FACULDADE DE ENGENHARIA DA UNIVERSIDADE DO PORTO

Cork to Enhance Damage Tolerance in Composite Systems

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Integrated Masters in Mechanical Engineering

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Abstract

Composite materials are nowadays extensively used, and their development is being studied to keep constantly improving the knowledge and optimizing their capabilities. One of the main concerns with their use is the impact behavior and in particular, their damage tolerance. Since it has been proven that adding a tough material as interlayer can be a solution to tackle this problem, this study aimed to understand how can cork improve the damage tolerance in composite systems. Due to cork's properties, the fact that it is a natural material, and its importance to the portuguese economy, it comes as an interesting material to be studied to enhance the damage tolerance in composite systems.

This experimental work consisted in producing ten different laminates, with the exact same carbon-fibre prepreg and the same stacking sequence, only changing the interlayer material. As interlayer material, it was chosen to use cork films, Kraton™ and expanded cork granules with different thicknesses or concentrations and a reference laminate without any interlayer material. These laminates were cut into different specimens and subjected to tensile, impact and tensile after impact tests to characterize the material, understand their impact behaviour and assess their residual properties after being impacted with low energy levels.

The results obtained were not as concrete as it was expected, so it was not possible to take a conclusion of which cork format better enhances the damage tolerance, and which conditions are they best suited to be applied to do it (thickness or concentration). According with the results, adding cork films as interlayer material, highly improves the impact behaviour of the laminate, but on the other hand, their mechanical properties become highly compromised. In some particular situations, the thinner cork film used also managed to show some good results against the reference laminate in the tensile after impact test. Regarding expanded cork granules, their results in almost all the tests were not exactly consistent, but for some specific situations of concentrations and/or tests, they managed to show better results than the reference material. It is believed that this discrepancy of results is due to the spreading technique used to apply the expanded cork granules, that didn't created an homogeneous layer, but resulted in some highly concetrated areas of the laminate and others with lower concentrarion.

Keywords: Laminates, Composite Systems, Cork, Mechanical Properties, Impact Behaviour; Carbon-Fibre; Damage Tolerance

Resumo

Os materiais compósitos são actualmente extensivamente usados e o seu desenvolvimento está constantemente a ser estudado para que o conhecimento sobre os mesmos possa ser melhorado e assim, optimizar as suas capacidades. Uma das maiores preocupações associadas ao uso de compósitos é o comportamento quando são submetidos a impactos, e em particular, a sua tolerância ao dano. Uma vez que foi provado que a adição de materiais tenazes ao laminado como *interlayer* poderá ser uma solução para combater este problema, este estudo visa compreender como é que a cortiça pode melhorar a tolerância ao dano em sistemas compósitos. A cortiça, devido às suas propriedades, o facto de ser um material natural e importante para a economia portuguesa, torna-se um material interessante para ser estudado como agente de melhoramento da tolerância ao dano em sistemas compósitos.

Este trabalho experimental consistiu em produzir dez laminados diferentes, com exatamente o mesmo pré-impregnado de fibra de carbono e a mesma sequência de empilhamento, mudando apenas o material de *interlayer*. Estes laminados foram cortados em diferentes provetes e submetidos a testes de tração, impacto e tração após impacto com o objectivo de caracterizar o material, compreender o seu comportamento ao impacto e avaliar quais as suas propriedades residuais depois de terem sido impactados com baixos níveis de energia.

Os resultados obtidos não foram tão concretos como era esperado, então não foi possível tirar uma conclusão de qual o melhor formato de cortiça para melhorar a tolerância ao dano, e quais as condições óptimas para a sua aplicação (espessura ou concentração). De acordo com os resultados, a adição de filmes de cortiça como material intercamada, melhora bastante o comportamento ao impacto do laminado, mas por outro lado, as suas propriedades mecânicas ficam altamente comprometidas. Em alguns casos particulares, o filme mais fino de cortiça usado foi capaz de mostrar bons resultados em comparação com o laminado de referência nos ensaios de tração após impacto. Relativamente aos grânulos de cortiça expandida, os resultados em praticamente todos os ensaios não foram consistentes, mas em algumas situações específicas de concentração e/ou ensaios, foram capazes de mostrar melhores resultados que o material de referência. Acredita-se que esta discrepância de resultados seja devida à técnica de espalhamento usada para depositar os grânulos de cortiça expandida, que não permitiu a criação de uma camada homogénea, mas resultou em algumas áreas altamente concentradas do laminado e outras com menor concentração.

Palavras-chave: Laminados, Sistemas Compósitos, Cortiça, Propriedades Mecânicas, Comportamento ao Impacto; Fibra de Carbono; Tolerância ao Dano

Acknowledgements

The ending of this dissertation project is not just the ending of a 5 months work, but it is also the culmination of a five years experience in this faculty, and I would like to share my deepest thanks and acknowledgements for all these people and entities.
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This dissertation was also possible due to the contribution of Prof. Dr. Pedro Camacho that kindly shared the prepreg that was used, to AISOL (Amorim Isolamentos, José Andrade) for providing the expanded cork granules and to ACC (Amorim Cork Composites, Lino Rocha) for offering the cork films.
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João Camacho

"Long you live and high you fly
And smiles you'll give and tears you'll cry
And all you touch and all you see
Is all your life will ever be."

Pink Floyd

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Symbols and Abbreviations

ASTM	American Society for Testing and Materials
BVID	Barely Visible Impact Damage
CAI	Compression After Impact
DEM	Departamento de Engenharia Mecânica
FEUP	Faculdade de Engenharia da Universidade do Porto
ISO	International Organization for Standardization
INEGI	Instituto de Ciência e Inovação em Engenharia Mecânica e Engenharia Industrial
LVI	Low Velocity Impact
PMI	Polymethacrylimide
RTM	Resin Transfer Molding
TAI	Tensile After Impact

b	Specimen width [m]
h	Specimen thickness [m
σ	Stress [Pa]
ε	Strain [%]
E	Young's Modulus [Pa]
E*	Reduction of Young's Modulus after impact [%]
Ea	Absorbed Energy [J]
L_0	Distance between the grips of the strain gauge [m]
ΔL_0	Distance increase [m]
F	Applied Force [N]
γ	Superficial tension [Pa]
θ	Contact angle [rad]g
UTS	Ultimate Tensile Strength [Pa]
UTS*	Reduction of Ultimate Tensile Strength after impact [%]

Chapter 1

Introduction

Nowadays, technological knowledge is evolving at a high speed and thus, companies are starting to implement the new findings in their products. One of the biggest impacts that technology had and still has, is what we know about materials. It was natural for human kind to craft materials that can combine low density and good mechanical properties, without neglecting economical reasons.

Composite materials can be defined as a material system made from two or more materials (carbon fibre and epoxy resin, for instance), ending up having a material with better properties than the single materials.

When these materials started to blossom, its usage was mainly in highly demanding applications (aerospace & aeronautics, for instance), such that the final cost was not a criteria but, as technology and manufacturing processes started to evolve, the prices decreased and now, it's possible to see composite systems in more common applications, starting to replace traditional engineering materials, such as steel or aluminium.

On the other hand, composites systems are still expensive (when compared with traditional materials), the manufacturing technology is still dependent, on some extent, on skilled hand labor with limited automation and standardization [10] and, due to their usual anisotropy and the different elements, are still very complex materials, specially on what damage is concerned.

1.1 Historical Development

Human kind, as a specie, is tightly related with the materials that was used. It first started with using stones (ceramics) to create tools and weapons with natural polymers and composites (wood). The following period, was marked by the usage of metals (gold, copper, bronze and iron), and in the last century steel and aluminum had a dominant role in the development of the human civilization.

On today's world, polymers, ceramics and composites are again reclaiming their importance but, before men used the natural form of these materials, now men are engineering their own materials[10].

The concept of fibrous reinforcements dates back thousands years ago, but it was in the nineteenth century that iron rods were used to reinforce masonry, the first step to what now is called steel-reinforced concrete.

The use of reinforced plastics in aircrafts and electrical components started in the forties and in 1942, the first fibreglass boat was made. The usage of advanced composites in aircraft components started in 1968 and in the late late 1970, the applications of composites expanded widely to other industries such as marine, automotive, sports and biomedical.

Composites technology suffered a rapid development in the last four decades, using the driving force of the aerospace industry that demanded weight saving and great performance. Today, there is also the need for quality assurance, reproducibility, and predictability of behaviour over the lifetime of the structure.

New advancements are still happening, such as new types of carbon fibres with higher strength and ultimate strain, thermoplastic matrices to be used under certain conditions, smart composites, the utilization of nanocomposites and multiscale hybrid composites with multifunctional characteristics. The development of composites is also taking place in the manufacturing processes, due to their influence in the final properties and quality assurance. Although the technology is still in development, it has now reached a stage of maturity[10].

1.2 Applications

As it was aforementioned, the usage of composites started mainly in the aerospace and aeronautics industry, not just because of the great performance with low weight, but also the possibility to design a large variety of materials with different property combinations.

Today, it is possible to find composites in a broad variety of industries such as sports and leisure (helmets, surf boards, etc.), automotive industry, military, marine and bioengineering. A great example of current usage of composites is the Boeing 777 that has this type of materials in its fairing, floor beams, wind trailing edge surfaces and the empennage (Fig. 1.1).

1.3 Importance of Damage Tolerance in Composite Systems

The main advantages of composite systems were presented, nevertheless composite materials are usually fragile and anisotropic, making them sensible to stress intensity factors, such as low velocity impact [11] created by accidental loads during production, service or maintenance, for instance. The damage suffered by a composite material is responsible for a change in the internal tension distribution, and thus reducing the load capacity of the material.

The low velocity impact is by far, the most dangerous damage that can occur, due to the extensive damaged areas with delaminations and/or matrix rupture. It can also happen that the phenomenon is not assessed when it is visually inspected. This reason states the importance of studying damage, and impact situations in particular. Impact resistance is characterized by the

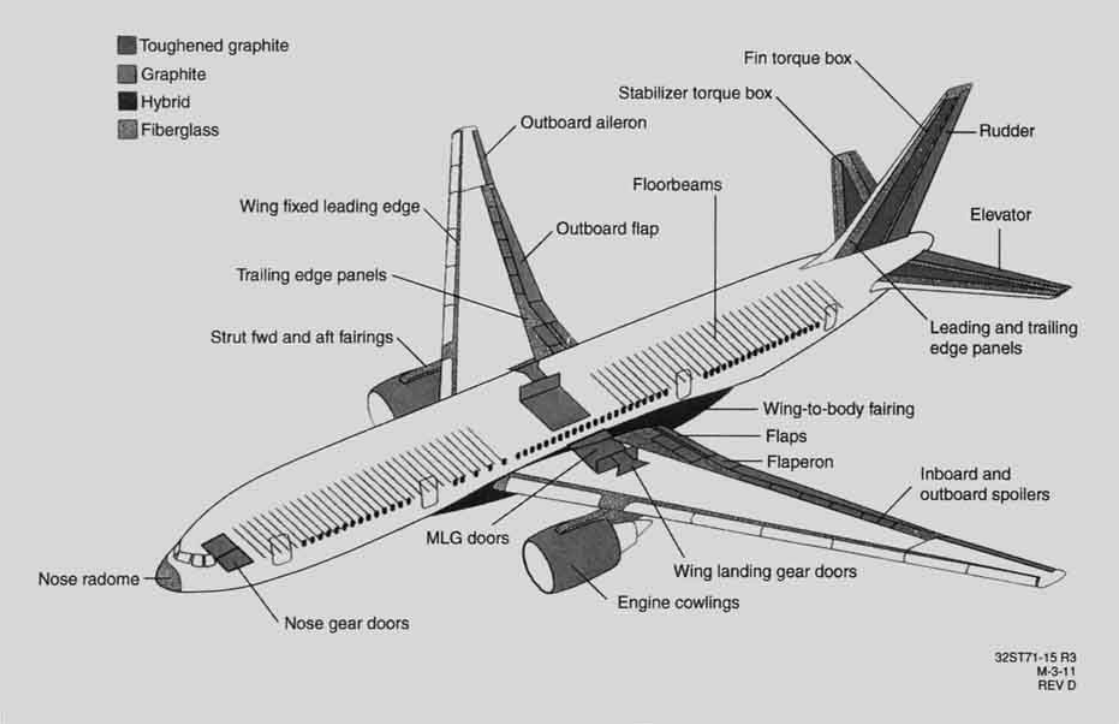


Figure 1.1: Usage of composite systems in Boeing 777 [10]

capacity of the material to not suffer permanent damage, and damage tolerance is the capacity of a damaged composite to maintain its original strength and stiffness.

1.4 Cork to enhance Damage Tolerance in Composite Systems

Since damage tolerance is such an important topic in composite systems, different solutions and materials have been studied to improve this problem, and one of them is cork, which is what this study is all about. Cork plays an important role in portuguese economy and in the environment. On today's world, there is a conscience for the environmental problems and scarcity of resources.

Cork is a material that can solve both of these problems, since it's a natural material and it comes from the cork oak, by which the cork is removed from the tree every 10 years, on average, without causing any big harm. Concerning the properties, cork presents itself with low density, high compressibility and flexibility, good chemical stability, good thermal and acoustical insulation and also an interesting energy absorption capacity, making it an interesting solution to improve damage tolerance in composite systems.

1.5 Goals

This dissertation aims to perform an experimental study that keeps the work that has been done so far about the usage of cork to enhance the damage tolerance in composite systems. In order to do it, it was decided to produce different laminates, keeping the same carbon-fibre prepreg and

keeping the same stacking sequence, variating only two interlayers of the laminate with different solutions, to later compare the results. It is intended to use as interlayer two different cork formats: cork films of different thicknesses and expanded cork granules with different concentrations. In order to assess if these additions to the laminate cause an improvement of the damage tolerance, it was also decided to use Kraton™ granules (also with different concentrations), a commercial available solution, and a reference material, without any added material as interlayer.

1.6 Structure and Summary of the Chapters

- Chapter 1 - This chapter presents the motivation to study this possible solution, by including a brief explanation of this dissertation's theme and the context of this study. It also includes the exposition of the goals and purposes of this project.
- Chapter 2 - Chapter two includes the a literature review of the fundamental theoretical concepts that are important under the context of composite systems, low velocity impacts and damage tolerance. It also exposes the state of art in this field of expertise, by presenting what has been done so far under this topic. These chapter also looks to answer the decisions and choices that were made during the laminates' design and production, and experimental tests.
- Chapter 3 - Here there is a description of the whole experimental and laboratorial procedure, mentioning how was the fabrication of the laminates (and specimens), and the mechanical tests that were performed.
- Chapter 4 - Chapter four presents all the results obtained during the mechanical tests and analysis of the results.
- Chapter 5 - The fifth and last chapter aims to answer the questions proposed, through a conclusion, and presents some future work suggestions.

Chapter 2

Literature Review

2.1 Composite Materials

A composite material is a material that consist of two or more combined constituents which are combined at macroscopic level and are not soluble in each other. Its properties highly depend on the properties of its constituent phases. One of the phase is called reinforcement, which is harder and stronger, and the other phase is not so mechanically resistant, but also provides interesting properties to the whole material, called matrix. Both constituents are usually arranged to have one or more dispersed phases, reinforcements, in a continuous one, the matrix.

The properties of a composite are a function of its constituents, their relative amounts and the interaction between them. The geometry of the dispersed phase is one of the most relevant factors to take into account, such as the shape, size, distribution and orientation[8]. Besides all of this factors, they can usually be easily controlled and changed to get the desired properties.

Using composite materials, gives the engineers a lot of advantages, since it's possible to craft their own material according with the applications, which it not so easily done with other traditional materials such as steel, aluminium or polymers. These traditional materials, usually present uniform mechanical properties in all directions since they are isotropic and homogeneous, which doesn't happen in composites because, most of the times, they are anisotropic or orthotropic.

2.1.1 Constituinte Materials

2.1.1.1 Reinforcement

As the name says, the reinforcement is the resistance phase of the composite material and thus, it has the highest relevance for the final product. Although it depends on the final application, it is desirable that the reinforcement has high stiffness, high strength and the lowest density possible[10].

A composite can be reinforced by particles or by fibres. The particle reinforcement provides a higher stiffness value to the matrix, but it doesn't improve much the global mechanical resistance properties. The reinforcement usually comes in the shape of fibres, that can have different length and/or orientation, influencing the degrees of anisotropy. Due to the small section area, the fibres

cannot be used alone, so they usually are immersed in a polymeric matrix, which helps to transfer the load between the fibres and protect them from the outside environment.

Fibre reinforced materials, comparatively to metals, have better specific resistance and modulus, but the absolute properties have lower values.

The most used reinforcement materials are the glass fibre, boron, carbon and aramid (Kevlar®). Most of the fibrs have a linear behaviour as it is shown in Figure 2.1 and 2.2.

An introduction on carbon fibre is going to be made, since it's the material used during this study.

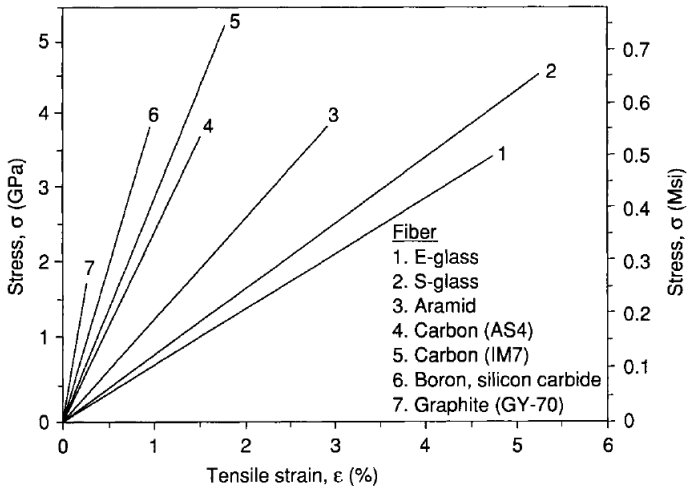


Figure 2.1: Stress-strain curves of typical reinforcing fibres[10]

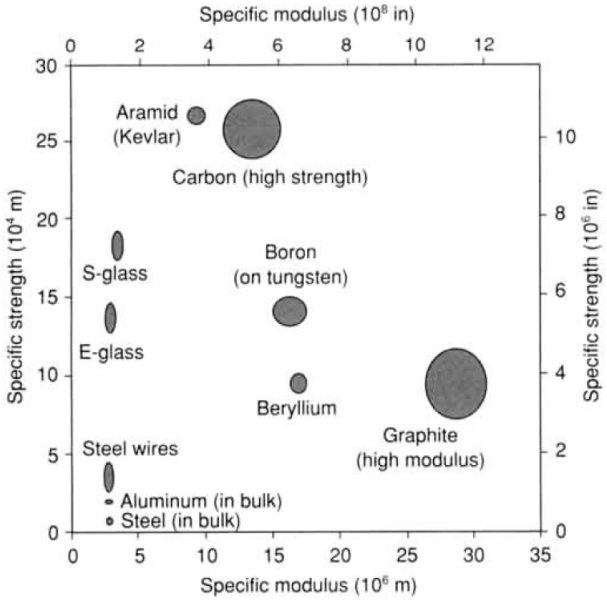


Figure 2.2: Performance map of fibres used in structural composites[10]

Carbon Fibre

As it was mentioned before, the most used fibres are glass fibre, boron, carbon and aramid (Kevlar®), but carbon is the most commonly used reinforcement in advanced (i.e., non fibreglass) polymer matrix composites and it comes with different properties depending on the manufacturing process[10].

Although the name of the reinforcement is carbon fibre, carbon is an element. The stable form of crystalline carbon at ambient conditions is graphite. Carbon fibres are not totally crystalline, but have both crystalline (graphitic) and noncrystalline regions, these last ones don't have a three-dimensional arrangement of hexagonal carbon network, as it happens with graphite.

Fibres have a diameter that normally range between 4 and 10 μm, and usually they are coated with an epoxy protection that can also improve the adhesion the the polymer matrix. The manufacturing process that produces the fibres is relatively complex, but it has been developing and now it's relatively inexpensive and cost effective[8].

Carbon fibres have the highest specific modulus and specific strength of all reinforcing fibre materials and they are able to retain their values at high temperatures, but they can suffer from high-temperature oxidation. At room temperature, this material is not affected by moisture or by a wide variety of solvents, acids and bases.

This type of fibre are now being extensively used in sports and recreational equipment, such as fishing rods, golf clubs, filament-wound rocket motor cases, pressure vessels and aircraft and helicopters structural components.

2.1.1.2 Matrix

Matrix is the other phase of a composite material, and the four types of matrices used are polymeric, metallic, ceramic and carbon, but the more extensively used are the polymeric ones, that can be thermoplastics or thermoset. Polymeric matrices are also easier to manufacture due to the relatively low temperature to process.

