking about materials, either the composite system is protected by a layer of antioxidants, or the composite material has a mechanism that allow it to be less expose to AO. The ideal solution may even be a mixture of both, where it is possible to protect the structure from degradation using for example a retractable component that is also covered with an antioxidant layer. In fact, using antioxidants as coatings is already being used in polymer materials and one of the main challenges of designing an antioxidant material is the strength and type of interaction between antioxidant and polymer that directly affects the efficiency of the protection (Brito, et al., 2021).

Another possibility arises from the plant defensive mechanisms against oxidative stress. As mentioned previously in the chapter describing the state of the art, animals and plants have other than biochemical responses to this danger. While animals can move around and avoid danger exposure to oxidative stress rich environments, plants do not have that option and instead developed a controlling mechanism of that exposure. In fact, what happens is that plants have little holes in their tissue called stomata and uses them by opening and closing to regulate the exposure of ROS.

Such as wood and bone, guard cells are considered to be fibre composites, therefore a mechanical model can be made (Fratzl & Weinkamer, 2007). Hence, the walls of guard cells' are anisotropic bio-composite materials with microfibrils reinforcing the softer matrix material that is composed of lignin, pectin rich hemicellulose, and structural protein (Marom, Shtein, & Bar-On, 2017).

This proximity of the cellular tissue with a mechanical model makes it easier to visualize the viability of a composite material that shares the same functionality. However, this work is focused on ageing and having a composite that opens and closes does not seem very useful, except if this functionality is used to control the AO in the previous example. Here, the use can be interesting not as a composite itself, but an outer layer to protect the main composite system by controlling the intake of AO, therefore possibly enhancing the durability.

This outer layer protection is very common in the modern world, as coating protections are widely used in many applications and for all sorts of materials. But the idealization made here is towards a mechanism that would serve as coating, and not, for example, the simple use of a spray that is directly applied on the material, such as ceramic coating spray that protects cars' paint from the surrounding environment (water, dirt, and others). Instead, this outer layer protection would have the same function and mechanisms has the stomata in plants.

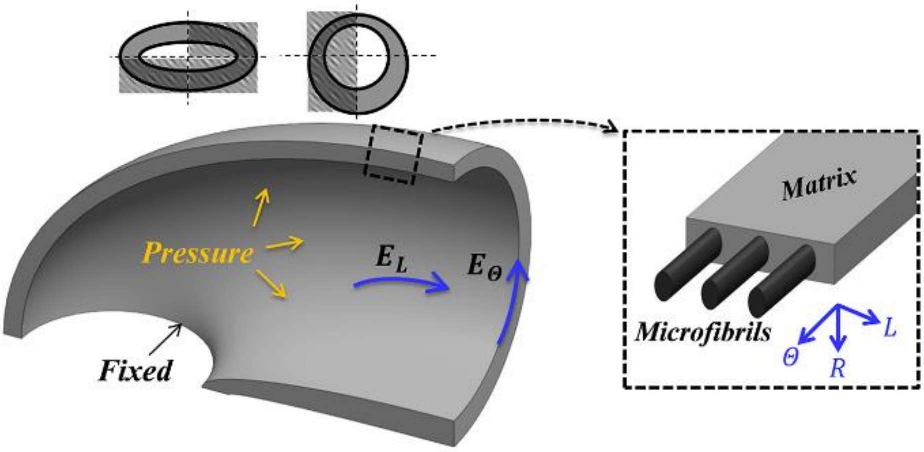

Figure 37: Mechanical model of the guard cell (Marom, Shtein, & Bar-On, 2017)

In fact, some materials with similar mechanisms already exist and are used in many applications. That is the example of the breathable fabrics that are designed to control the water transmission from one side to the other. This closely woven fabric has incorporated a combination of microporous and hydrophilic membranes that were developed with inspiration in biomimetics (Mukhopadhyay & Midha, 2008). Even though the function is different, the principle is basically the same, controlling the amount of exchange between two sides of a certain element.