The purpose of the matrix in the system is mainly to bind the fibres together, acting as a medium by which the external stresses are transmitted and distributed to the fibres and thus, only a small percentage of the load is applied on the matrix phase. Matrix also serves the purpose of protecting the fibres from surface damage, such as mechanical abrasion or chemical reactions with the environment, that can induce surface flaws and from there, form cracks, leading to a failure of the material at low stress interactions. Due to the matrix material's softness and plasticity, the matrix prevents the propagation of brittle crack from fibre to fibre, that otherwise would induce a catastrophic failure. In order for this to happen, it is important that the adhesive bonding forces between fibre and matrix are high[8].

For the experimental work, it was used an epoxy resin, a polymer matrix. Some details about this type of matrix is presented in this document.

Polymer Matrix

As it was mentioned before, the most common class of material used as matrix is polymer, which

can be thermoplastic or thermoset. The biggest difference between them is that thermoset do not melt upon reheating, and at high temperatures, it starts decomposing. Thermosets are the most predominant type of matrix system, and they undergo polymerization and cross-linking during curing with a hardening agent and heating. Thermoplastics are fully polymerized polymers and can be altered physically by softening or melting. Still regarding thermoplastics, they can be processed in less time and have a higher glass transition, but have a high temperature-dependent behaviour. They are also much less sensitive to moisture absorption and exhibit higher fracture toughness, but a short fatigue life.

The most common polymers used in composite matrices are unsaturated polyester, epoxies, polyimides, phenolic and vinylesters (thermsets) and polypropylene (PP), polyphenylene sulfide (PPS), polysulfone, poly-ether-ether-ketone (PEEK) (thermoplastics).

Thermoset resins offer some advantages over thermoplastic ones, such as better fibre wetting, which decreases the porosity level, and they also require lower temperature and pressure to manufacture, which may lead to a cheaper and energy saving process and they are easier to machine. On the other hand, thermoset resins require a longer curing time, and thus a smaller production rate and it was mentioned before, since they cannot be reheated it's harder to recycle.

When choosing a resin to use as a matrix, the characteristics to take into consideration are: adhesion capacity to be able to use the full potential of the resin in terms of mechanical properties, although this characteristic also depends on the fibre and surface treatment, mechanical properties, stress crack resistance, fatigue resistance and degradation due to the contact with water or other substances.

The curing process of polymers begins with the growth and ramification of molecular chains, which increases the molecular weight of the material. During this process, the resin might contract due to the rearrangement and re-orientation of the resin molecules in the liquid and semi-liquid phase. As an example, polyester and vinyl ester can contract about 8%, but epoxy resin, which goes through a low molecular rearrangement and not a lot of volatile products, contracts about 2%. This low contraction results in better mechanical properties, since during the contraction, there are some internal tensions that can weaken the material[11].

Polymer curing happens through chemical reactions, with the help of some additives such as: amine, anhydride, carboxylic acids, phenols and alcohols. The velocity of the process can be easily controlled through the adquade selection of curing agents and catalysts.

Epoxy Resin

Epoxy resins are the most extensively used in advanced composites. Some of the advantages were already mentioned, when talked about thermoset resins. They consist on the absence of subproducts formed during the cure, low contraction while and after curing, resistance to solvents and other chemical products, and fatigue and creep resistance. They also have a good performance both at ambient and high temperatures: usually epoxy resins can be used until 80-120ºC, some of them can even be used at 240ºC. Due to the existence of ether and hydroxyl groups, they have even better impregnation capacity, when comparing with other thermoset resins. Although epoxy

resin has numerous advantages, it is more expensive when compared to other polymers, that's why it's a material used more often in demanding applications. Besides, epoxy resin has also a brittle behaviour, but there are some methods that can improve it's ductility, such as adding thermoplastic in the resin.

2.1.2 Classification

Composite systems can be classified into three main divisions: particle-reinforced composite, fibre-reinforced composite and structural composite (Fig. 2.3), subdividing into, at least, two subdivisions each[8].

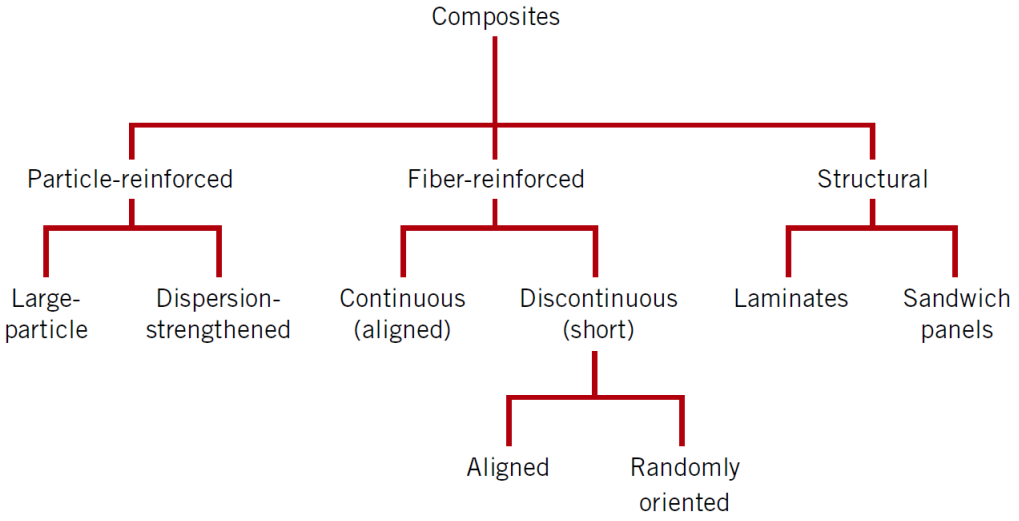


Figure 2.3: Classification scheme for the various composite types [8]

2.1.2.1 Particle-Reinforced Composite

In this classification, particles are classified as a non-fibre material of small dimensions, and can be subdivided into large-particle and dispersion-strengthened composites. The distinction is made according with the reinforcement or strengthening mechanism. Dispersion-strengthened particles are normally much smaller, with diameters ranging between 10 and 100 nm. When using large particles reinforcement, the interaction particle-matrix cannot be treated on the atomic or molecular level, but it is rather used continuum mechanics. Since the reinforcement is a particle, the discontinuous phase is equiaxial, meaning that dimensions are approximately the same in all directions.

Globally, particle reinforcement can improve the stiffness of the polymeric matrix, but this reinforcement does not have a considerable contribution for the improvement of the tensile strength, especially because high stiffness elements in a brittle matrix can lead to lot of stress concentration zones.

The improvement of mechanical behaviour depends on strong bonding at the matrix-particle interface. The interactions that lead to strengthening, occur on the atomic or molecular level - similar to that for precipitation hardening, as it happens with the metals, for instance, where the matrix bears the major portion of the load and the small dispersed particles hinder or impede the motion of dislocations.

2.1.2.2 Fibre-Reinforced Composite

Fibre-Reinforced composites are considered the most important composites[8]. This reinforcement is often used when the goal is to produce materials with high strength and/or stiffness on a weight basis.

They can be subdivided into continuous fibres or short fibres. In the case of short fibres, the ratio length/diameter is between 5 and 1000, with diameters of about 0.02 to 100 μm. These fibres are too short to produce a significant improvement in strength, mainly because the load transmission effect by the matrix is more relevant, when compared with continuous fibre reinforcement. Regarding continuous fibres, they have a high length/diameter ratio, and the diameter can range from 3 to 200 μm, depending on the fibre type. The fibres used in this reinforcement type are stiffer and stronger, when compared with the bulk material. The load transfer happens according to its orientation and, in this situation, the purpose of the matrix becomes mainly to keep the fibres together.

These reinforcement types can be be formed by unidirectional or multidirectional layers. Using unidirectional layers gives the composite high tensile modulus in the direction of the fibres, but regarding load in the perpendicular direction, it has a weak load capacity. When several layers are stacked, they are called laminate and when these layers are from different materials, it is called a hybrid laminate.

2.1.2.3 Structural Composites

The most common structural composites are the laminate composite and the sandwich panels. These structural composites are normally composed of both homogeneous and composite materials, and the properties of the final composite depend not only on the properties of the constituent materials but also on the geometrical design of the various elements.

Laminate Composites

Laminate composites are composed of two-dimensional sheets or panels stacked together that can have a prefered high-strength direction. These sheets or panels are called laminae (or plies, or layers), can have various thicknesses and consist of different materials. With all of these variables, the designers and engineers have a huge flexibility to craft their own material according with the final application, being possible to obtain the desired stiffness and thickness due to its anisotropy.

The layers are stacked and cemented together and the orientation of the high-strength direction varies with each successive layer. Usually, the layers are bonded together by the same material of the matrix, making it unnecessary to add more materials[25][10].

Laminate composites have relatively high strength in a number of directions in the two-dimensional plane, however, the strength in any given direction is lower than it would be if all the fibres were oriented in that direction. The orientation of a ply is given by the angle between the reference x-axis and the major principal material axis (fibre orientation or wrap direction) of the ply measured in a counterclockwise direction on the x-y plane (Fig. 2.4).

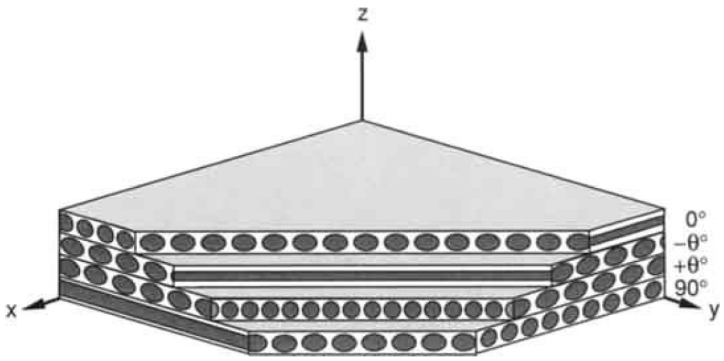


Figure 2.4: Multidirectional laminate and reference coordinate system[10]

The mechanical behaviour of a laminate is different from the behaviour of a single layer, but the behaviour of the whole laminate depends on the behaviour of each layer and the stacking sequence (Fig. 2.5).

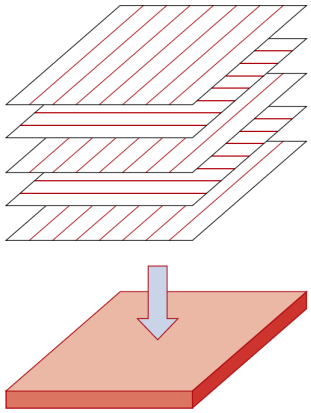


Figure 2.5: Stacking of successive oriented fibre–reinforced layers for a laminar composite[8]

Laminate composites have a designation that indicates in a specific manner the number, type, orientation and stacking sequence of the plies. The configuration of the laminate indicating its ply composition is called the layup. The configuration indicating the exact location or sequence of the various plies, is called the stacking sequence. Some examples from Daniel Ishay[10] can be consulted in Figure 2.6[10].

Unidirectional six-ply:	$[0/0/0/0/0/0] = [0_6]$
Crossply symmetric:	$[0/90/90/0] = [0/90]_s$
	$[0/90/0/90/90/0/90/0] = [0/90]_{2s}$
	$[0/90/0] = [0/\overline{90}]_s$
Angle-ply symmetric:	$[+45/-45/-45/+45] = [\pm 45]_s$
	$[30/-30/30/-30/-30/30-30/30] = [\pm 30]_{2s}$
Angle-ply asymmetric:	$[30/-30/30/-30/30/-30/30/-30] = [\pm 30]_4$
Multidirectional:	$[0/45/-45/-45/45/0] = [0/\pm 45]_s$
	$[0/0/45/-45/0/0/0/0/-45/45/0/0] = [0_2/\pm 45/0_2]_s$
	$[0/15/-15/15/-15/0] = [0/\pm 15/\pm 15/0]_T = [0/(\pm 15)_2/0]_T$
Hybrid:	$[0^K/0^K/45^C/-45^C/90^G/-45^C/45^C/0^K/0^K]_T = [0_2^K/\pm 45^C/\overline{90}^G]_s$

where subscripts and symbols signify the following:

number subscript = multiple of plies or group of plies

s = symmetric sequence

T = total number of plies

$^-$ (overbar) = laminate is symmetric about the midplane of the ply

In the case of the hybrid laminate, superscripts K, C, and G denote Kevlar®(aramid), carbon (graphite), and glass fibers, respectively.

Figure 2.6: Designation of composite laminates[8]

Sandwich Composites

Sandwich composites are designed to provide solutions that are lightweight (usually come in the form of a beam or a panel) and have a relatively high stiffness and strength. They are composed of two outer sheets or faces, separated by a thicker core that is adhesively bonded to the sheets (Fig. 2.7).

The outer sheets, or faces are made of a relatively stiff and strong material, usually aluminum alloys, fibre-reinforced polymers, titanium, steel or plywood. These components are the responsibles to give to the structure a high stiffness and strength to the structure and must be thick enough to withstand tensile and compressive stresses from the loading.

Regarding the core, it is typically made of one of these threes material categories: rigid polymeric foams (phenolic, epoxy or polyurethane), wood (balsa wood) and honeycombs (aluminium alloy or aramid polymer). The main functions of the core are to provide continuous support for the

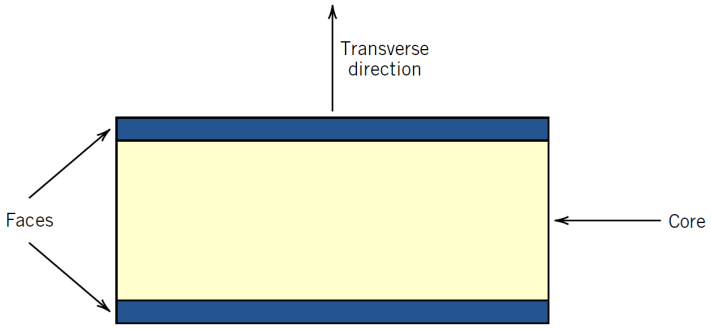


Figure 2.7: Scheme of a cross section of a sandwich panel[8]

faces, withstand transverse shear stresses and provide high shear stiffness to resist the buckling of the panel (so it should be thick enough).

As it was aforementioned, honeycomb cores are widely used in sandwich composites. They are structures-thin foils that have been formed into interlocking hexagonal cells, with axis orientation perpendicular to the faces planes (Fig. 2.8). The strength and stiffness depend on the cell size, cell wall thickness and the material used as honeycomb.

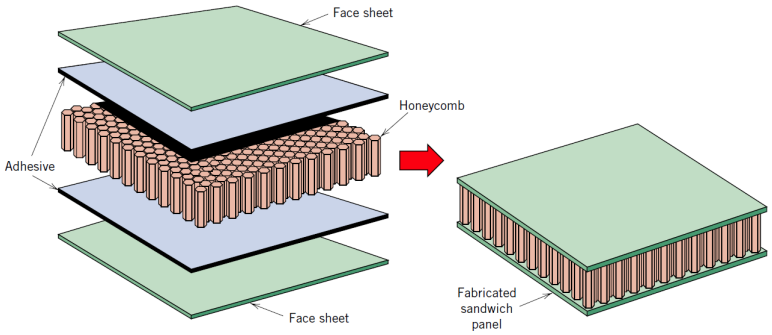


Figure 2.8: Construction of a composite sandwich panel with a honeycomb core[8]

2.1.3 Hybrid Composites

This relatively new type of composite is obtained by using two or more different types of fibres in a single matrix and thus, it is possible to obtain better combination of properties than composites containing only a single fibre type. This system can be combined in a wide variety of ways which will affect the overall properties.

Composite laminates that contain plies of two or more different material types are also called hybrid composites, more specifically interply hybrid composites. An example of such combination might be a composite laminate that is made up of unidirectional glass/epoxy, carbon/epoxy and aramid/epoxy stacked together in a specific sequence[10].

The overall behaviour of hybrid composites comes from a weighted sum of the advantages and disadvantages of each component.

It is also possible to incorporate alternative materials such as industrial waste or even materials that come directly from nature such as natural fibres or natural resins and thus contribute to world sustainability.

2.1.4 Manufacturing Processes

The way a composite part is processed and manufactured, highly influences the final properties, and thus it's one of the most important steps in the application of composite materials. The manufacturing process should be selected concurrently with material selection and structural design and, in the case of composite systems, it is governed by the matrix used[10].

Nowadays, there is still a barrier to the more extensive use of composite materials, and most of it is due to the high cost, which can be attributed to lack of cost-effective fabrication methods and the necessity for post process inspection to ensure quality of the material and part. With this being said, the final product must meet some general requirements, namely the fact that it must be free of defects (voids, cracks, fibre waviness), have uniform properties, be fully cured (having expected properties, for example: stiffness, strength, fatigue endurance) and reproducibility[10].

The finished product should also fulfil some specific goals of manufacturing, for example: control of reinforcement location/orientation, ply thickness, fibre volume ratio, voids, residual stresses and final dimensions. Regarding the process itself, the temperature must not exceed preset values, temperature distribution must be reasonably uniform throughout the part and an uniform cure must be accomplished in the shortest possible time.

There are a huge number of fabrication methods that are used today. Some examples of the ones more often used are: autoclave, vacuum bag and compression molding, filament winding, injection molding, pultrusion and resin transfer molding (RTM), but in this chapter, only the prepreg production process is going to be explained, since it's the one used during the experiments. In all of these processes, there is a set of limitations, more specifically on the production rate, size, geometrical shape allowed, structural quality, homogeneity of the part, automatization and cost.

2.1.4.1 Prepreg Production Process

A prepreg is the composite industry term for continuous-fibre reinforcement pre impregnated with a polymer resin that is only partially cured, made to be ready for fabrication of composites. This material is delivered in the form of a tape that consists of layer of parallel or woven fibres that were already, as mentioned before, partially cured to be molded and fully cured by the industry without having to add any resin. There are prepregs with different reinforcements, but the more extensively used are the common fibres: carbon, glass and aramid, and both thermoplastic and thermoset resins are used. These prepreg tapes are made to meet certain specifications such as fibre volume ratio, ply thickness, and degree of partial cure (B-staging)[8].

The prepreg manufacturing process for thermoset polymers is represented in figure 2.9. It begins by collimating a series of spool-wound continuous-fibre tows, which are then pressed between sheets of release and carrier paper, by the mean of heated rollers in a process called calendering. The release paper sheets have already been coated with a thin film of heated resin of low viscosity to impregnate the fibres. A doctor blade guarantees a uniform thickness and width of the resin film by spreading the resin on the fibres. As the impregnated tape is spooled, the release paper sheet is removed[8].

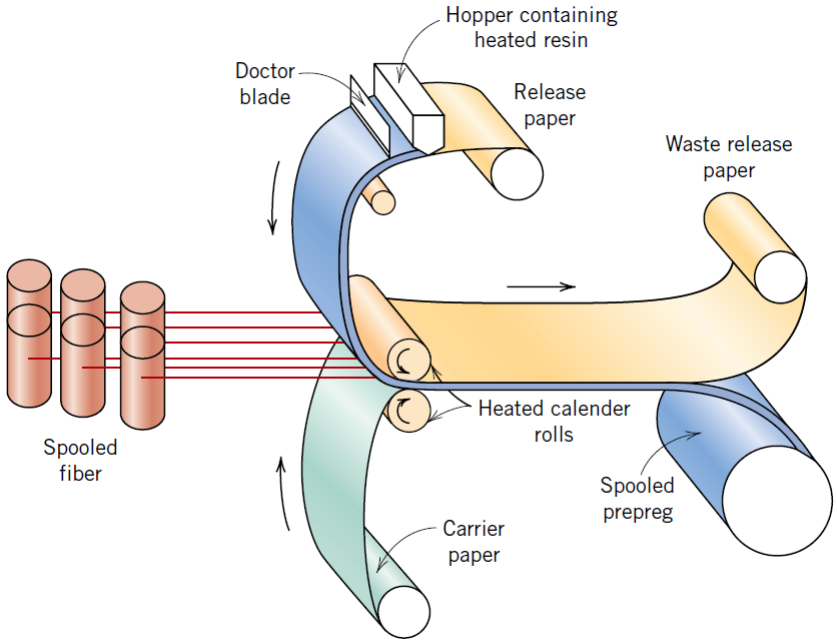


Figure 2.9: Construction of a composite sandwich panel with a honeycomb core[8]

The industry fabricates the composite firstly with the lay-up, laying the prepreg on a surface with the specific number of plies to provide the desired thickness (after the removal from the carrier backing paper). The stacking may be unidirectional, but more often, the fibre orientation is alternated to produce a cross-ply or angle-ply laminate. This lay-up can be made entirely by hand (hand lay-up), where the operators cut the length of the tape and position them in the desired orientation, but the lay-up can also be machine cut and then hand laid. The fabrication cost can be reduced by automation of the prepreg lay-up and by using other manufacturing procedures, eliminating the need for hand labor and thus, making it cost effective. After the lay-up, the prepreg goes through the application of heat and pressure simultaneously[8].

The prepreg is characterized by the resin content, which is usually about 32-45% by weight, tack (self-adhesive), drape ability (ability to conform to shapes), shelf life, out time and gel time[10].

Since the material is pre impregnated, it must be kept refrigerated at approximately -18ºC until the final use. Even when using it, the room temperature must be minimized since, for instance,

thermoset matrix undergoes curing reactions at room temperature. If the material is properly handled and stored, thermoset prepregs have a lifetime of at least six months or even longer.

2.1.5 Applications

Nowadays composite systems are used in a wide variety of industries for a wide variety of purposes. Some of the industries where composites are more extensively used are going to be mentioned.

Aeronautical and Aerospace Industry

These industries were the booster for the study and investigation of this type of materials due to the constand demand for structures with low weight, great performance and that the final price, although has some importance, is not the main factor. It all started in the decade of 1970 in military aircrafts for secondary structural components, and latter on commercial aircrafts.

Nowadays, the usage of composite systems in aircrafts is well established in structural components. As an example, A380, from Airbus, has about 25% of its weight in composites (figure 2.10).

The main advantages, besides the decrease of weight, is the reduction of mechanical joints (allowing a decrease of manufacturing and assembly costs and, once again, a decrease of the structure's weight) and fatigue and corrosion resistance.

Constructions and Infrastructures

It is a field of application where there is constant investigation, since the replacement of steel and concrete by composites allow a decrease of weight and an increase of corrosion resistance, resulting in an increase of structure's life and a lower maintenance cost.

It is also possible to see composites being used in buildings isolation, doors, windows and floors, since it is possible to create composites that slow the fire progression.

Automobile Industry

The reduction of weight is one of the main motivating factors, making the automobile industry one of the main interested industries.

Fibreglass presents the bigger relevance in this industry, since carbon fibre is still quite expensive. The high production rate demanded by this industry, also limits the usage of epoxy resins due to the higher curing time.

In motor sports, the priority is high speed, being the final cost a secondary factor and, in this situations, carbon fibre and epoxy resins are more extensively used in the body, chassis, interior and suspension.

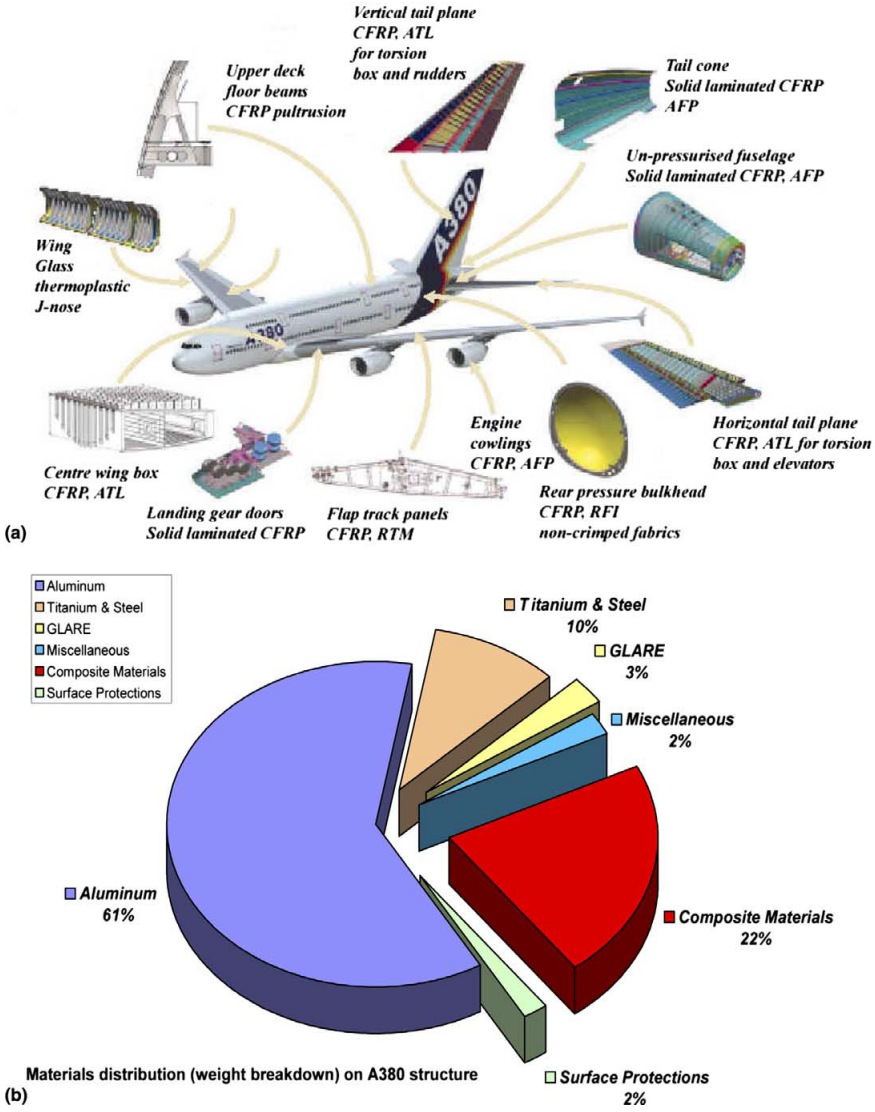


Figure 2.10: Usage of composite materials in A380 a) Components with carbon fibre b) Materials distribution (weight breakdown) on A380 structure[27]

Sports Industry

Once again, low weight is one of the main motivation factors. Also corrosion resistance, good mechanical properties and ease of conformation make composite materials an interesting solution for the sports industry.

It is possible to see this type of materials in bikes, rackets, helmets and golf clubs, for instance.

Naval Industry

Ship hulls, decks and interiors. These are the main parts where composites are used in this industry. About 90% of recreational crafts have fibreglass reinforced unsaturated polyester.

In competition boats, carbon fibre is prefered to be used due to lower weight and better mechanical properties.

Biomedical Applications

Composites are also used in applications that include the diagnostic, treatment of diseases and its prevention. Some concrete examples are: implants, surgical and diagnostic devices, pacemakers, wheelchairs, mobility support equipments, packaging of certain drugs and instrumentation for chemical analysis.

Implants are one of the most challenging application, since it requires that the material has to be biocompatible, resistant to corrosion and fatigue and has to be able to maintain its properties over a long period of time. Usually materials such as carbon matrix and biocompatible polymeric matrices are used for these applications.

2.2 Cork

Cork is one of the most versatile natural raw materials known that has been used for many centuries. It is a very lightweight material, elastic, flexible and a good electrical, thermal, sound and vibration isolator [13]. Depending on the type, it is also impermeable to gases and liquids

It comes from the bark of the oak (*Quercus suber L.*) tree, which is periodically harvested (every 9-12 years, depending on the culture region) (Fig. 2.11). It flourishes only in specific regions of the western mediterranean (Portugal, Spain, Southern France, part of Italy, North Africa) and China. Europe has about 60% of total production area (cork forest) and produces more than 80% of the world's cork, being Portugal the major cork producer with about three-quarters of all the cork[24].

Figure 2.11: Harvesting of cork oak[14]

Cork forests are of extreme importance to Europe's southern semi-arid regions, since they prevent desertification and contribute to the perfect habitat for many animals and plants of the region. These forests are also extremely important with respect to the retention of carbon dioxide (CO_2) and thus, contribute to the reduction of the greenhouse gas emissions and better climate. In Portugal, cork oak forest is responsible for the retention of about 5 million tons of CO_2 per year.

The cork oak produces three qualities of tissue. The first harvested is the virgin cork, which is irregular in structure, thickness and density, it is hard-rough and crumbly. After it is the reproduction cork from the second striping, which is more regular than virgin cork. Following, it comes reproduction cork from subsequent stips, than can be used for cork stopper production, while all types of cork can be used for agglomerates (Fig. 2.12).

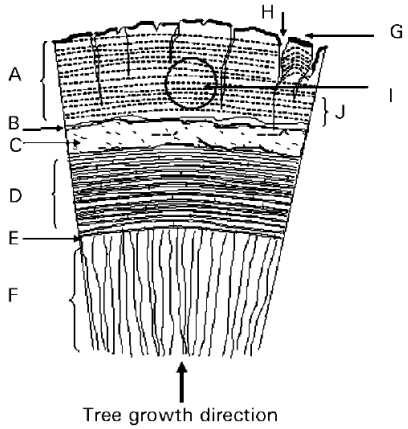


Figure 2.12: Schematic representation of axial section of cork oak tree; (A) cork (suberose tissue), (B) subero-phellogenic change, (C) phellogenium, (D) liber tissue, (E) liberwood change, (F) wood, (G) bark, (H) lenticular channels, (I) area for stopper production, (J) annual growth rings[24]

Removing cork increases the water loss of the tree from the exposed surface and affects the tree growth (wood).

2.2.1 Structure

Cork is composed of an aggregate of cells, about 43 million per cubic centimeter. The cell's dimensions can vary a lot even in the same plank.

Since the lateral cell walls (parallel to the radial direction) have a random orientation, the material can be considered transversely isotropic, meaning that all directions perpendicular to the radial direction (axial and tangential) are nearly equivalent (Fig. 2.13). Since there is a certain anisotropy in cork's cellulars structure, the properties of cork will also be anisotropic.

Schematic representation of cellular disposition in cork growing section; arrows indicate names of the three sections and corresponding directions in cork planks.

The cells itself, can be described as a rectangular prisms, that are packed base-to-base in columns parallel to the radial direction of the tree. In this direction, the cells appear to have a 4- to 9-sided polygons (heptagonal, hexagonal and pentagonal) shape, and on the axial and tangential section, the structure resembles a brick wall[24].

Cork cells are close and hollow. Inside, they contain a gas, presumably similar to air [24]. This is the secret of this material, since the mixture of gases that fill each cell are responsible for

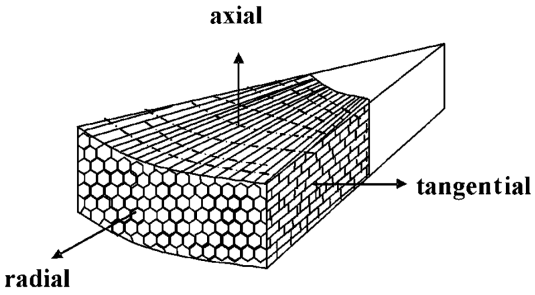


Figure 2.13: Representation of cellular disposition in cork. The arrows indicate the names of the three sections and corresponding directions[24]

cork's lightness, compressibility, elasticity and the fact that cork can be compressed half of its size without losing any flexibility.

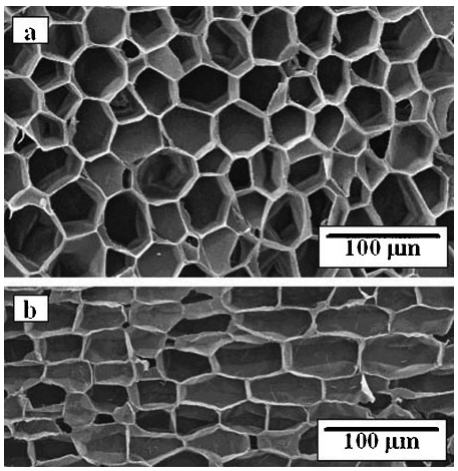


Figure 2.14: SEM micrograph of cork: a)radial section; b) tangential section [24]

2.2.2 Chemical Composition

The specific properties of cork highly depend on its chemical composition and, in turn, the chemical composition depends on factors such as the geographic origin, climate and soil conditions, genetics origin, tree dimensions, age and growth conditions [24].

As it happens in other tissues' cells, cork's cell walls can also be divided into two kinds of components: structural and non-structural. The structural components are mainly suberin (40%), lignin (22%) and polysaccharides (cellulose and hemicellulose) (18%), although the presence of polysaccharides are not considered relevant to cork's cell wall properties. The non-structural components consist of the extractables (15%) (Table 2.1).

Table 2.1: Differences in results of quantitative analysis of cork chemical composition [24]

	Virgin Cork		Reproduction Cork					
	Caldas	Pereira	Gil	Caldas	Pereira	Parameswaran	Holloway	Carvalho
Component	(1986)	(1981)	(1998)	(1986)	(1981)	(1981)	(1972)	(1968)
Suberin	45	45	42	48	33.5	33	37	50
Lignin	27	21	21.5	29	26	13	14.8	19
Polysaccharides	12	13	16	12	25	6		13
(celulose and hemicellulose)								
Extractables	10	19	13	8.5	13	24	15.8	15
Ash	5	1.2		2.1	2.5	...	3	
Others	...	0.8	7	...	...	6	...	...

More specifically, the cellular structure of cork wall consist of a thin lignin rich middle lamella, a thick secondary wall made from suberin and wax lamella and a thin tertiary wall of polysaccharides (Fig. 2.15 and 2.16).

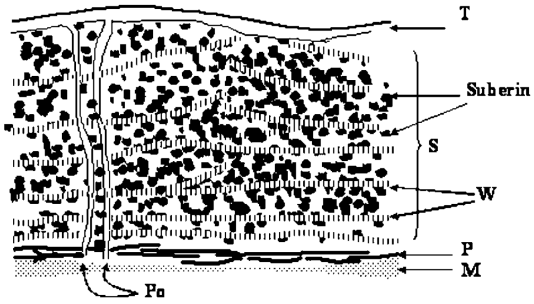


Figure 2.15: Structure of cork oak cell wall;(T) tertiary wall, (S) secondary wall, (W) waxes and suberin, (P) primary wall, (M) medium lamella, (Po) pore [24]

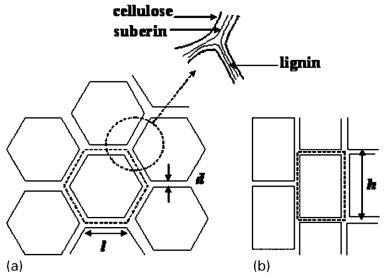


Figure 2.16: Schematic representation of cork cells; a radial section: l, prism base edge; d, wall thickness; b tangential/ axial section (perpendicular to radial direction): h, prism height; detail of cellular structure walls of cork showing its main components [24]

Suberin is the main component of cork's cell wall. It is a biopolymer (polyester type) that confers low permeability to the cork, allowing cells to be hermetic and thus, able to retain all the gases that were inside the cells, explaining the low thermal and electrical conductivity of cork. Ligning is the component that gives hardness to cork's cell walls.

2.2.3 General Properties

Cork has a set of unique properties that any other product, natural or artificial, until now, wasn't able to match [11].

Cork's specific mass can vary widely depending on it's age and treatments to which it was subjected. Normal values are somewhere between 120-240 kg/m^3. The low value of specific mass can once again be explained by the high gas content of the small cells.

The gas content and the cell size can also account for the very poor heat transfer properties of cork.

Another important characteristic of cork is the poor sound transmission. Owning to the low density and high porosity, most of the incident sound waves are absorbed and transformed into heat.

Some other special properties of cork are: good chemical stability, be impermeable to liquids and gases, good thermal insulator, fire resistant (does not release gases), low electrical conductivity, excellent energy absorption capacity and high friction coefficient. Some values of these properties can be consulted on table 2.2.

Table 2.2: General Properties of Cork; R, measured in radial direction; NR, measured in non-radial directions [24]

Property	**Value**	**Ref.**
Friction coefficient, boiled	0.2−1.2 (cork/glass and cork/steel)	111
	0.97 (cork/cork, R)	111
	0.77 (cork/cork, NR)	111
	0.76 (cork/glass, R)	111
	0.35 (cork/glass, NR)	111
Specific mass kgm^{-3}	120−180 (amadia)	29
	160−240 (virgin)	29
Surface energy, dispersive component, mJm^{-2}	24−38 (40ºC)	32, 108
	41 (25ºC)	171
Thermal conductivity, $Wm^{\check{}1}K^{\check{}1}$	0.045 (cork)	1
	0.025 (air)	1
	0.2 (cork cell walls)	1
Electrical conductivity, $Sm^{\check{}1}$	1.26×10^{-10} (25 °C)	100
	1.67×10^{-13} (50 °C)	100
Acoustic resistivity, $kgm^{\check{}2}s^{\check{}}1$	1.2×10^5	177
Specific heat, $Jkg^{\check{}1}K^{\check{}1}$	350	1
Thermal diffusivity, $m^2s^{\check{}1}$	1×10^{-6}	1
Water diffusion coefficient, $m^2s^{\check{}1}$	4×10^{-10} (NR)	1
	1×10^{-11} (R)	1

2.2.4 Mechanical Properties

Looking at the compression stress-strain curve displayed in figure 2.17, it is possible so see three different regions. Each region is related to three mechanisms responsible for the properties of flexible cellular materials [24]. In the first region, cork has a linear behaviour until 7% strain, which corresponds to elastic bending of the cell walls. The second region exhibit an almost horizontal plateau until a strain value of about 70%, caused by progressive buckling of the cell walls. The last region (sudden rise of the curve), is characterized by the start of the cells crushing until the collapse stress and strain of cork. This behaviour leads to a considerable absorption of energy when the material is under compression.

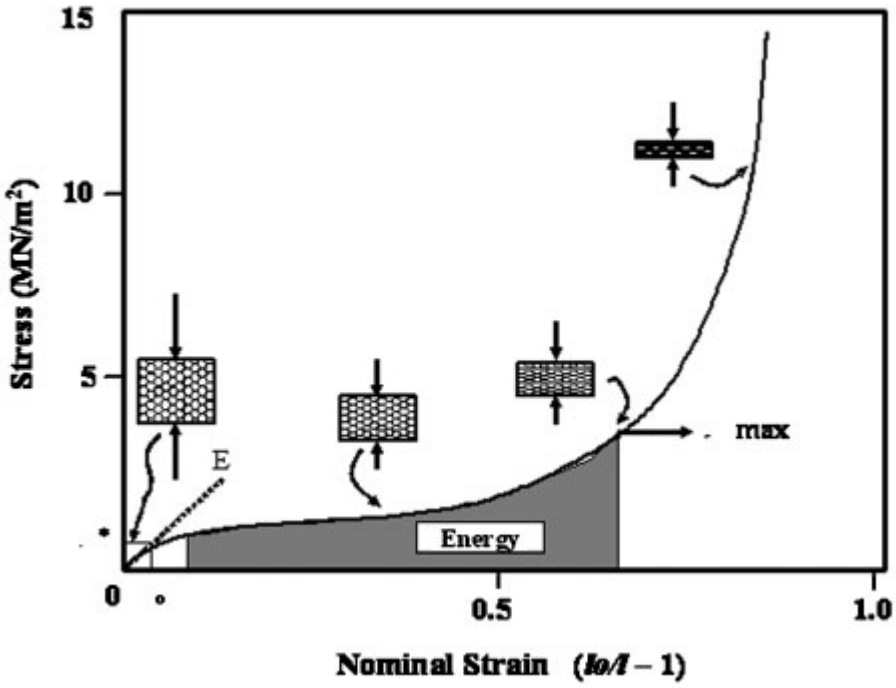


Figure 2.17: Typical compressive stress–strain curve for cork

Cork has a different behaviour when subjected to tension. In compression, the Young's modulus is smaller than in tension due to the stiffness of the undulated plates (cell walls), which increases as the amplitude of the undulation decreases, and when the material is subjected to compression, the amplitude increases, whereas in tension, it decreases [19].

The average stress-strain curve from tensile tests displays different behaviours depending on the direction of the load (axial, radial or tangential). The non-radial directions are quite distinctive when compared to the radial direction curve (Fig. 2.18). In the radial direction, there is an intermediate unstable region due to successive appearance of micro-cracks, which only propagate across only a few surrounding cells [19].

When cork is subjected to a severe impact situation, it is capable to recover 85% of the original thickness and it can recover 100% in less severe impacts, seldom presenting delaminations during these solicitations, making cork an excellent natural energy absorber [19].

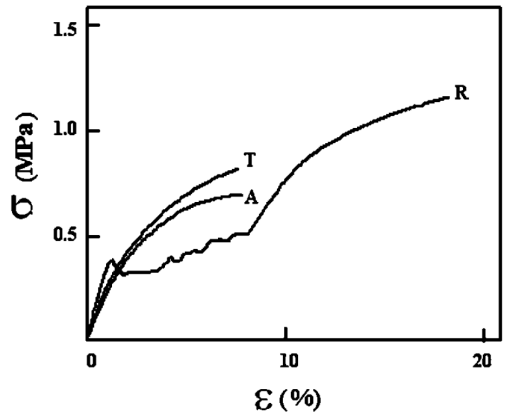


Figure 2.18: Stress–strain curves in tensile tests for cork, in all directions: T, tangential; A, axial; R, radial [24]

Table 2.3: General mechanical properties of cork; R, measured in radial direction; NR, measured in non-radial directions [24]

Property	**Value**	**Ref.**
Compressive modulus, natural cork, unboiled, MPa	8−20 (R)	105, 107, 111
	13−15 (NR)	107
Compressive modulus, boiled, MPa	6 (R)	107
	8−9 (NR)	107
Compressive modulus, heat treated at 100 °C, 28 days, MPa	11 (R)	105
	11 (NR)	105
Compressive modulus, heat treated at 150 °C, 28 days, MPa	15 (R)	105
	14 (NR)	105
Tensile modulus, boiled, MPa	38 (R)	97
	24−26 (NR)	97
Collapse (buckling) stress, boiled, MPa	0.75−0.8 (R)	24, 111
	0.6−0.7 (NR)	24, 111
Collapse (buckling) strain, %	4 (R)	24
	6 (NR)	24
Fracture stress under tension, MPa	1.0 (R)	24
	1.1 (NR)	24
Fracture strain under tension, %	5 (R)	24
	9 (NR)	24
Fracture toughness, boiled, MPa $m^{\frac{1}{2}}$	60−30	97
Poisson's ratio, boiled	0−0.097 ($\nu_{R/NR}$)	24, 100
	0−0.064 ($\nu_{R/NR}$)	24, 100
	0.26−0.5 ($\nu_{R/NR}$)	24, 100
Loss coefficient at 0.01 Hz	0.1−0.3	24, 78

2.2.5 Processing

Before start using cork, natural cork bark passes through several selections and manipulations. All cork must be boiled before working to make it more pliable and to fully expand its lenticels [24].