There exist some variety of waterproof breathable fabrics, one of them, under the trade name of Stomatex, was inspired by the transpiration system implemented in plants, also known as stomata. The composite technical textile is knitted in a way to possess several small domes with little pores on the centre of each, and with the movement of the textile any excess of moisture and heat rises into the domes and is release (Maity, Chauhan, & Pandit, 2022) (Wood, 2019). This specific fabric is used in compression garments for athletes, to improve recovery and performance, or to give medical assistance (Wood, 2019).

In addition, has it formerly seen for rocks and adobe materials, water has a significant influence on the creep deformation rate of the natural materials. Therefore, it may be also interesting the usage of a system that can also controls the water transmission, which may result in an improve creep resistance.

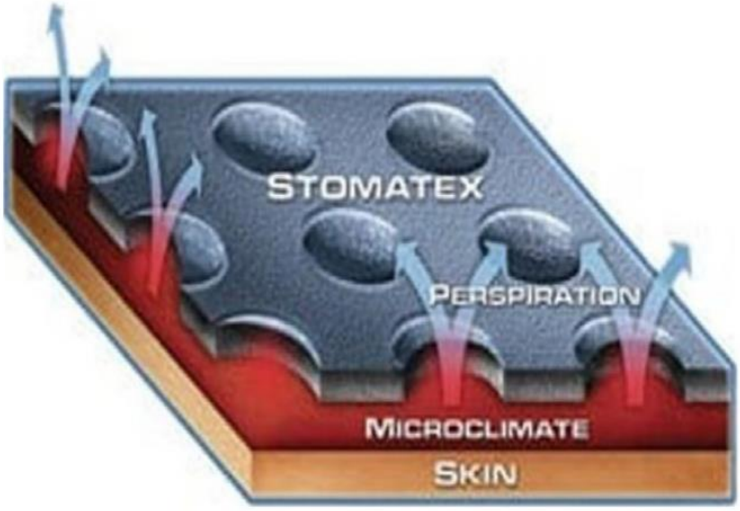

Figure 38: Structure of Stomatex (Maity, Chauhan, & Pandit, 2022)

When talking about AO and the dangers that it could bring to a material, in fact the talk should be regarding all the environment that a material is exposed to, such as humidity, radiation, temperature, etc. Depending on the composite system and on the application, it has different environmental factors to be considered. Nowadays the focus is towards climate change and the implications it can have on materials.

As it was seen for the hydra, they have the capability to renew their cells to maintain their age which results in negligible senescence. This potential of regeneration is all over animals and it allows them to delay ageing and to treat injuries. Applying this mechanism to materials would consist in regenerating the matrix when mechanical properties start to deteriorate. The possibility that in the future composite systems could behave closely to living organisms would open many doors in resisting ageing.

Using morphallaxis as the regeneration type would assume that any void or crack in a material could be fulfilled by re-patterning existing material. Materials with human tissue have already been experimented, in fact, recent research at the Wyss Institute developed a 3D bioprinter that allows to manufacture living tissues with considerable cellular density and implanted vascular channels (Skylar-Scott, et al., 2019). So, the integrability of living tissues or organisms in materials is not that distant.

But the use of living organisms is even not necessary to integrate the repairing function in materials. The use of computational equipment may serve this purpose. For instance, chips nowadays are getting smaller and smaller while keeping improving the computational capacity. This opportunity would be just like adding a micro-computer to a composite material where sensors could be used to detect any mechanical properties loss or any defect and possibly react to it by adapting the material. In the same way that living organisms already do to fight against ageing.

As a matter of fact, recent developments have brought us closer for materials to react and adapt to environmental stimulus, where the use of integrated circuits allows for the materials to be able to have sensing and actuating functionalities (Helou, Grossmann, Tabor, Buskohl, & Harne, 2022). This new intelligent material is comparable to the role that plays the brain in the human or animal body. The recent “engineered living material” could be very interesting if adapted to sensor and act upon failure modes that take place during the composite life cycle caused by creep.

Concrete, a composite system widely used in civil engineering, is the materials choice in several structural buildings like bridges. Engineering a bridge is not an easy task and due to its exposure to the environment it becomes even more complicated. Climate change came to intensify some factors that have an implication in the lifespan of bridges. Rapid temperature alteration, aggressive liquids and increasing rate of carbon dioxide (CO_2) are some of the current concerns to sustain this complex structural construction, especially considering that CO_2 has a chemical reaction with calcium hydroxide and hydrated calcium silicate present in the concrete (Broomfield, 2007).