Initially, cork cells are collapsed and wrinkled, so the material undergoes a boiling treatment for 1 hour at 100ºC [24]. This makes the interior gas in the cells to expand, creating a very tight and uniform cell structure. The aftermath result is a 30% expansion of the initial volume and elimination of water soluble components that were previously on the cork.

2.2.6 Wetting

One of the properties of high importance while dealing with laminate composites is the capability of the material to adhere to the resin, in this situation, the ability of cork to adhere to the resin (wetting).

In order to create a strong bond, it is necessary that the resin has the ability to wet and to spread on cork's surface. To be able to do it, resin needs to have a low viscosity and a small angle of contact between cork and the adhesive.

When a liquid drop is put on the top of a surface, it might spread or stay as a drop, with a certain angle θ with the surface (Fig. 2.19). When the contact angle is zero, the liquid spreads. If it's lower than 90º, the liquid wets the solid, if higher than 90º, the liquid doesn't wet the solid.

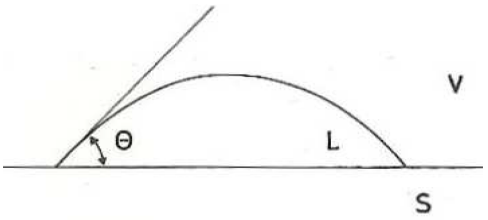


Figure 2.19: Contact angle between the surface and the liquid [12]

Cork has a very low surface tension when compared with other materials (similar to the values of polymers). Small values of surface tension, imply low wetting and high contact angles.

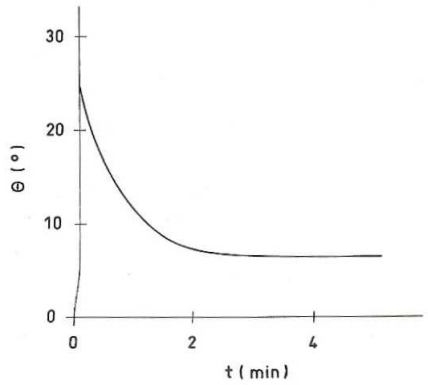


Figure 2.20: Variation of contact angle with time of a cork-polyester system

2.2.7 Cork Agglomerates

Cork agglomerates can be divided into two categories: composition corkboard and expanded corkboard.

Composition corkboard is made of granules that have been joined together using different synthetic or natural binding agents, usually urethane, melaminic or phenolic resins. A mixture of granules and glue and/or other additives are put into a mould, which is closed and heated at a temperature usually higher than 120ºC for 4 up to 22 hours. After heating, the block might be cooled down and slice it into sheets. The physical and chemical characteristics of the binder determine the strength of the agglomerate and therefore its applications [13].

The main difference between composition corkboard and expanded corkboard is that expanded corkboard is made only with cork, that means, without any external binding agent or any other added material [13].

The manufacturing process of a expanded corkboard consists on basically putting the material into a mould and then, in a closed autoclave at high temperature (approximately 300ºC) and pressure (around 40 kPa). The cork cells expand by unfolding the cell wall corrugations, resulting in an increase of cell volume of about 100%. This process induces thermomechanical degradation of the cork cell wall and the degradation of the byproducts act as natural adhesives between the granules to form the corkboard and it's the degraded cell walls that is responsible for the final dark appearance and the weight loss (approximately 30% of the initial weight). After forming, the blocks are transferred to a cooling machine that injects recycled water (at close to 100ºC) for drying and stabilization [24].

This expanded black agglomerates are produced from the lowest quality and residual corks. Usually,the residual cork comes from cork that is unsuitable for other applications, from wasted cork and residues from other industrial processes.

One important advantage of insulation corkboad is its resistance to chemical and biological agents, since it only reacts in the presence of strong acid solutions. The properties of this type of material are summarised on table 2.4.

Globally, cork agglomerates are able to bond will all standard laminating resins (unsaturated polyester, polyurethane, vinyl ester, epoxide and phenolic). They also work well in hand lay-up and RTM, and are compatible with most prepregs. They also support high processing and working temperatures, since its mass loss is relatively small (approximately 6%) until 200ºC, however at 450ºC, the agglomerate suffers complete carbonization, completely destroying cork cells' structure. Cork is also easy to machine with standard tools and the agglomerates have excellent conformability [19].

Table 2.4: Properties of expanded cork agglomerate [24]

Property	**Range of values**
Specific mass, kgm^{-3}	100-130
Working temperature, K	97-383
Thermal conductivity (20 °C), $kJm^{-1}s^{-1}K^{-1}$	3.1×10^{-5}
Specific heat (20 °C), $kJkg^{-1}K^{-1}$	1.7-2.1
Thermal expansion coefficient	40×10^{-6}
Permeability to steam, $kgPa^{-1}s^{-1}m^{-1}$	4.2×10^{-12} to 12×10^{-12}
Tensile strength, MPa	0.05
Compressive strength at 10%, MPa	0.25
Bending tension, kNm^{-2}	1.6×10^{-4}

2.2.8 Applications

The main application of this material is cork stoppers, since cork has a set of characteristics that makes it the ideal material for it such as, being waterproof and being highly elastic. The elasticity is of high importance for cork stoppers since the membrane cells are flexible and therefore cork stoppers become under pressure inside bottle neck and, when released, return to the original shape.

Although cork stoppers is the goal standard of cork applications, its usage goes way beyond. Some examples are the thermal insulation in refrigerators and rockets, or acoustic and vibration insulation in machines such as compressors, hydraulic presses, turbines, generators or motors. Cork's acoustic and vibration Cork's acoustic and vibration proprieties can also be used in submarines or in recording studio.

Another possibility to use cork is in coatings/coverings in walls, ceilings and floors, or in packaging, since cork is able to compress when impacted, protecting the package.

It is also possible to see cork integrated into oil absorbent products and organic solvents for pollution control, or shoe soles and handles due to it's friction (anti-sliding) properties. Out of the engineering scope, cork is also integrated in design elements for it's aesthetics.

2.2.9 Innovation and Cork Powder

Cork industry has been growing in the past years in Portugal. Therefore, investigation has been growing as well, specially on exploitation of cork products and byproducts, such as water used in the cork boiling process or cork powder. These byproducts are not considered waste anymore, but as feedstock for new applications.

Concerning cork powder, its usage has been growing due to its low cost and properties, namely the absorption capacity. An example is the absorption of contaminating materials or the removal of heavy metals in residual waters. Cork powder is also used as a filling material (to improve the

properties of another base material) to improve fire resistance, chemical stability or mechanical properties, such as compressing resistance.

2.3 Low Velocity Impact

As it is being constantly mentioned in this document, composite materials have seen a rapid growth in the last decades, however, their susceptibility to impact damage is still one of the major concerns, since the induced damage can significantly reduce its structural integrity.

Low or high velocity impact damage can be introduced as a result of different events during manufacturing, normal operations, maintenance, or event as impact of hailstones, runway debris, bird strikes, etc.

It is important also to define what does low and high velocity mean, and there are several definitions on the literature. The usual one states that an impact event is considered to be a low velocity impact if the contact period of the impactor is longer than the time period of the lowest vibrational mode. In this regime, the support conditions are of extreme importance since the stress waves generated from the impact point have time to reach the edges of the structural element, resulting in a full-vibrational response. In common epoxy composites, the transition to a stress wave-dominated impact occurs at impact velocities between 10 and 20 m/s [21].

In opposition, when dealing with a high velocity or ballistic impact, the contact period of the impactor is much smaller than the time period of the lowest vibrational mode of the structure. The response of the structural element is governed by the local behaviour of the material in the neighborhood of the impacted zone. In this type of events, the support conditions don't matter when dealing with the impact response [21].

2.4 Basic Types of Damage in Composite Systems

There are a huge variety of damage that composite systems can suffer. These damages can happen due to different events such as static load, fatigue, moisture, corrosion or as mentioned before, low energy impacts. The last one is, in particular, potentially dangerous, since it can produce damages under the surface of the material that are not visible, but they still contribute the the decrease of some mechanical properties. There is the concept of Barely Visible Impact Damage (BVID), that stands for the extent of which the damage is not clear from the surface, but causes debilitating internal damage. If a composite laminate is subjected to a low velocity impact with sufficient energy, impact could cause various damages, such as matrix cracks, delaminations, fibre breakage and fibre-matrix debonding [26].

2.4.1 Fibre Breakage

Fibre-reinforced composites are made of a bundle of fibres but, the failure of them are not the same and don't occur at the same time. Due to differences in fibre diameter and defects induced

during the fibres manufacturing process, or even during the manufacturing process of the material itself, can make fibres to rupture at different applied stresses.

The breakage of a fibre creates a stress concentration zone that can lead to the rupture of neighbour fibres and, thus rupture of the material itself.

Sometimes, fibres are coated by a material that reduces the possibility of rupture of a fibre due to the influence of the rupture of neighbour fibres by "isolating" the rupture effects.

2.4.2 Matrix Cracking

This type of damage is characterized by localized, partial, through-the-thickness cracking (Fig. 2.21). It can happen at relatively small loads, for instance, during thermal expansion of a curing cycle, since the matrix cracking is generated by overstressing of the matrix through various loading conditions [10].

Fortunately, these type of cracks don't have an immediate effect on the component's strength since, in order for the whole composite to break, fibres also have to break. However, they should not be ignored, since the open component is susceptible to further environmental degradation, for instance: moisture, corrosion or the influence of other chemicals and fluids. Also, matrix cracks can be a source of delamination initiation, that will further degrade the component and eventually, lead to its failure [10].

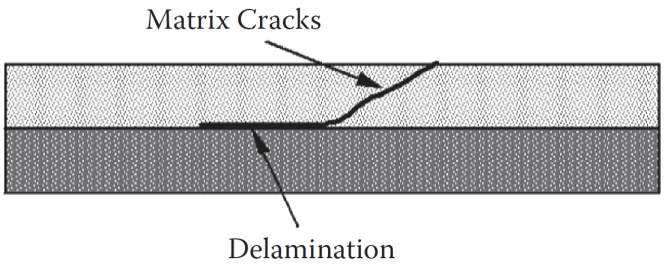


Figure 2.21: Matrix crack and delamination initiation [15]

2.4.3 Fibre/Matrix Debonds

This type of damage consists of a separation at the fibre/matrix interface (Fig. 2.22). Fibre/matrix debonding comes from an excessive local shear-transfer stresses, particularly where short fibres are present. It will result on a loss of shear transfer and degradation of the overall strength of the laminate, since most of the good properties of composite materials comes from strong bonds between fibres and the matrix. Besides, fibre/matrix debonding is hard to detect.

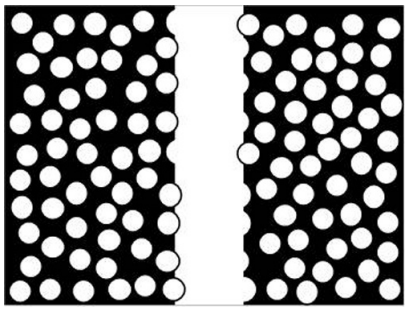


Figure 2.22: Fibre-matrix debonding [15]

2.4.4 Delaminations

Delaminations, also called interlaminar cracking, are one of the most frequent types of damage found in advanced composite materials. They are considered a matrix defect, where in-plane matrix cracks than run parallel to the fibre direction, propagate between plies of a laminate.

This type of damage can appear and grow in both static and cyclic tensile loading, but they are a damage typically associated to compression and shear stresses, causing significant degradation to the material's compressive and shear strengths. When a delaminated composite material is subjected to tension, the residual strength is generally reduced by only 10 to 15%.

Delaminations are quite difficult to detect since the damage shown on the surface is considerably lower than the damage that happens on the inside of the laminate. Another problem associated with this type of damage is that delaminations near the surface grow in a stable manner and induce a negligible strength loss, but the larger the delamination and the deeper it is located within the laminate, the larger is the strength loss. Besides, delaminations induce local interlaminar stresses.

The laminate response to delaminations is influenced by the delamination size, location, laminate orientation/stacking sequence and by the test environment. They can happen in three different single modes (or combinations between them) (Fig. 2.23):

- Mode I (opening), associated with tensile stresses;
- Mode II (shearing), associated with in-plane shear stresses;
- Mode III (tearing), associated with out of plane shear stresses.

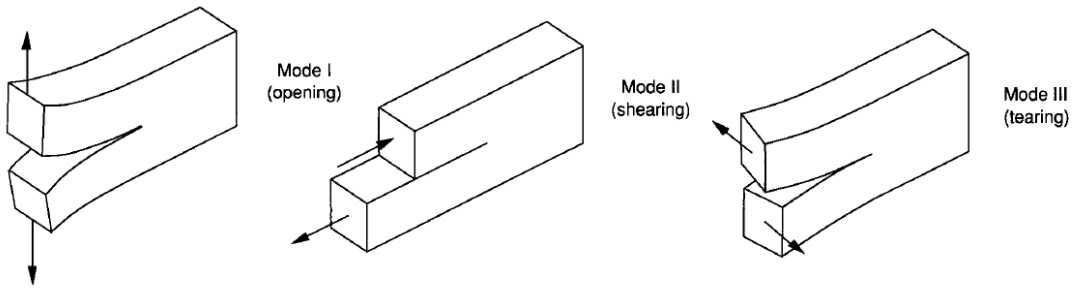


Figure 2.23: Basic delamination modes [10]

2.5 Damage after Impact and Damage Tolerance

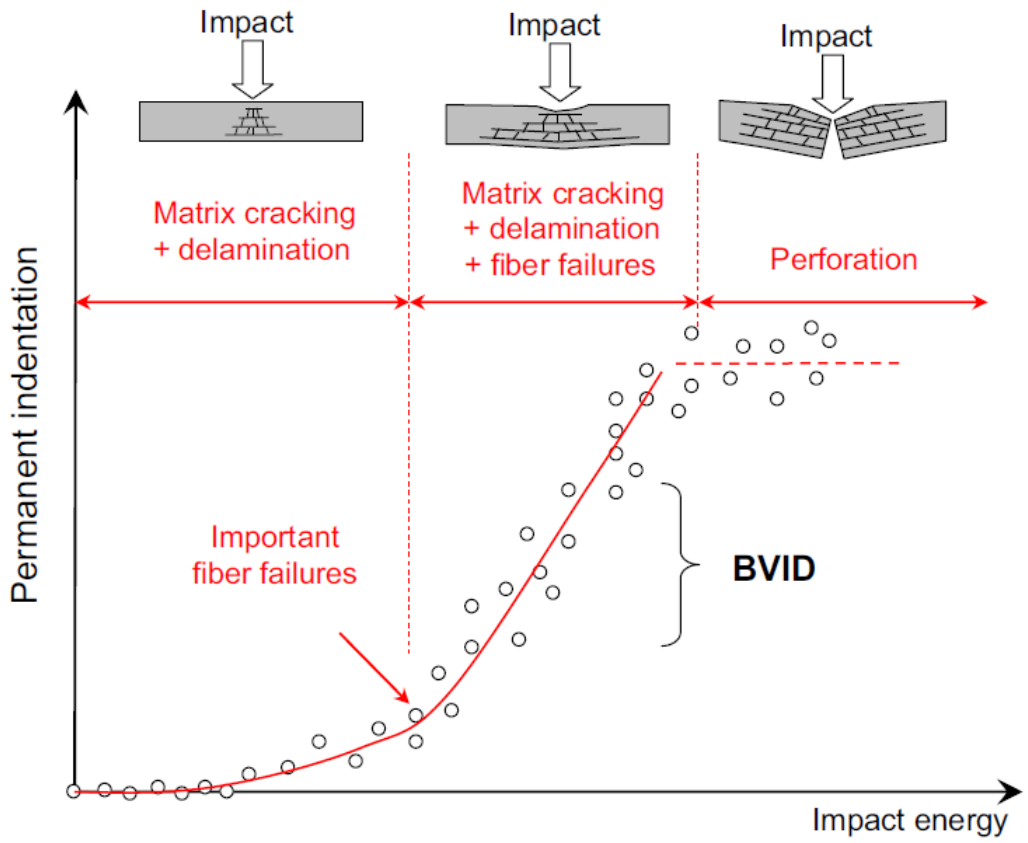


Figure 2.24: Schematic evolution of permanent indentation versus impact energy level [22]

Permanent indentation is a crucial element to design a composite structure using impact damage tolerance. Figure 2.24 shows the typical evolution of the permanent indentation versus impact energy level. The evolution presented in the graph admits three different regions.

The first part refers to low-impact energy levels, during which the damage consists of small extended-matrix cracking and delaminations. During the unloading (rebound of the impactor), the shear matrix cracking and delaminations remain partly open, leading to a relatively small permanent indentation (generally less than BVID). These matrix cracks, resin and fibre debris, that were created during the impact, block and prevent the closure [22].

The second part concerns the second level of energy, and it's where the BVID is usually reached. In this energy level, besides matrix cracking and delaminations, there is also fibre fractures mainly located under the impactor, responsible to a faster increase of permanent indentation. These fibre fractures are due to compression loadings under the impactor and traction in mid-thickness or lower part of the plate [22].

Generally, there is no fibre failure in the lowest ply because delaminations in this area tend to unload it. The main fibre failures are generally located between the mid-thickness and at the

non-impacted side of the plate. These fibre breakages, have detrimental effects on the residual strength of the material after impact.

It is in this part, that the most critical cases of impact damage tolerance happen, since the impacts might produce a permanent indentation slightly lower than BVID and therefore undetectable by visual inspection. On one hand, it is important to prevent fibre failure to avoid excessive decrease of the residual strength after impact, and on the other hand it is important to promote the fibre failure to improve detectability of impact damage [22].

As it it possible to see by Fig 2.24, the last part deals with a large energy level impact, near perforation. This part is "paradoxically" less dangerous that the previous case, due to the fact that the damage is easily detected [22].

2.6 Mechanical Tests

2.6.1 Tensile Test

The tensile test is probably the most simple and used mechanical test done [11]. It is used to investigate the tensile behaviour of materials, meaning: the tensile strength, tensile modulus and other aspects of the tensile stress/strain relationship.

The principle is that a specimen is extended along its major longitudinal axis at constant speed, until it fractures, or until the stress (load) or the strain (elongation) reaches some predetermined value. During this process, the load sustained and the elongation are measured.

In case of tensile tests for isotropic and orthotropic fibre-reinforced plastic composites [6], it is possible to use three different specimens types shown on figure 3.6a.

Figure 2.25: Type 1B specimen[7, 6]

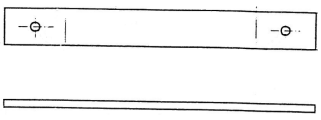


Figure 2.26: Type 2 specimen[7, 6]

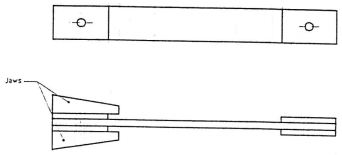


Figure 2.27: Type 3 specimen[7, 6]

2.6.2 Drop-Weight Test

The drop-weight test consists of having a symmetric, flat laminated plate that is subjected to an out-of-plane concentrated impact, perpendicularly to the plane of the laminated plate. The impact consists of a drop-weight device with a hemispherical tup, and thus its possible to control the potential energy, controlling the mass and/or drop height of the impactor (Fig. 2.28). Usually this test induces low-velocity impacts.

The test is able to establish quantitatively effects of stacking sequence, fibre surface treatment, variations in fibre volume fraction, processing and environmental variables on the same resistance of a particular laminate, when it is submitted to a drop-weight impact. It is also possible to use this information to compare quantitatively the relative values of damage resistance parameter for composite materials with different constituents. The damage resistance parameters are also highly dependent upon several factors including: specimen geometry, layup, ply thickness, stacking sequence, impactor mass, velocity and tip, environment conditions and boundary conditions. Damage response parameters include: dent depth, damage dimensions, through-thickness locations and force versus time curve. On the other hand, the damage response is a function of the test configuration, so comparisons can only be made if the materials have identical test configurations and test conditions.

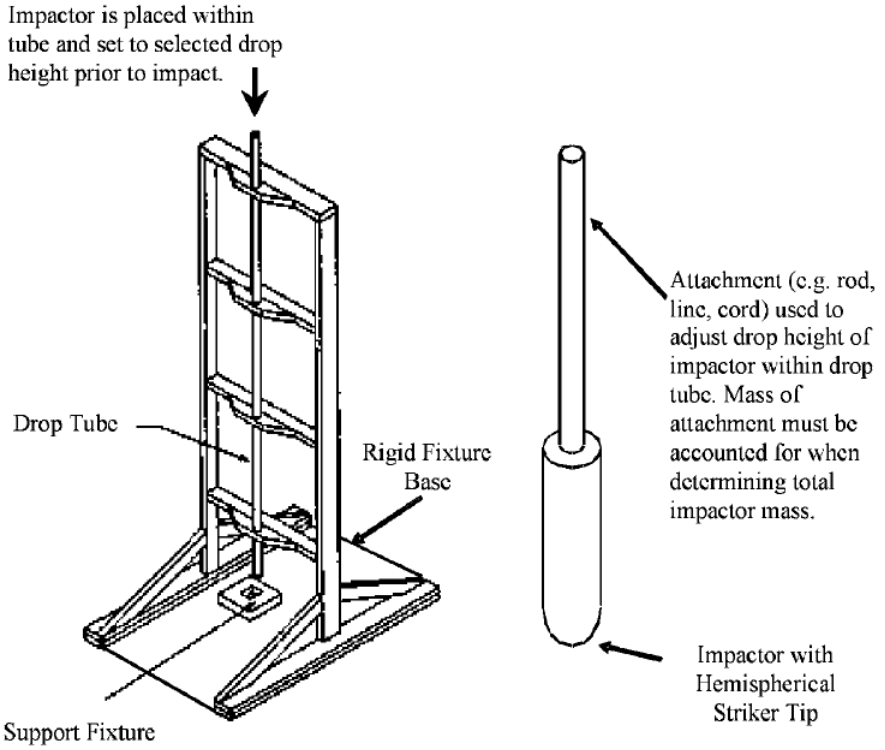


Figure 2.28: Impact Device with Cylindrical Tube Impactor Guide Mechanism [1]

2.7 State of Art

It was already mentioned before in this document the material used for this study, and the problems that composites have with impact events. Fibre-reinforced polymers, specially CFFP, have brittle-type behaviour, which can constitute a serious problem in terms of damage tolerance [**?**]. Carbon fibre reinforced with epoxy resin have the tendency to have delaminations when the material is submitted to impact or cyclic loadings.

Some factors that can influence the impact damage resistance of composite laminates include the interface angle, ply orientation relative to a fixed axis and ply grouping [26]. Although these parameters are referred to impact damage resistance, they are the foundation for the prediction of residual strength (tension and compression) after impact [26], which is of extreme importance in the application of composite materials due to the fact that if a composite laminate that has not completely failed after being subjected to an impact load, and it has internal and/or surface damages, it may still carry loads [18].

2.7.1 Methods to Enhance Damage Tolerance

The damage tolerance of composite laminates can be globally improved if the initiation and growth of delamination can be either prevent or delayed. The delaminations' control focus usually on the improvement of the interlaminar fracture toughness and in the reduction of the interlaminar stress [18].

The most relevant parameters that influence the damage tolerance include: matrix toughness, fibre-matrix interfacial strength, fibre orientation, stacking sequence, laminate thickness and support conditions.

Two of the most frequent methods to enhance the damage tolerance are the interlayer toughening and matrix toughening [16]. These and other methods are mentioned in the following section.

Interlayer Toughening

By using cork, Interlayer toughening was the method chosen to enhance damage tolerance in composite systems.

It can be split into two types: homogeneous and heterogeneous.

The homogeneous type consist of adding a resin film (thermoplastic or thermosetting) with a higher toughness that the toughness of the matrix used. Thermoplastic resins are more suitable for the purpose of damage tolerance enhancement, since they have better impact properties [16].

Concerning the heterogeneous type, it stand for the introduction of rubber or thermoplastic particles between the composite layers. The particle size plays an important role in the final composite performance since, if particles are too small, they can become embedded in the fibres and, if particles are too big, they can behave as porosities or defects [18].

This method revealed good results in the improvement of interlaminar fracture resistance, but on the other hand, it can increase the material's weight and volume, and loss of mechanical properties such as tensile strength and young's modulus, when compared with the same material without the additives [18, 16].

Matrix Toughening

Another way to improve the damage tolerance of composite systems is by adding other materials in the matrix, increasing its fracture toughness, and thus increasing the interlaminar fracture toughness of the whole material. The fracture toughness of an epoxy resin can be increase by adding

elastomers, reducing cross-link density, increase the resin chain flexibility between cross-links, or a combination of all three [18].

Adding thermoplastic resins also improves the fracture toughness of the composite. Depending on the thermoplastic used, it is possible to obtain three different matrix states: homogeneous mixture with the resin, separated by phase or particles that can appear spread in the matrix [11].

Stacking Sequence

Stacking sequences create a mismatch of Poisson's ratios and coefficients of mutual influence between adjacent layers, which can create high interlaminar and shear stresses at the free edges of a laminate. Changing the stacking sequence can change the interlaminar normal stress from tensile to compressive, so that the opening mode delamination can be concealed [18].

Interply Hybridization

This method is another way to reduce the mismatch of Poisson's ratios and coefficients of mutual influence between consecutive plies [18].

Stitching

One way to minimize the delaminations is by increasing the reinforcement through the thickness of the laminate. One way to do it is by using the stitching method.

Using this method does not create relevant effect on the amount of fibre ruptures or interlaminar damage in impact loadings, but it contributes to the distribution of the damage throughout the thickness of the laminate, slowing down the delaminations near the impacted zone and highly increases the compression after impact [11].

Although stitching may not prevent the occurence of free-edge delamination, it can considerably reduce the rate of delamination growth in the interior of the laminate. On the other hand, introducing stitches can damage the fibres and create resin concentrations, that might lead to a decrease of young's modulus and tensile strength [18].

It was also verified that stitching is only effective until a certain impact energy level, since for higher energies, the results with or without stitches is practically the same [11].

Z-Pinning

Similar to the stitching method in the sense that it improves the mechanical properties throughout the thickness, reducing the risk of delaminations. The difference is that in this situation, the reinforcement is in the form of metallic pins.

It was shown that it increases the impact resistance of structures in a wide range of impact energies, but the insertion of metallic pins, slightly decreases the amount of absorbed energy [11].

It was also verified that this method is more effective for thicker laminates or higher energy impact levels [11].

2.7.1.1 Cork to Enhance Damage Tolerance in Composite Systems

The influence of cork in the damage tolerance of composite systems has been studied during the past years during research projects and master's thesis. In this section, some of the most relevant discoveries about cork as a way to enhance the damage tolerance in composite systems will be mentioned.

In a paper from 2003 [16], the increase of damage tolerance of carbon fibre reinforced plastic was studied by adding tough materials as interlayers, such as cork or Kraton™ rubber. Three "families" of laminates were made: reference, laminate with cork powder and laminate with Kraton™ rubber powder to study it. The results showed that the reference laminates were the ones with better mechanical properties, but had the worst impact behaviour and worst damage tolerance values. Concerning the laminates with Kraton™ powder, they showed the best damage tolerance values, among the solutions studied, but worst mechanical properties. Laminates with cork powder managed to have a compromise between the other two "families", since they showed both mechanical properties and damage tolerance values between the other families values.

In a master's thesis [11], the objective to determine if cork was a feasible option as a interlaminar toughening solution and how was it possible to maximize cork's contribution. In order to solve this problem, laminates with different cork film configurations and stacking sequences were made. The results were different from what it was expected, since they showed that cork was probably not a good solution for this problem or its usage in this research was not effective. Cork as interlayer toughening, increased the toughness and energy absorbed when the material was bended but, there was a decrease of the material's stiffness, yield and shear strength. During Charpy impact tests, the results showed that the lower the amount of cork, the bigger is the amount of energy absorbed, but the author assumed that it might have happened due to weak bonding between the resin and the cork.

In another master's thesis [20], the aim was to understand the influence of the position and quantity of cork films to enhance the damage tolerance and impact behaviour of composite systems. In order to do it, eight laminate plates with different configurations were made. The laminates had varied the number of cork films and their position among four glass fibre layers. Results showed that adding cork films to the laminate substantially reduces the mechanical properties of the material. After the impact of 2 and 4 J, the plates with cork showed a lower reduction of mechanical properties when compared with the reference laminate. It was confirmed that the main disadvantage in adding cork films to a composite laminate was the considerable reduction of the mechanical properties, but the main advantage was, according with the results, the lower reduction of mechanical properties after being submitted to a low energy impact. It was also found that cork's films position in tensile tests didn't reveal a considerable importance, but in impact tests, cork films in the center ended up absorbing more energy.

In the field of sandwich structured composites, the use of cork was also studied. The research [**?**], explores the use of cork based composites for structural composites in the context of aerospace applications, where carbon-epoxy sandwich skins with a cork-epoxy core damage tol-

erance capacity was studied. The results showed that the use of cork-epoxy cores lead to a higher energy absorption with a lesser damage extension of both the core and the facesheet, supported by the results of the residual strength tests after impact, which the cork-epoxy cores withstand higher values of ultimate load when compared with PMI specimens, and by the smaller damaged area caused by impact of the same energy level. In the same research, it was studied composite laminates with embedded cork granules within the laminate, and it showed an improvement of resilient properties under impact loadings.

Still in the field of carbon-cork sandwich composites for aerospace applications, another study [23] showed that cork agglomerates performance depends on the cork granulate size, the type of reinforcing elements and the bonding procedure used for the cohesion with the matrix material, via impact tests of different types of sandwich specimens. In the same study, sandwich components with enhanced cork agglomerates were compared to high performance foams, and it showed that the first ones have a higher energy absorption capacity.

Another investigation on cork agglomerates [9] aimed to develop cork agglomerates with enhanced mechanical properties and evaluate their performance when integrated as core materials in sandwich structures. In this research, cork agglomerates with enhanced mechanical performance were fabricated with epoxy resin and their main properties were compared with both conventional cork agglomerates and high strength core materials usually used in sandwich components (foams). The results suggested that, compared with the high performance foams, the optimized cork agglomerates have a higher energy absorption capacity with minimum damage occurrence. The experimental tests also revealed that cork agglomerates' performance essentially depends on: cork granule size, its specific mass and bonding procedure used for the cohesion of the granulates.

Chapter 3

Experimental Procedure

As it was aforementioned, this study aims to understand how cork can enhance the damage tolerance in composite systems. In order to do it, a specific stacking sequence for the laminates has been chosen, and in two interfaces of the laminate (between plies), it was added a tough material. Since the main material of the study was cork, two of the interply materials were cork films with different thicknesses and 4 of them were expanded cork granules with different concentrations. In other to compare the cork solutions with other materials, there was also a reference laminate (without any added material as interlayer) and Kraton ™ rubber granules with 3 different concentrations.

3.1 Laminates

To produce the specimens, 11 different laminates with area $300 \times 300 mm^2$ were produced having the following stacking sequence:

$$[(0/+45)/(0/-45)/interface/(45/90)/(-45/90)/(90/-45)/(90/45)/interface/(-45/0)/(45/0)]$$

As it is possible to see, the interfaces were put in the critical part of the sequence, since according with the literature, the critical interface (damage wise) is when there is a biggest difference in angles of the plies (in this situation: 90º difference). The interfaces applied and the designation use are summed up in the table 3.1 the second laminate was use to collect 6 more tensile test specimens.

3.1.1 Prepreg

As it was mentioned before, 11 laminates were fabricated with different interfaces. The composite plies chosen were a thin-ply carbon fibre pre-impregnated with an epoxy resin.

Table 3.1: Designation and quantity used for each laminate

Laminate Designation	Interface	Quantity
REF	-	2
C1	Thin cork film	1
C2	Thick cork film	1
K30	Kraton™ granules $30g/m^2$	1
K40	Kraton™ granules $40g/m^2$	1
K60	Kraton™ granules $60g/m^2$	1
B10	Expanded cork granules $10g/m^2$	1
B20	Expanded cork granules $20g/m^2$	1
B30	Expanded cork granules $30g/m^2$	1
B40	Expanded cork granules $40g/m^2$	1

3.1.1.1 Thin-ply Carbon-Fibre Prepreg

The biaxial prepregs used came from the Chomarat Composites company, and since there were two different configurations ((0/+45) and (0/-45)), two models were used to build the laminate. The references of the materials are:

- C-PLY™ SP 0/45 75/75 CT3,4 12K HS [4]
- C-PLY™ SP 0/-45 75/75 CT3,4 12K HS[3]

They are a biaxial spread-two carbon NCF (Non Crimp Fabric) with 75gsm per ply.

3.1.1.2 Matrix

The prepreg matrix used was a HexPly® M21[5] from the HexCel company. It is a high performance, very tough epoxy resin that cures at 180ºC. It is primarily used in aerospace structures. It has the particularity of exhibiting excellent damage tolerance, especially at high energy impacts, since it has excellent toughness. Some of the other features are its high residual compression strength after impact and the low exotherm behaviour (allowing simple cures of thick structures). It is also a resin best suited to press or autoclave cure, ending up obtaining optimum mechanical performance.

3.1.1.3 Interface Materials

In this section, some of the most relevant characteristics of the materials used as interfaces are mentioned. This materials and configurations were chosen due to their toughness capacity.

Cork

Since cork was the main focus of this study, various forms of this material were used as interface. Either being a film or being granules, one important thing about adding this material as interface, is the fact that cork should go to a climate chamber for a period of 12 to 24 hours with a temperature

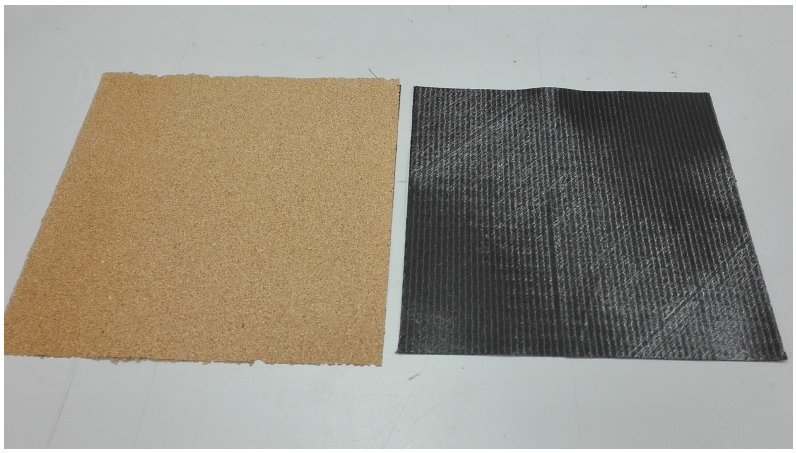


Figure 3.1: Thin cork film being applied on the laminate as interlayer

that can range between 50 to 70 ºC to reduce the moisture and dust particles in the cork, to promote a better adherence with the laminate. The chamber used was the FITOCLIMA 300 EDTU that was in the composites workshop of INEGI.

Thin Cork film - C1

The film used (Fig. 3.1) has the reference 8245 from the company Amorim Cork Composites. This material is composed by cork granules with size between 1 and 2 mm binded with polyurethane Some of the characteristics of this material are represented on table 3.3.

Table 3.2: Properties of 8245 cork film

Property	**Value**	**Unit**
Specific mass	150-210	kg/m^3
Tensile strength	≥ 300	kPa
Compressibility	30-50	%
Recovery	≥ 70	%

In order to have more specific values, it was used three samples to collect some measurements, and the final average results are shown on table 3.3.

Table 3.3: Properties of 8245 cork samples

Width [mm]	**Height [mm]**	**Thickness [mm]**	**Mass [g]**	**Specific mass [kg/m^3]**
296	308.5	0.363	6.85	186.44

Thick Cork Film - C2

Another cork used material as an interlayer was a “thick” cork film. The film used has the reference name CORECORK NL20 from Amorim Cork Composites. This material has interesting

characteristics that makes it a good candidate as damage tolerance enhancer, such as good mechanical properties (Table 3.4), low density, flexibility, excellent conformability and the fact that is a stable material. CORECORK NL20 also has some characteristics that can guarantee some success in the integration within a laminate composite such as: good processing characteristics, possibility to be easily integrated into fast cycles of production, the ability to withstand process temperatures up to 180º C and excellent resin compatibility for: epoxy, polyester, phenolic, vinylester and polyurethane.

Table 3.4: Properties of CORECORK NL20

Property	**Value**	**Unit**
Specific mass	200	kg/m^3
Compressive Strength	0.5	MPa
Compressive Modulus	6.0	MPa
Tensile Strength	0.7	MPa
Shear Strength	0.9	MPa
Shear Modulus	5.9	MPa
Thermal Conductivity	0.042	W/mK

In order to be sure about some of the properties of this material to use it as an interlayer material, some measurements were taken. These properties are represented on table 3.5.

Table 3.5: Measures taken from CORECORK NL20 samples

Sample	**Length 1 [mm]**	**Length 2 [mm]**	**Thickness [mm]**	**Mass [g]**	**Specific mass [kg/m^3]**
1	200.50	202.25	0.80	7.35	226.57
2	196.50	202.50	0.82	7.18	221.18
3	200.50	201.50	0.82	7.10	214.32
Average			0.81	7.21	220.69

Expanded Cork Granules - B10, B20, B30 and B40

Another way that cork can be used as an interlayer tough material (Fig. 3.2), is in its granules form (Fig. 3.3). This material is a by-product obtained during the expanded insulation corkboard production. The expanded cork was considered as one of the options, since the "normal" one could suffer some degradation during the laminate processing. As the expanded one was already processed, it can withstand the pressures and temperatures used.

Some of the characteristic of this material were mentioned in 2.2.7. Table 3.7 shows some properties of this material.

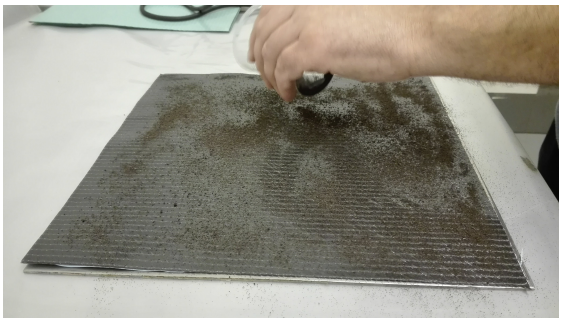


Figure 3.2: Expanded cork granules being deposited as an interlayer material

Figure 3.3: Expanded cork granules

Table 3.6: Properties of expanded cork

Property	Results
Thermal Conductivity	0,041 W/(m ºC)
Loose Bulk Density	60 kg/m^3
Reaction to Fire	Class E

During this experiment, the granules had a size of about from 0 to 1 mm and the designations and respective concentration is shown on table 3.7.

Table 3.7: Properties of expanded cork

Designation	Concentration	Mass for each ply
B10	10 g/ply	0.9 g
B20	20 g/ply	1.8 g
B30	30 g/ply	2.7 g
B40	40 g/ply	3.6 g

Kraton™ Granules

In order to compare the results obtained with cork, it was used another material, Kraton™ granules, with different concentrations. For this study, it was used the material with a designation Kraton™ D-1102 and some of its properties can be consulted in table 3.8.

Table 3.8: Properties of Kraton™ D-1102

Propriety	Value	Unit
Specific mass	0.938	g/cm^3
Apparent (bulk) density	0.40	g/cm^3
Solution viscosity	900 to 1500	mPa.s
Tensile stress (300% strain)	2.90	MPa
Tensile stress (yield)	33.0	MPa
Tensile elongation (break)	880	MPa

Kraton™ is a high performance material, which is a linear copolymer based on styrene and butadiene with about 29,5% of ramified styrene. It is used to increase the performance of a wide set of products. This versatility is due to its molecular structure, which can be precisely controlled and the molecular structure can be customized according with specific applications. Some examples of this increase of performance are the mixing with polyolefin, styrenes and other engineering thermoplastics, which Kraton™ can highly increase the impact resistance at ambient and low temperatures and can also improve the mechanical resistance at higher temperatures. Kraton™ can also be used, for instance, in asphalt that will be used in roads and roofs, or can also be used in shoe soles, increasing the resilience and the elasticity.

The designation used for these laminates are described in the table 3.9.

Table 3.9: Designation of the laminates with Kraton™ and their respective Kraton™ concentration

Designation	Concentration	Mass in each ply
K30	30 g/m^2	2.7 g
K40	40g/m^2	3.6 g
K60	60g/m^2	5.4 g

3.2 Hot Plates Press Curing

After preparing each laminate, the assembled laminates were submited to a curing cycle in a hot plate press. The model of the machine used was SATIN 40370.