Carbonation is this chemical reaction, and even though this problem assimilates more degradation of the material, ageing effects cannot be ruled out (Liisma, Sein, & Järvpõld, 2017) (Bjarnadóttir, 2022). Therefore, the ideas and mechanisms discussed for helping resisting AO effects could likewise be used here to allow bridges to maintain their durability throughout time. Moreover, carbonation is not the only effect that attacks bridges, there is also corrosion and degradation due to environmental consequences (temperature and humidity cycles).

Figure 39: Concrete bridge showing severe problems on the surface caused by carbonation and environmental factors (Hoffmann Architects, 2018)

Looking now for what was seen in natural materials, many uncertainties remain because most of the results have many variables, such as composition, texture, type of material, etc. Two of the established features in rocks that had an impact in durability was the homogeneity and the grain size, this could apply to FRPs when talking about the arrangement between matrix and the reinforcement could play an important role in durability. The term homogeneity cannot be used in composites, but it can apply to the arrangement of the fibre in the matrix, regarding the organisation of fibres.

Both the homogeneity and grain size make reference to rocks and metals respectively, which have a completely different structural organization. And even though, the structural display in composite systems is totally different and behaves in unique ways, these factors on rocks' creep resistance could be visualize in terms of the transverse organisation of the FRP. Therefore, the study of the pattern of fibres in the matrix, the shape of fibres and the size of fibres may be interesting for analysing the effects on creep resistance. In fact, this consists of exploring even more the effects of fibre pattern on composite materials.

It is important to note that even if it was said previously that fibres in general do not present viscoelastic behaviour, which does not mean that they are not relevant for creep performance of the composite system, especially because their distribution and display will have a big impact on the matrix. For that reason, when looking at increasing creep performance, fibres cannot be overlooked.

A great example of the importance of fibres design, is the case study in automotive applications where the use of flat glass fibres is compared to the conventional glass fibres with circular design. In this study, the creep performance of the flat fibres appeared to have a positive impact in the short term, while also improving fatigue and physical properties (Omnexus, 2022). This geometrical feature in the fibres has shown to have a big effect that should not be underestimated.

An investigation regarding the generalize composite design can also be done, taking into consideration that the composite material is anisotropic and all sorts of designing parameters of the fibres, transversely and longitudinally, play an important role such as orientation, shape, continuity, size, and others. In fact, with today capabilities a machine learning program can facilitate this analysis.

Designing materials is of extreme importance to achieve the increasing demands for engineering applications. However, defining a composite design does not always mean to choose the perfect combination of matrix and fibres to achieve the most suitable composite, sometimes the focus must turn to the respective structure of the material to customize properties. These materials are called metamaterials, which are described as artificially engineered advanced materials that are created to induce tailored properties in a material, as shown in Figure 40 where the metamaterial designed expresses enhanced compressibility (Chohan & Singh, 2022).

To the best of the author knowledge, there is not any new metamaterial that has been developed to increase the creep resistance. This could be a good research subject. Alternatively, instead of focusing on increasing creep behaviour the focus could go to improve elastic properties and then study the effect it has on creep rate. In metals, it was seen that increasing elastic properties could significantly enhance creep resistance.

In addition, it can be retrieved from a recent article the use of artificial intelligence to develop new designs for metamaterials according to targeted properties (Bessa, Glowacki, & Houlder, 2019). This computational data-driven method developed by Bessa et al. helps future designs to be more easily generated while taking into consideration the desired properties, the choice of base materials, manufacturing processes and length scales (Bessa, Glowacki, & Houlder, 2019).