In order to cure, the laminate had a curing cycle of 2 hours at 180ºC and 7 bar. The heating had the limitation of a maximum value of 2ºC/min and the cooling was done with water until the temperature reached the ambient temperature keeping the pressure of 7 bar.

3.3 Specimens Preparation

After cooling, the laminates already cured had to be checked to guarantee that the curing process was successful and afterwards, rectified. After rectification, they were measured and the values are shown on table 3.11.

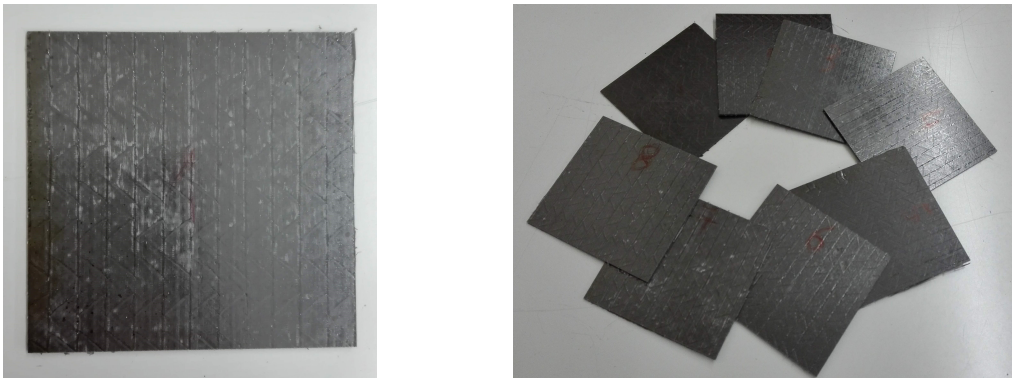
Table 3.10: Measures taken from the laminates after rectification. The thickness value is the average value of the thickness measurements of taken from each side

Laminate	Length 1 [mm]	Length 2 [mm]	Thickness [mm]	Mass [g]
REF1	289.5	290.0	1.04	148.84
C1	292.5	289.5	1.33	164.50
C2	288.0	289.5	2.03	179.88
K30	290.5	289.5	1.28	155.95
K40	297.0	296.0	1.29	161.95
K60	297.0	297.0	1.18	167.80
B10	296.5	297.0	1.09	159.80
B20	297.0	300.5	1.12	164.70
B30	298.0	298.0	1.34	165.20
B40	298.5	297.0	1.39	166.60
REF2	299.0	296.5	1.19	158.50

Using a circular saw, the laminates were cut in specimen shape according with the dimensions defined by the norms, in a scheme that was previously defined. When cut, the specimens were properly cleaned, dried and labeled. With one laminate, it was possible to obtain all the specimens necessary to the mechanical tests that were previously defined. Table 3.11 shows the specimens obtained from 1 laminate and the dimensions for each specimen.

Table 3.11: Dimensions and quantity of the specimens for each test.

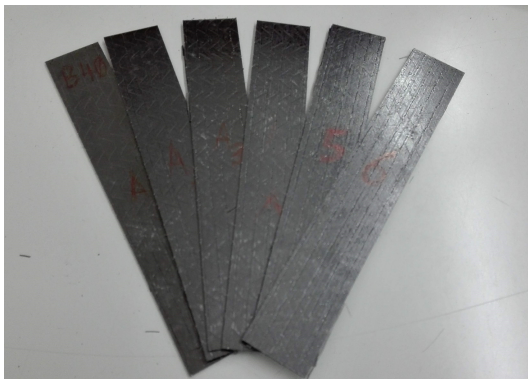
Test	Dimensions	Norm	Quantity	Specimen Photo
Drop-weight impact	60×60	ASTM D5628–96R01	8	Fig. 3.4
Tensile test	250×25	ISO 527-4	4	Fig. 3.6
TAI	150×25	-	6	Fig. 3.5



(a) Impact specimen (b) Set of impact specimens

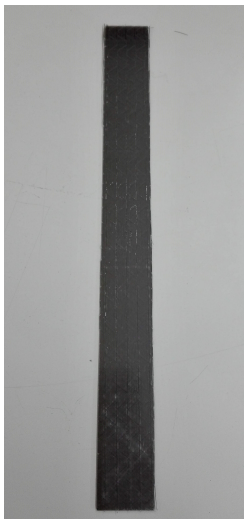
Figure 3.4: Drop-weight impact specimens

(a) TAI specimen

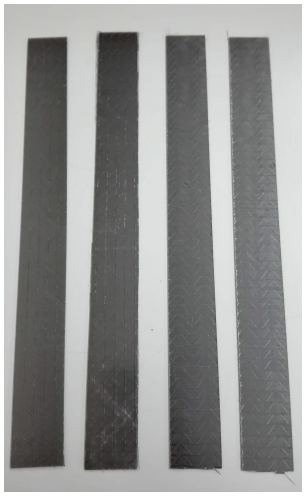


(b) Set of TAI specimens

Figure 3.5: TAI specimens



(a) Tensile test specimen



(b) Set of tensile test specimens: 2 longitudinal and 2 transversal specimens

Figure 3.6: Tensile test specimens

In the case of some mechanical tests, it was not possible to obtain enough specimens due to limitations of laminate size. Also, the TAI specimens didn't follow any norm, since it's a test that is not mentioned in any norm, just in some papers.

3.4 Mechanical Tests

3.4.0.1 Tensile Test

The tensile test performed for this study were based on the norm ISO 527-4[7, 6] using the INSTRON model 4208 machine, located in the laboratory of mechanical tests of INEGI. The tests were performed at a velocity of 2mm/min and type 2 specimens without holes and Fig. 3.6).

In order to obtain all the required values, it was used a strain gauge INSTRON that was able to measure the displacements with precision. This device had a reference distance of 50 mm and a maximum displacement of 5 mm. Also, using the data aquisition of the applied force and the displacements, it was possible to calculate the tensile stress through equation 3.1 and the gauge with equation 3.2. The Young's modulus also comes from the measurements obtained using the slope of the curve stress-strain between 0.05 and 0.25% strain.

$$\sigma = Fbh \quad [MPa] \tag{3.1}$$

being:
F, the applied force [N]
b, the width of the specimen[mm]
h, the thickness of the specimen[mm]

$$\varepsilon = \Delta L_0 L_0 \quad [\%] \tag{3.2}$$

being:
ΔL_0, the displacement increase [mm]
L_0, the distance between the grips of the stain gauge [mm]

3.4.0.2 Low Velocity Impact Test

Regarding the Low Velocity Impact (LVI) test, the norms used were ASTM D 5628-96R01 and ASTM D7136 D7136M - 05[1] and using the ROSAND – Instrumented Falling Weight Impact Tester, Type 5 H.V. machine.

The Falling Weight Impact Tester machine is composed of 3 parts: a tower, where the weight is dropped, a control unit and a computer for data acquisition. The tester machine has also a system that "grabs" the impactor after the first impact, in order to not allow multiple impacts (Fig 3.7b)

(a) Detailed view of the Impact Tester (b) Pneumatic impact "grabbing" system

Figure 3.7: ROSAND – Instrumented Falling Weight Impact Tester, Type 5 H.V.

It was chosen an hemispherical impactor with 16 mm diameter and it was also chosen to use the available pneumatic support that clamps the specimen.

The impact energies used were 5, 8 and 13 J for each interlayer toughening hypothesis. This energy values are possible controlling the mass of the set impactor mass + additional mass and the drop height.

3.4.0.3 Indentation Tests

Using the impacted specimens, it was also possible to measure the dent depth after the impact and at certain times after the test to see if there was some restitution of this value. The times chosen were: right after the impact, 24 hours later, 1 week later and 1 month later.

These values were measured by using an analog dial indicator with a 100 mm range and 0.01 mm precision.

In order to collect these indications, 4 measures were taken around the impacted area of the specimen to obtain the average "zero" level of the non deformed surface and another measurement on the lowest point of the final dent depth. The indentation was the "zero" value minus the lowest point.

3.4.0.4 Tensile After Impact (TAI) Test

Tensile stress after impact test[17] is an effective test to measure the residual properties after an impact. It was chosen this test instead of CAI (Compression after Impact)[2] due to the fact that TAI specimens are smaller, ending up occupying a shorter area on the laminate. It consists, as the name says, performing a tensile stress after the specimen had suffered an impact (of low energy, in this situation).

The impact test was really similar to the one described on 3.4.0.2, but for this test, it was used an hemispherical impact with 7 mm diameter and using energy of 2.5, 3.5 and 5.0. Regarding the tensile tests, they were pretty similar to the ones described on section 3.4.0.1.

Chapter 4

Tests Results, Analysis and Discussion

4.1 Tensile Tests

In the context of this study, this test had the purpose to characterize the laminates, giving informations about the mechanical properties of the different laminate solutions, and understand the influence of the each interlayer material on those properties. This test is also useful, because afterwards it is possible to compare these properties with the impacted equivalent specimens, evaluating the loss of mechanical properties. In order to make this mechanical test, each laminate have 4 specimens: 2 longitudinal (L) and 2 transversal (T), in order to assess if the fibres' direction had any influence on the properties studied here, despite the fact that all the laminates were balanced. Since there was two reference laminates, it was possible to obtain six more specimens (3 longitudinal and 3 transversal). With these two reference laminates, it is also possible to see if the properties change considerably with the production of the laminates.

4.1.1 Reference - REF1 and REF2

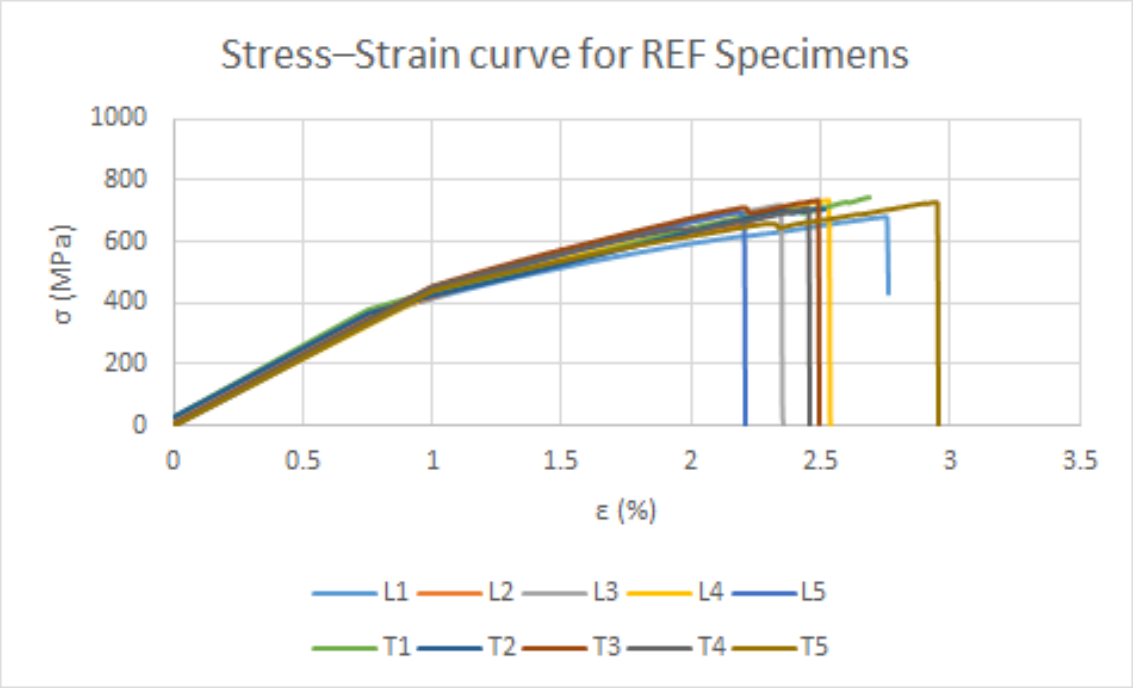


Figure 4.1: Stress–Strain curve for REF Specimens

Table 4.1: Dimensions and mechanical properties of REF tensile tests' specimens

Specimen	**Width b [mm]**	**Thickness h [mm]**	**UTS [MPa]**	**Young's Modulus E [GPa]**	**UTS [MPa]**	**Young's Modulus E [GPa]**
L1	24.60	1.20	680	46.2		
L2	24.50	1.17	661	45.4		
L3	24.72	1.25	723	44.4	699	44.7
L4	24.72	1.22	734	43.6		
L5	24.75	1.22	696	43.7		
T1	24.68	1.15	748	46.7		
T2	24.78	1.17	704	45.1		
T3	24.92	1.15	732	45.4	725	45.3
T4	24.68	1.18	715	45.5		
T5	24.78	1.20	728	44.1		
Average	24.64	1.21	699	44.9		
Standard deviation	0.09	0.03	30	1.0		

4.1.2 Thin Cork Film - C1

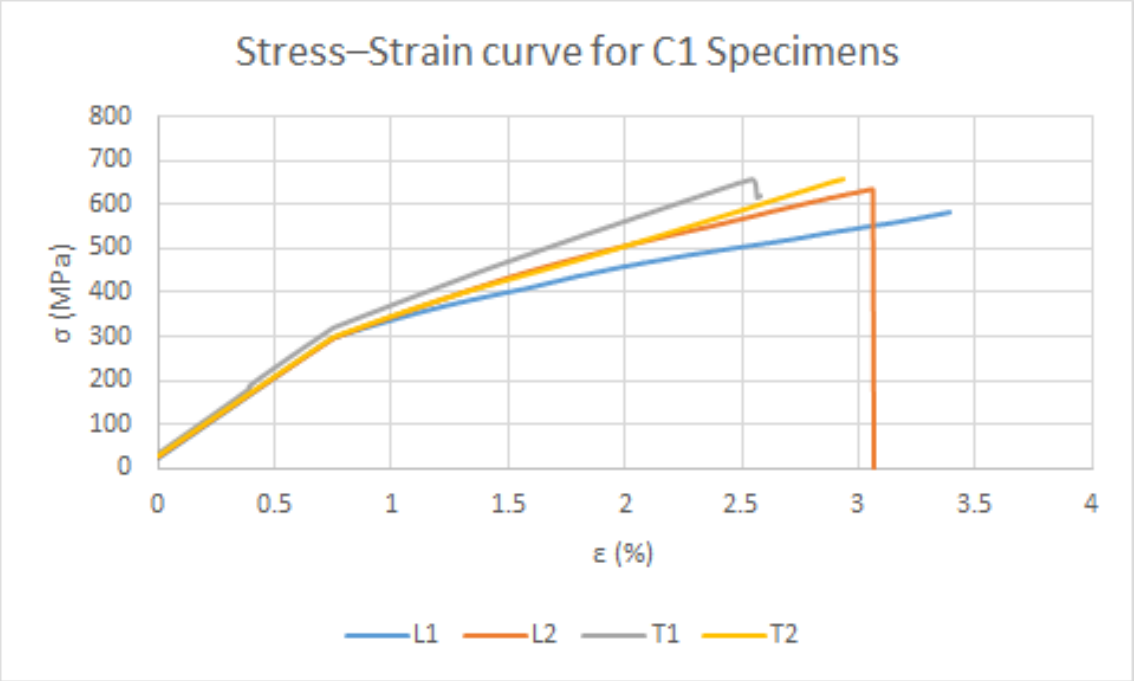


Figure 4.2: Tensile test of C1 specimens

Table 4.2: Dimensions and mechanical properties of C1 tensile tests' specimens

Specimen	**Width b [mm]**	**Thickness h [mm]**	**UTS [MPa]**	**Young's Modulus E [GPa]**
L1	24.77	1.45	584	36.6
L2	24.77	1.45	633	36.2
T1	24.82	1.45	659	37.5
T2	25.03	1.45	658	36.4
Average	24.85	1.45	634	36.6
Standard deviation	0.11	0.01	30	0.5

4.1.3 Thick Cork Film - C2

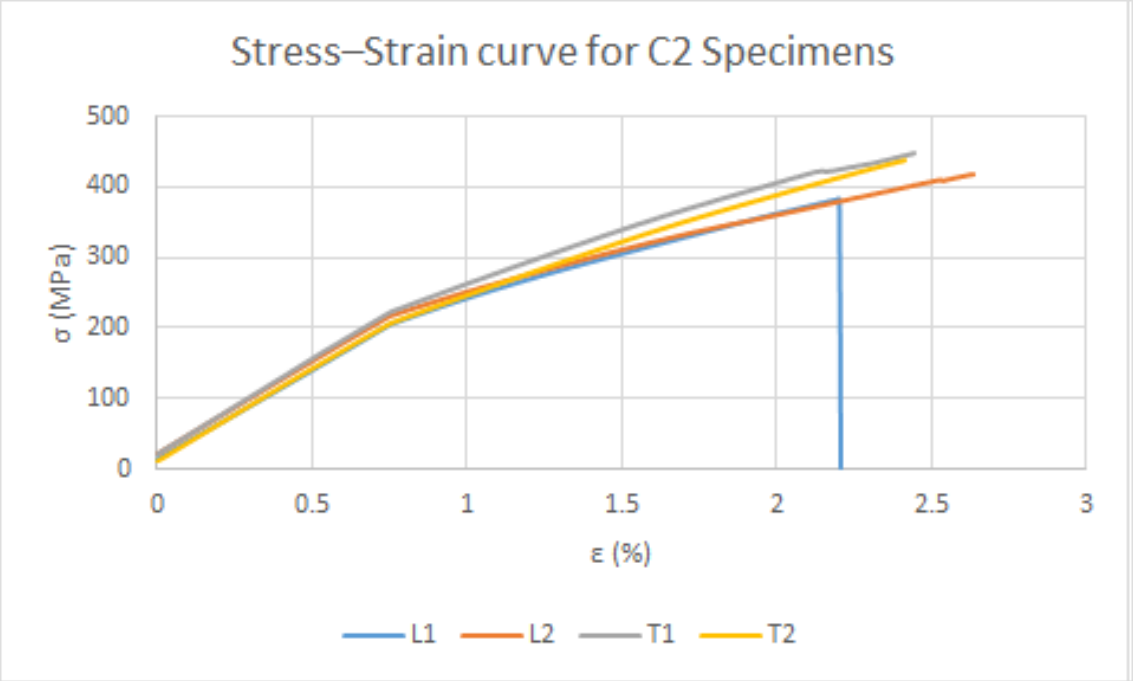


Figure 4.3: Tensile test of C2 specimens

Table 4.3: Dimensions and mechanical properties of C2 tensile tests' specimens

Specimen	**Width b [mm]**	**Thickness h [mm]**	**UTS [MPa]**	**Young's Modulus E [GPa]**
L1	24.72	2.00	383	25.3
L2	24.77	1.98	417	26.2
T1	24.72	1.88	448	27.2
T2	24.76	1.95	439	26.4
Average	24.74	1.95	422	27.2
Standard deviation	0.02	0.05	25	0.7

4.1.4 Kraton™ granules 30 g/m^2 - K30

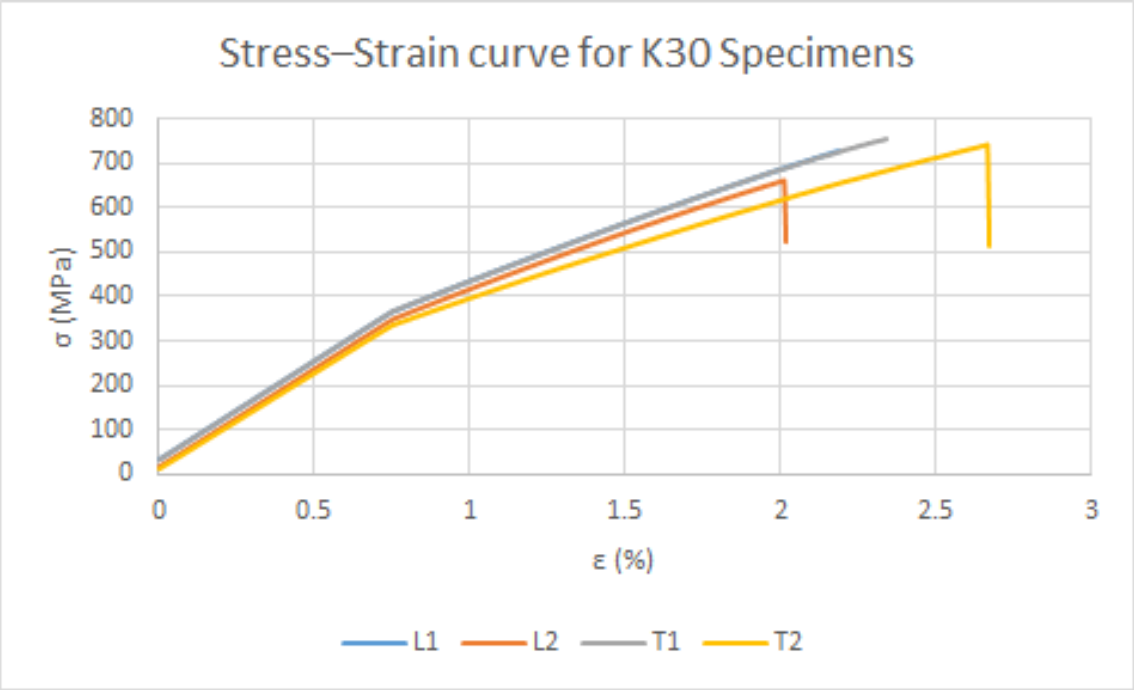


Figure 4.4: Tensile test of K30 specimens

Table 4.4: Dimensions and mechanical properties of K30 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus E [GPa]
L1	24.72	1.20	725	44.1
L2	24.80	1.20	662	44.0
T1	24.80	1.22	753	44.4
T2	24.77	1.25	741	43.1
Average	24.77	1.22	720	43.9
Standard deviation	0.03	0.02	35	0.5

4.1.5 Kraton™ granules 40 g/m^2 - K40

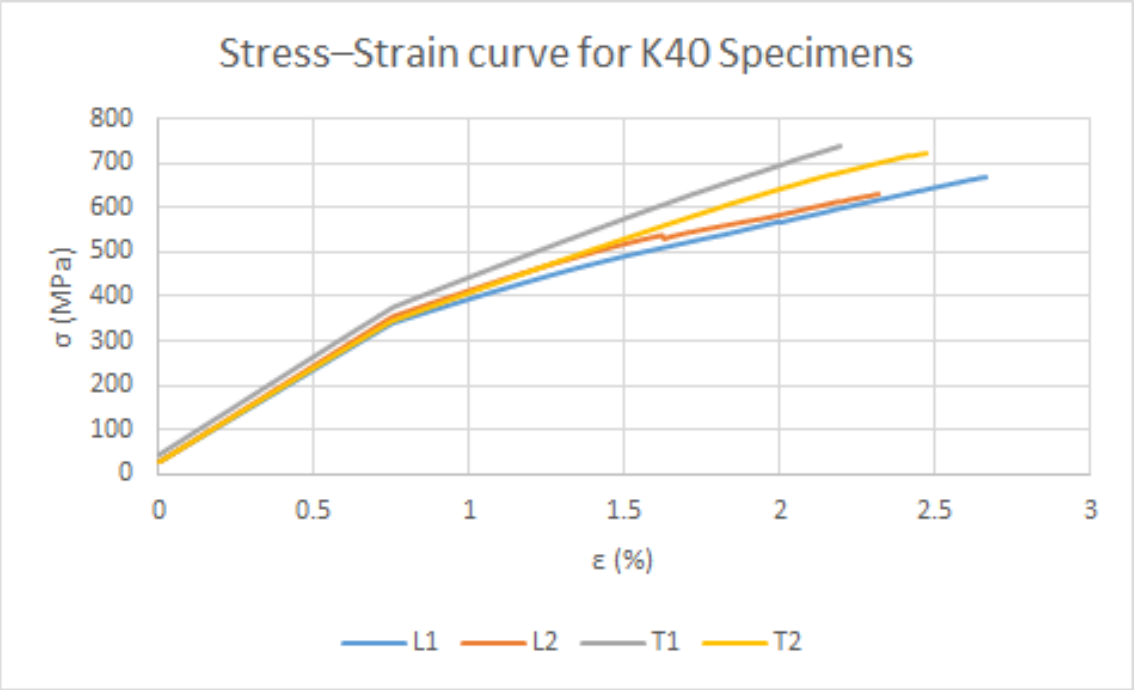


Figure 4.5: Tensile test of K40 specimens

Table 4.5: Dimensions and mechanical properties of K40 tensile tests' specimens

Specimen	**Width b [mm]**	**Thickness h [mm]**	**UTS [MPa]**	**Young's Modulus E [GPa]**
L1	24.73	1.30	667	41.4
L2	24.72	1.25	630	43.2
T1	22.23	1.22	738	44.2
T2	24.78	1.27	723	42.5
Average	24.12	1.26	690	42.8
Standard deviation	1.09	0.03	43	1.01

4.1.6 Kraton™ granules 60 g/m^2 - K60

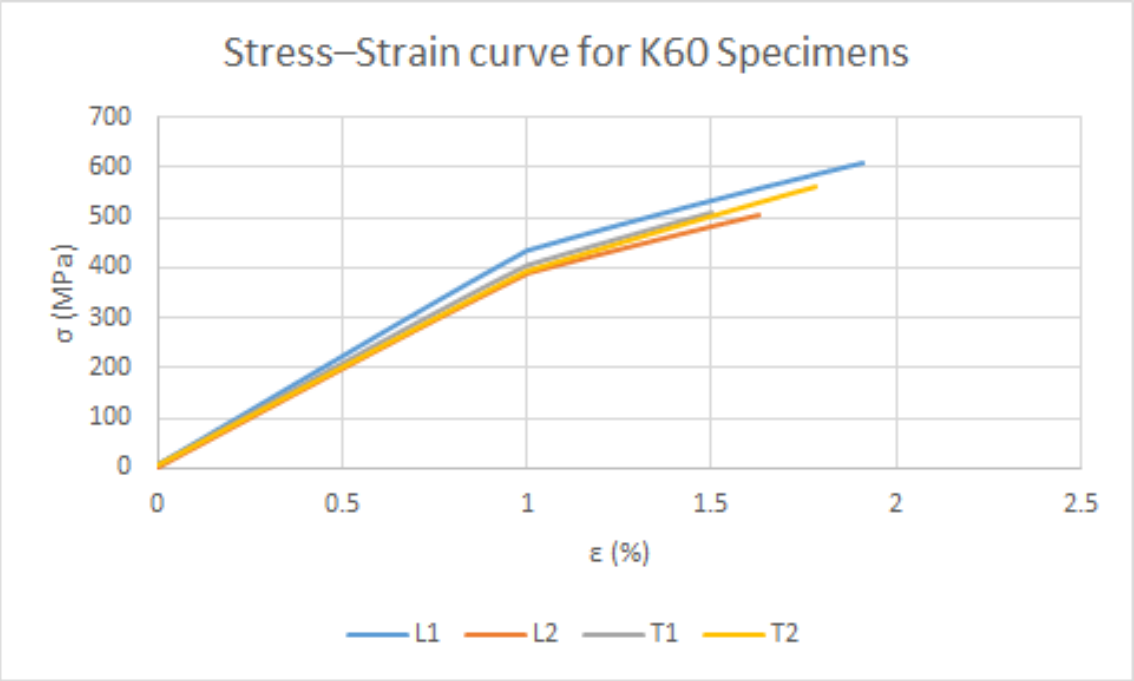


Figure 4.6: Tensile test of K60 specimens

Table 4.6: Dimensions and mechanical properties of K60 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus E [GPa]
L1	25.13	1.30	701	43.0
L2	25.12	1.32	627	39.7
T1	25.13	1.25	688	40.7
T2	25.18	1.30	692	39.3
Average	25.14	1.29	677	40.7
Standard deviation	0.02	0.03	29	1.4

4.1.7 Expanded cork granules 10 g/m^2 - B10

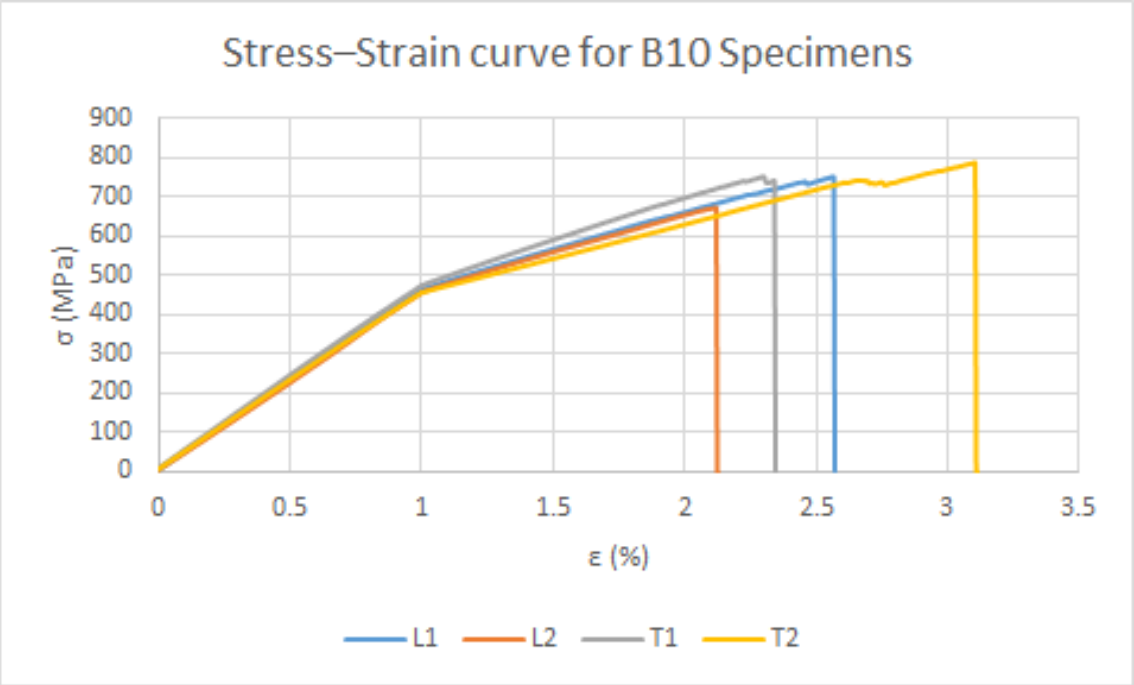


Figure 4.7: Tensile test of B10 specimens

Table 4.7: Dimensions and mechanical properties of B10 tensile tests' specimens

Specimen	**Width b [mm]**	**Thickness h [mm]**	**UTS [MPa]**	**Young's Modulus E [GPa]**
L1	25.13	1.22	750	46.3
L2	25.13	1.22	670	44.0
T1	25.13	1.13	749	47.4
T2	25.18	1.15	787	45.4
Average	25.14	1.88	739	45.7
Standard deviation	0.02	0.04	42	1.2

4.1.8 Expanded cork granules 20 g/m^2 - B20

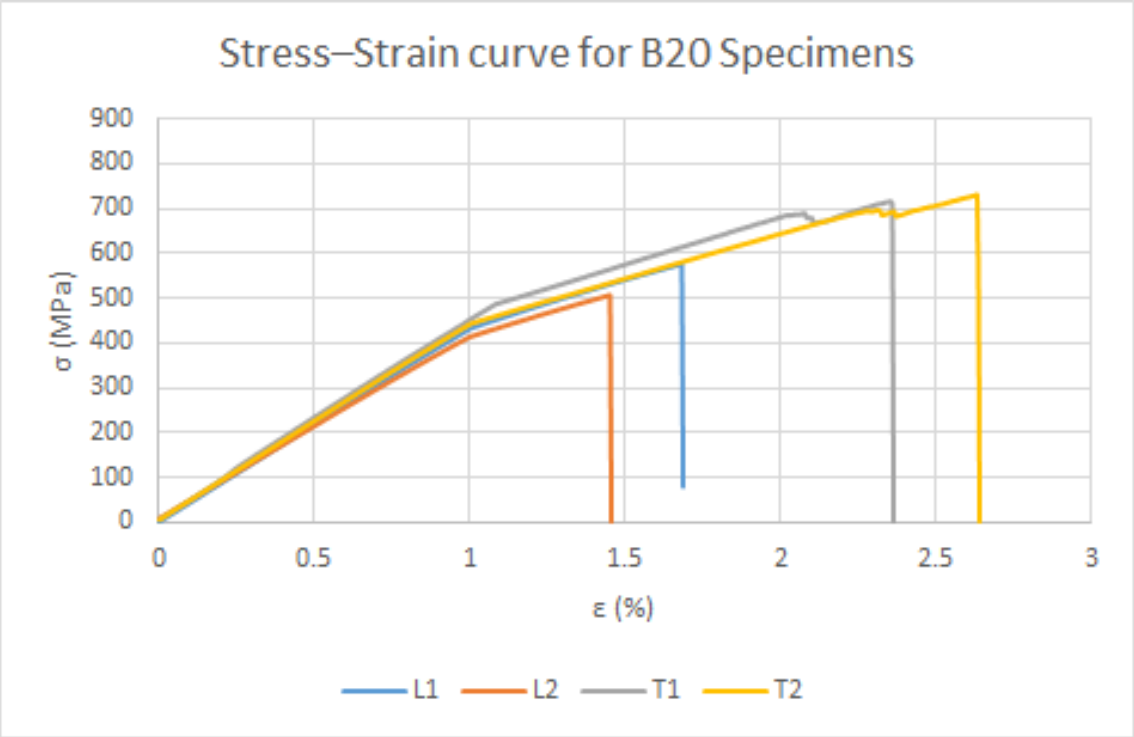


Figure 4.8: Tensile test of B20 specimens

Table 4.8: Dimensions and mechanical properties of B20 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus E [GPa]
L1	24.55	1.23	577	43.6
L2	25.08	1.27	509	41.5
T1	24.90	1.18	718	45.6
T2	24.90	1.20	733	45.6
Average	25.86	1.22	634	44.1
Standard deviation	0.19	0.03	94	1.7

4.1.9 Expanded cork granules 30 g/m^2 - B30

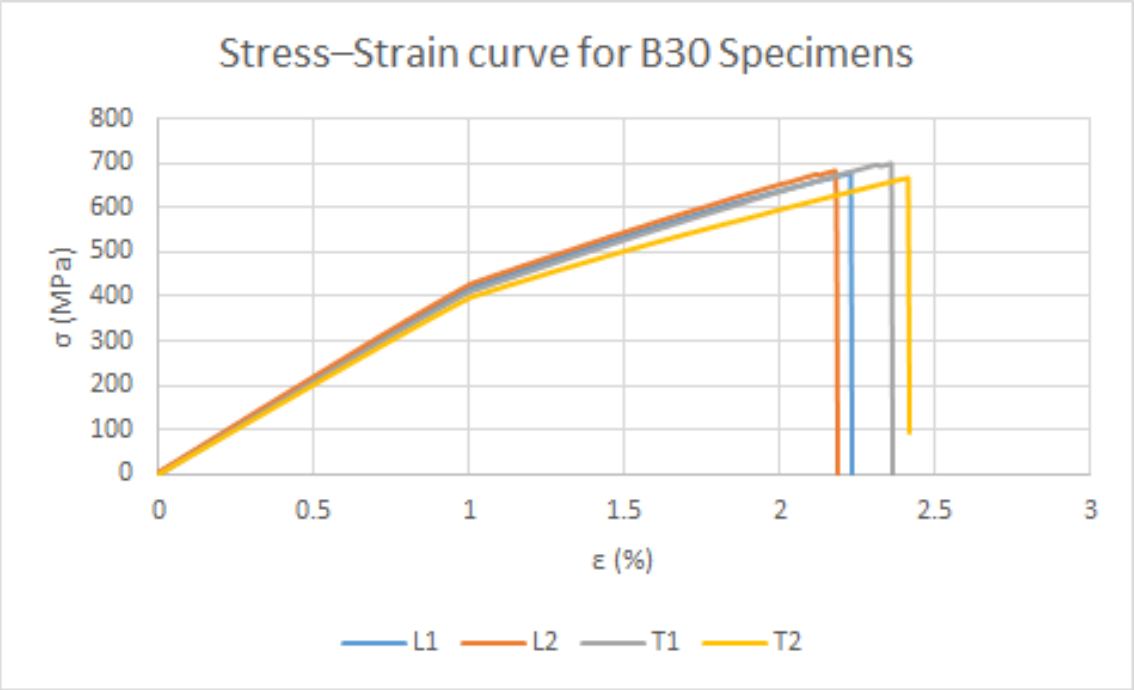


Figure 4.9: Tensile test of B30 specimens

Table 4.9: Dimensions and mechanical properties of B30 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus E [GPa]
L1	24.67	1.28	680	42.2
L2	24.70	1.28	681	43.3
T1	24.63	1.25	701	41.9
T2	24.70	1.30	668	40.4
Average	25.68	1.28	683	41.9
Standard deviation	0.03	0.02	12	1.04

4.1.10 Expanded cork granules 40 g/m^2 - B40

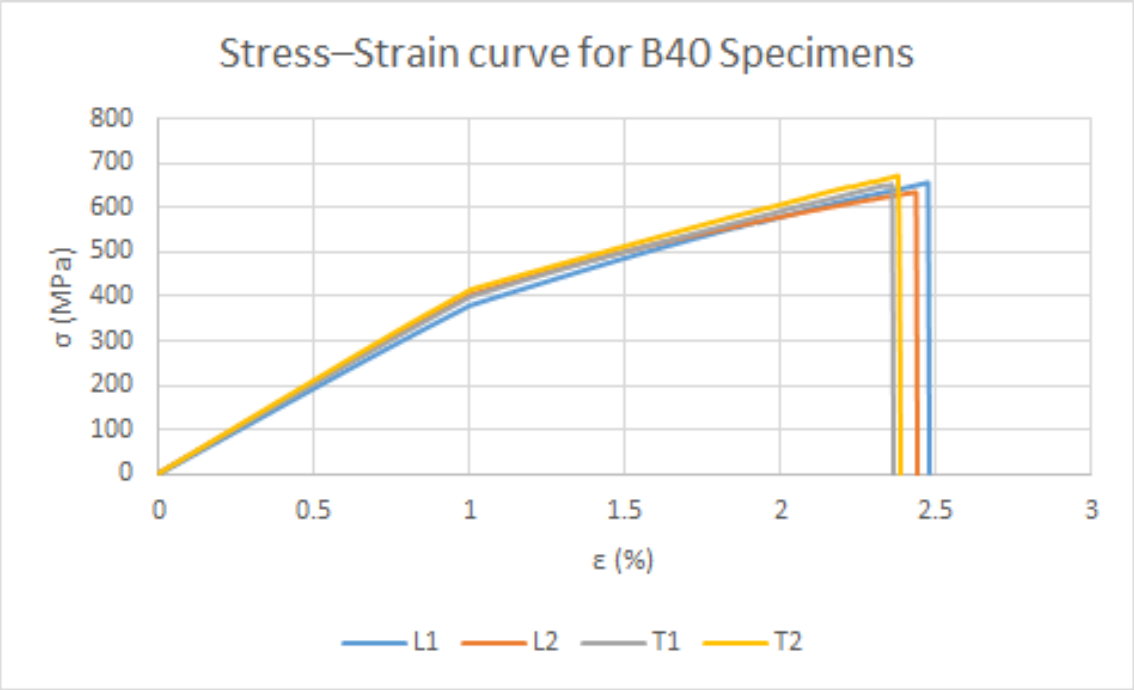


Figure 4.10: Tensile test of B40 specimens

Table 4.10: Dimensions and mechanical properties of B30 tensile tests' specimens

Specimen	**Width b [mm]**	**Thickness h [mm]**	**UTS [MPa]**	**Young's Modulus E [GPa]**
L1	24.78	1.28	680	42.2
L2	24.75	1.28	681	43.3
T1	24.72	1.25	701	41.9
T2	24.47	1.30	668	40.4
Average	24.68	1.33	654	40.5
Standard deviation	0.12	0.05	13	1.3

4.1.11 Analysis of Results

In this section, there is a global analysis of the test results. REF's results will be compared with the different groups of interlayer material and in the end, a comparison between all of them will be made.

An initial comment can be made concerning the tensile-strain curves shown. It is possible to see that in all curves of tensile tests (and also TAI tests), a sudden change in the slope. This is not due to the materials' behaviour, but due to removal of the strain gauge, the strain began to be measured from the displacement of the grips. The testing machine is capable to make a correction of the slope, but due to distance, some slippage and other phenomena, this variation is not overcomed.