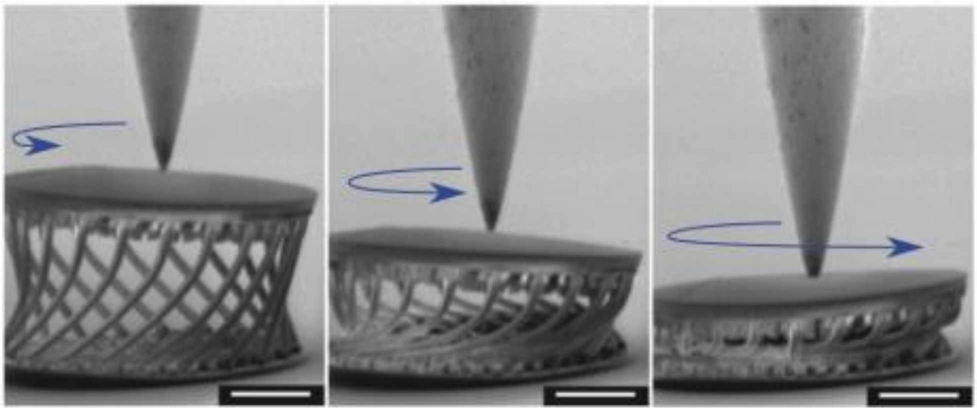

Figure 40: Example of metamaterial with super compressible characteristics (Bessa, Glowacki, & Houlder, 2019)

Currently, some solutions exist to enhance durability of materials based on biology studies. One of the studies refers to a biological coating that was analysed on marine concrete to see its influence and effect on durability (Lv, Cao, & Hu, 2021). The researchers used the *Crassostrea gigas* coating and concluded that the marine concrete highly benefits from this coating as the durability was enhanced (Lv, Cao, & Hu, 2021). This is a great example of a biomimetic use to improve composite materials by getting the inspiration from a species of oyster (*Crassostrea gigas*).

In another study, a concrete material with bio-based self-healing was made to deal with the durability problem. Here, the solution consisted in defending the matrix (the most crucial part of the composite when talking about ageing) with self-healing mechanisms, that have been investigated for a long time to reduce cracking issues. However, in this particular study microorganisms were added to the composite material through microbial-induced-calcite-precipitation (Nasser, Sorour, Saafan, & Abbas, 2022). This process consists of repairing cracked areas by using calcium carbonate to solidify in those areas (Jagadeesha Kumar, Prabhakara, & Pushpa, 2013). The biotechnological approach here described is very interesting but focuses on something different than what is pretended in this thesis, because the solution is more towards crack repair instead of ageing problems such as losing mechanical properties over time. However, the idea of inserting a microorganism in the composite's matrix looks very promising towards the implementation of some of the defensive mechanisms of animals and plants described in this work.

4. Proposal of Research Methodology

In this chapter, a research methodology to further explore more the topic of the thesis is proposed. In the previous chapters, the idea was to associate, as much as possible, the ageing of bio species and the long-term behaviour of natural materials with the creep resistance of composite systems.

The objective of the following research proposal is to respond more clearly to the thesis problem, how to enhance the durability of composites materials, while taking into consideration all the information that was gathered and discussed here. For that, two different research paths are suggested, which differ in the mechanisms studied that help confronting ageing and/or creep.

The method proposed consists of firstly investigating both paths to see if any product can be extracted from it, and then test those composite systems to see if they are able to prove the increase capacity in long-term resistance. Finally, it would be interesting to evaluate the mechanical properties of the composite system to see if any characteristics were lost in the process.

The first path is focusing specially on biomimetics and retrieving ideas from animals and plants. The ageing of these living organisms is considerable affected by environmental aspects such as ROS. However, they have different responses to it, as for animals the mechanisms seem to be more related to tissue and molecular responses, while in plants there is known physiological mechanisms that have been shown to have significant importance in the ageing process.

The mechanism mentioned is stomata, and it helps controlling the gas exchange with the plant and the exterior ecosystem. A further investigation here would consist of controlling the oxygen intake looking at the practical mechanism that was developed to control the humidity, also known as Stomatex. With the information gathered and with a deeper understanding of the technique, a possible solution for reducing oxygen exposure may be found, since this technique could be used for woven composites.

Of course, some considerations must be observed as the solution is not the same as implemented in Stomatex, since the working element here is oxygen and not water. Therefore, the differences in molecules must be considered, in terms of reactions, weight, dimension, and more important, in terms of state of matter. This last issue is crucial because water is presented in the liquid state, while the air is in a gaseous state, which means that the atoms and molecules of the components are not organized and are spread across a given volume.

In contrast to the textile feature use in Stomatex, the stomata mechanism can be managed by integrated circuits in the material where the sensor functionality would detect if the material had or not been over exposed to the ecosystem, such as radiation or AO, and would act upon that information. This could be used to protect materials in LEO that suffer from exposure to AO. One possible idea to use this advance material may be to program the integrated circuits to alternate the exposure part of the material according to damage or even time of exposure. Or, another idea would be to approximate the material behaviour to the stomata with an outer layer with holes that have the ability to open and close as programmed in the material.

Another idea that could help develop this solution might be the use of nanotechnology to prevent oxygen and air to infiltrate in the fabric. This technique is used in textile solutions to repel water and it might be possible to use it for other elements as well (Wood, 2019).

This possible solution, if feasible, would be also interesting to keep the capability of reducing humidity, as it is known to have a negative impact on the creep resistance of some polymer composites.

On the other hand, another path that seems interesting to investigate is the pattern improvement of the combination between fibres and matrix in an anisotropic composite material. This would result in an analysis of the transversal arrangement of the fibres to enhance the durability of the composite system.

Regarding the fibre arrangement, many results for creep endurance concerning fibre continuity and fibres volume were already analysed, but it would be also interesting to see the impact of different arrangements in fibres, an idea that appeared from observing the influence of homogeneity and grain size in rocks and metals, respectively. Possible factors to be explored together would be the pattern of fibres in the matrix, the shape of fibres and the size of fibres. Ideally, a heterogeneous shape and size of fibres may help reducing creep behaviour.

For this idea it would be interesting to make use of computational methods such as machine learning and digital twin where the focus is towards developing a new and innovative design for fibres that would allow an efficient layout to culminate the durability issue. This requires a lot of knowledge regarding these emerging technologies which makes it more difficult to develop the idea. The article published by Bessa et al may help to build up the machine learning program as they provide their software (Bessa, Glowacki, & Houlder, 2019).

However, for both the proposed suggestions there is a material recommendation to consider. From previous analysis stated in the bibliography, the use of a thermoset polymeric matrix would bring more resistance to creep thanks to its ability to show better reinforcement contribution for creep strain compared to thermoplastic matrices that present more sensitivity. Additionally, creep deformation is sensible to temperature especially above the glass transition temperature, and for that reason the polymeric matrix should also be selected with the highest T_g possible.

Meanwhile, the reinforcement material choice has also an impact on the performance of the composites in creep, even though most of the fibres do not present any type of viscoelastic behaviour. To choose properly the fibre material one should look at the elasticity properties as it appears to have a major role on MMC. So, the recommended fibres to use would be carbon fibre as it exhibits the highest elastic properties as shown in Table 1.

The following step is to take one of these two paths and predict their creep behaviours to analyse if there was an improvement or not. To do that, the development of the creep strength master curve should be enough, which is obtained by measuring creep resistance for different temperatures and then proceed to the conversion that will result in the final master curve of the composite or the matrix.

Finally, a general evaluation of mechanical properties should be done, using one of the four methods that were briefly mentioned in chapter 2.1.3. This would be key to understand if the material suffers any change in properties with the usage of the new solution.

5. Conclusions

Overall, this thesis explored how biomimetics can provide us with promising ideas on how to combat the ageing process of composites and increase their creep resistance. This is a rather difficult task as ageing and durability differ a lot between artificial and organic systems.

Indeed, some interesting mechanisms were discussed that could be useful in fighting against ageing in animals and plants although this do not come without a certain dose of uncertainty regarding precisely which factors are the most important to increase their lifespan. There is one theory of ageing that affects both animals and plants, which consists of reducing the oxidative stress caused by ROS.

The first promising investigation proposal centres around controlling the gas exchange between the material and the ecosystems, such as plants do with stomata. Another recommended research idea involves the arrangement of fibres and the implications it can have on creep resistance. This idea focuses on the analysis of rocks and the influence that grain size and texture homogeneity have on improving their durability.

These two proposals create opportunity to explore, in future works, this broad topic, where extensive research is paramount, and might provide extraordinary innovations in the next decade, such as the use of regeneration mechanisms in composite materials where the use of integrated circuits could enable it.

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