Comparison between C1, C2 and REF

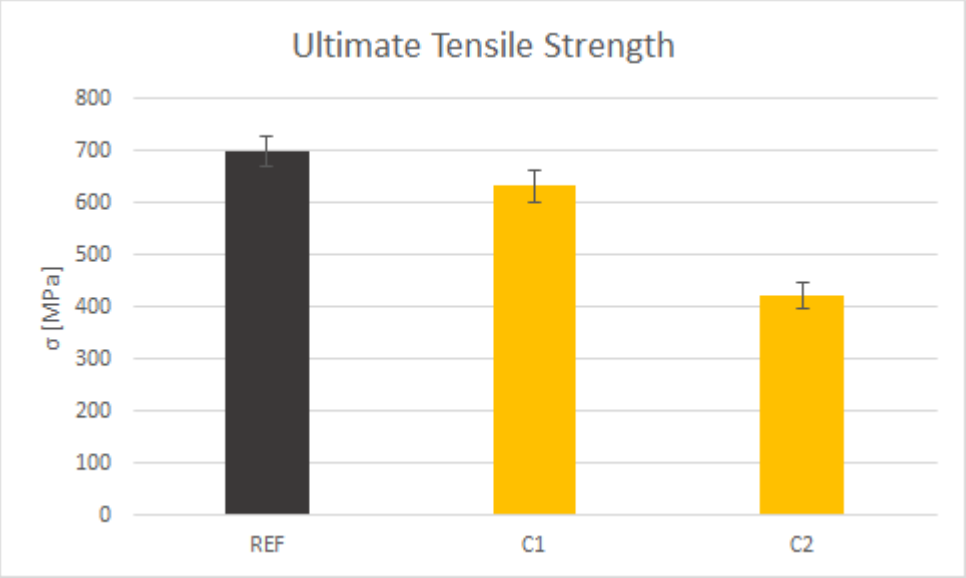


Figure 4.11: Ultimate Tensile Strengths' comparison between C1, C2 and REF

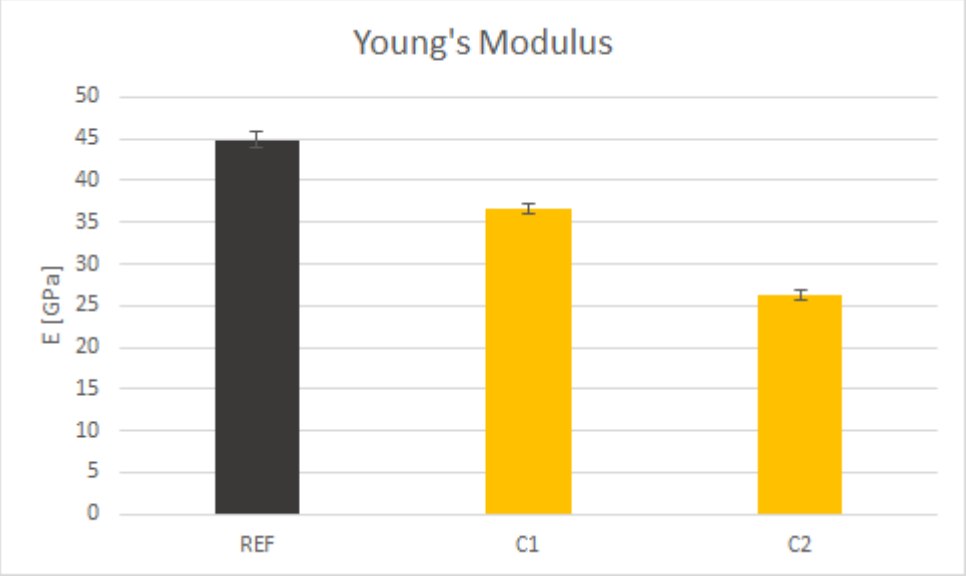


Figure 4.12: Young's modulus' comparison between C1, C2 and REF

Table 4.11: Mechanical properties of C1 and C2 and their reduction

Laminate	UTS [MPa] Average	UTS [MPa] Standard Deviation	E [GPa] Average	E [GPa] Standard Deviation	UTS Reduction [%]	E Reduction [%]
REF	699	30	44.9	1.0	-	-
C1	634	30	36.6	0.5	9%	18%
C2	422	25	26.23	0.7	40%	42%

The results from figure 4.11, 4.12 and table 4.14, show a sharp reduction of the mechanical properties, more specifically: on C1 the reduction is 9% for the UTS and 18% for the Young's modulus and regarding C2, there is a reduction of 40% on the strength and 42% on the Young's modulus. These results lead to the conclusion that the thicker the cork film, higher is the reduction of the mechanical properties, verification that was already expected since 8% of C1's thickness and 45% is cork.

Assessing the distribution of the values, it is possible to say that they are consistent. This verification is supported by the standard deviation of the values, which are relatively low, specially the Young's modulus ones.

Comparison between K30, K40, K60 and REF

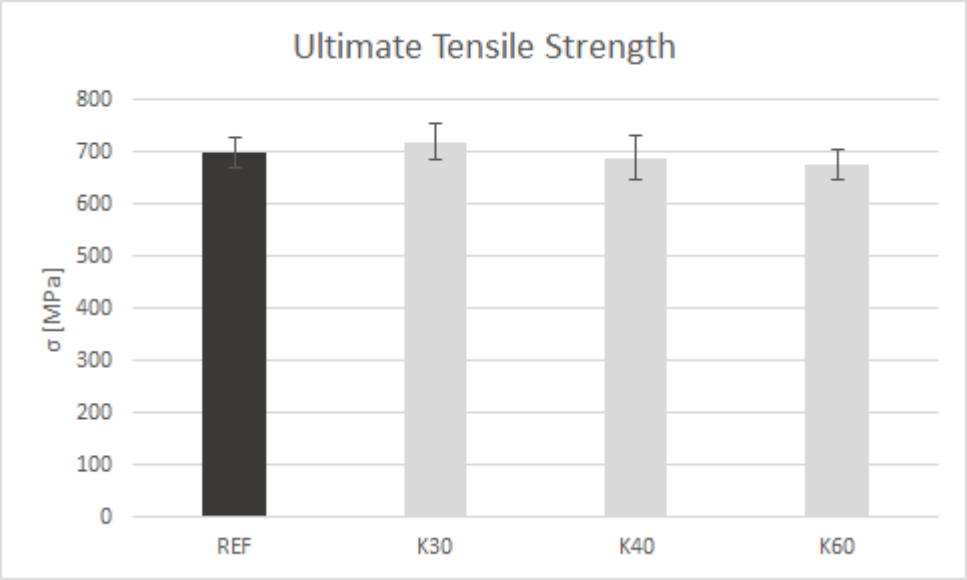


Figure 4.13: Ultimate Tensile Strengths' comparison between K30, K40, K60 and REF

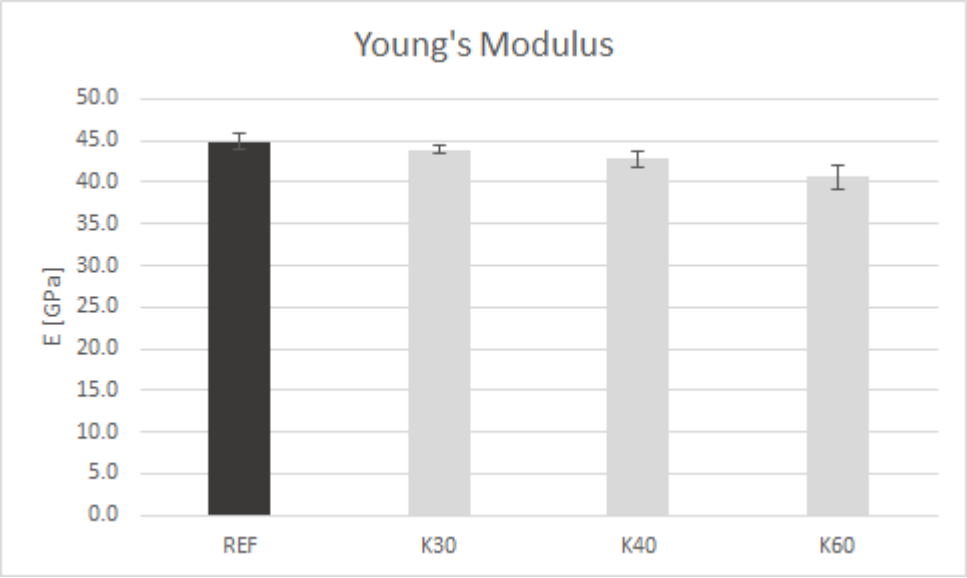


Figure 4.14: Young's modulus' comparison between K30, K40, K60 and REF

Table 4.12: Mechanical properties of K30, K40 and K60 and their reduction

Laminate	UTS [MPa] Average	UTS [MPa] Standard Deviation	E [GPa] Average	E [GPa] Standard Deviation	UTS Reduction [%]	E Reduction [%]
REF	699	35	44.9	1.0	-	-
K30	702	35	43.9	0.5	-3%	2%
K40	690	43	42.8	1.0	1%	5%
K60	677	29	40.7	1.4	3%	9%

In the case of Kraton™ granules, the reduction of mechanical properties is quite low (Table 4.12). Analysing K30 values, it is possible to see that the concentration is so low, that the results for tensile strength are higher than the reference, leading to believe that the usage of a small concentration might enhance the ultimate tensile strength. This might have happened due to the fact that this amout of granules makes the laminate more ductile, and although the Young's modulus is lower, the K30 laminate might reach a higher UTS than the reference, but with higher strain. Looking now at K60's results, the values point out that for a certain concentration, the decrease is considerable.

Once again, the standard deviation values are small, which leads to the conclusion that the values are consistent, specially on what Young's modulus is concern, although the previous results (cork films) are even more consistent.

Comparison between B10, B20, B30, B40 and REF

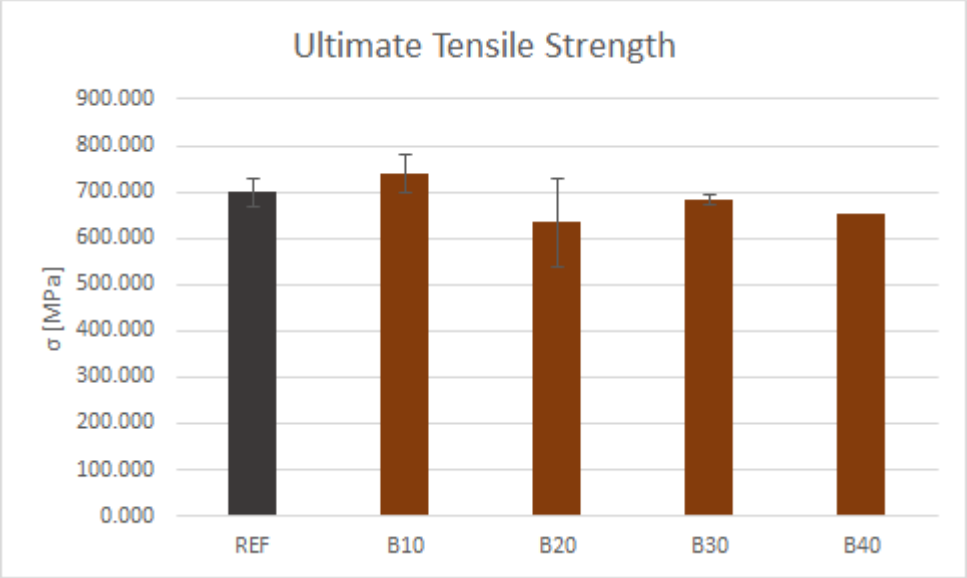


Figure 4.15: Ultimate Tensile Strengths' comparison between B10, B20, B30, B40 and REF

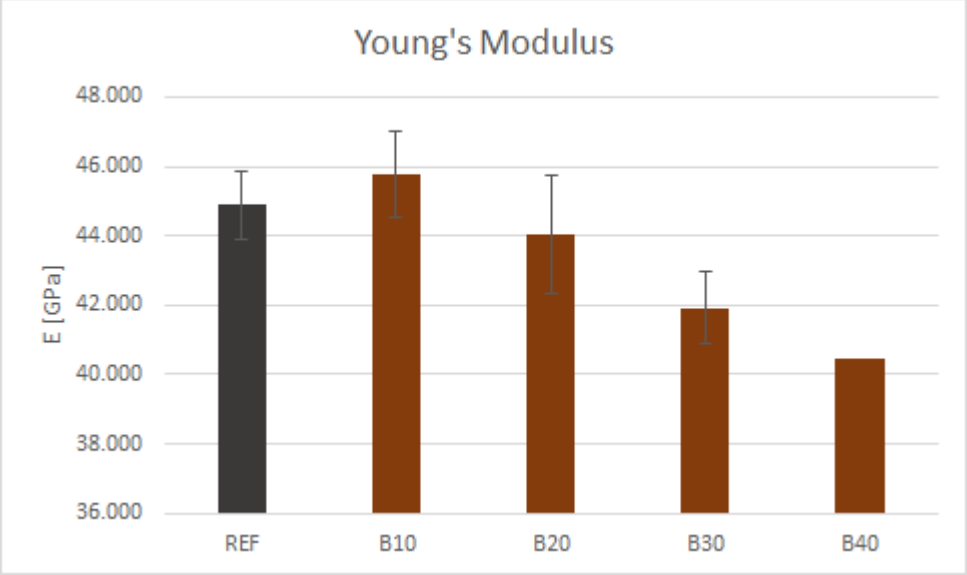


Figure 4.16: Young's modulus' comparison between B10, B20, B30, B40 and REF

The usage of expanded cork granules also leads to a small reduction of mechanical properties as it is possible to see by the table 4.13, figure 4.15 and 4.16. These results also support the verification made on the previous section, when mentioning that, for small concentrations of granules, there is an enhancement of the mechanical properties (UTS), which in this case happens on the B10 laminate. This might have happened for the same reason as K30, or also due to the dispersion, which is probably not homogeneous due to the spreading technique used and the small amount of

Table 4.13: Mechanical properties of B10, B20, B30, B40 and their reduction

Laminate	UTS [MPa] Average	UTS [MPa] Standard Deviation	E [GPa] Average	E [GPa] Standard Deviation	UTS Reduction [%]	E Reduction [%]
REF	699	35	44.9	1.0	-	-
B10	739	42	45.7	1.2	-6%	-2%
B20	634	94	44.1	1.7	9%	2%
B30	683	12	41.9	1.0	2%	7%
B40	654	13	40.5	1.3	7%	10%

these granules. Besides, looking at the experimental error, the resuls can be explained exactly as what happened on K30.

Concerning the values' distribution, it is clear that they are consistent, except the B20's values, which for UTS, shows a standard deviation of 94 MPa. This value can be explained by some phenomena that might have happened during the fabrication of the laminates, for instance, the non-homogeneous distribution of the cork granules throughout the surface. Anyway, if the experimental error is taken into consideration, the resuls make sence according to what was expected: the higher is the amount of these granules' concentration, the lower are the mechanical properties (comparing with the reference).

Comparison between all

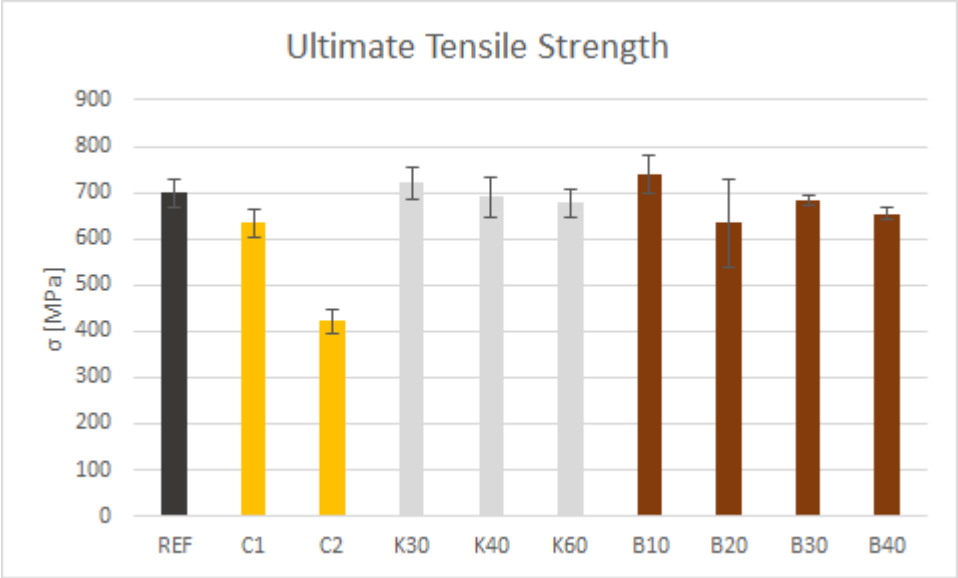


Figure 4.17: Ultimate Tensile Strengths' comparison between all laminates

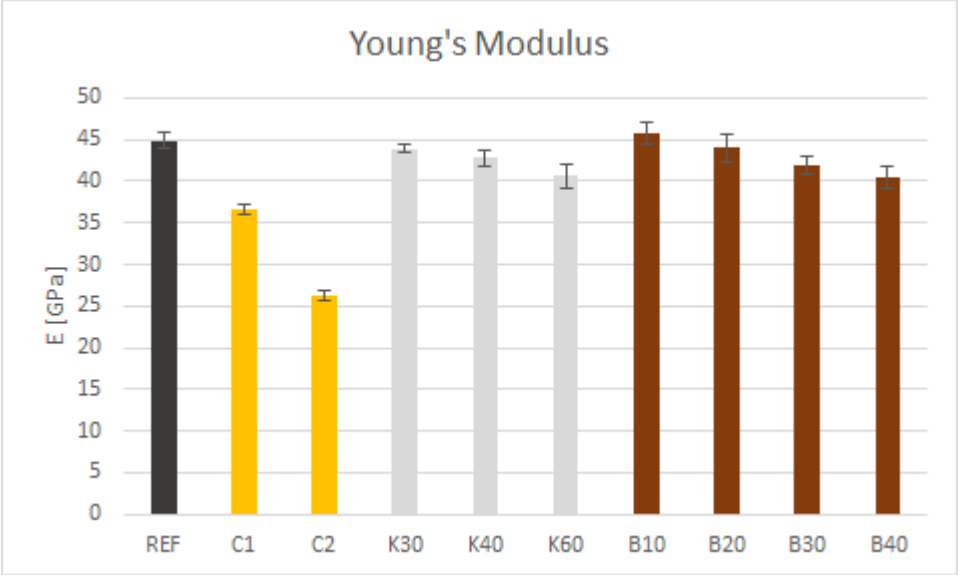


Figure 4.18: Young's modulus' comparison between all laminates

Analysing Fig 4.17 and 4.18, the graphs stand out that, using a cork film highly reduces the mechanical properties of the laminate, showing the lowest values for both ultimate tensile strength and Young's modulus. In case of C2, which is the lower, the properties almost decrease for half, when compared with the reference.

For both Kraton™ and expanded cork granules, it is possible to see that, the higher is the concentration of this granules, the higher is the loss of mechanical properties. It is also important to

Table 4.14: Mechanical properties of C1 and C2 and their reduction

Laminate	UTS [MPa] Average	UTS [MPa] Standard Deviation	E [GPa] Average	E [GPa] Standard Deviation	UTS Reduction [%]	E Reduction [%]
REF	699	30	44.9	1.0	-	-
C1	634	30	36.6	0.5	9%	18%
C2	422	25	26.2	0.7	40%	42%
K30	702	35	43.9	0.5	-3%	2%
K40	690	43	42.8	1.0	1%	5%
K60	677	29	40.7	1.4	3%	9%
B10	739	42	45.7	1.2	-6%	-2%
B20	634	94	44.1	1.7	9%	2%
B30	683	12	41.9	1.0	2%	7%
B40	654	13	40.5	1.3	7%	10%

save the possibility that some value of concentration between the enhancement and the decrease of properties, changes this conclusion, following a non-linear behaviour and thus, having an optimum concentration value.

Still about Kraton™ and expanded cork granules, the usage of these solutions have lead to a decrease of properties that was not so meaningful. It is even hard to decide which set of interlayer material lead to better properties, since it depends on the concentration and on other variables. For the same concentration, the "K group" lead to lower reduction (both on the modulus and strength), although this analysis is not completely accurate, since the materials mentioned have different specific mass values, and some other phenomena might disapprove this verification.

Looking also at the tables 4.1, 4.2, 4.3, 4.4, 4.5, 4.6, 4.7, 4.8, 4.9 and 4.10, it is possible to see that there is just a small difference between the longitudinal and transversal specimens, in some cases. This leads to the conclusion that, and since the laminates are balanced, there is not an influence on the orientation of the specimens. The small differences might be explained with some misalignments of the fibres' direction, making the laminate slightly unbalanced.

4.2 Low Velocity Impact Tests

The purpose of this test, in the context of this study, was to assess the impact properties of the laminates, to later compare the different solutions. It was given particular attention to the maximum impact force, deflection and absorbed energy. Since the main goal of this project is to study the damage tolerance, the energy absorbed will be of high importance, due to the fact that a laminate is damage tolerant until it is capable to dissipate elastically the energy that it was subjected to. In order to go a bit deeper on this topic, it was calculated the energy recovery rate, that will give a concrete value for the damage tolerance. This quantity is calculated according with equation 4.1.

$$EnergyRecoveryRate = \frac{E_{max} - E}{E_{max}} \tag{4.1}$$

being:
E_{max}, the peak energy
E, the final energy

This section will start by presenting the main information collected from impacts of 5, 8 and 13 J from all specimens, grouped by type of laminate, to latter discuss the differences among them. For each laminate, 8 specimens were used.

The specimens under this study had the designation of: ABC - X-Y. ABC stand for the laminate type, X for the specimen number and Y for the energy that the specimen was subjected.

4.2.1 Reference - REF

Table 4.15: Values from REF Specimens' impact test

Impact Energy	Specimen	Impact Velocity	Peak Force [N]	Peak Deflection [mm]	Final Deflection [mm]	Peak Energy [J]	Final Energy [J]
5 J	4	1.324	2365.59	4.65	2.41	5.33	4.48
	5	1.319	2378.24	4.45	2.23	5.27	4.22
	6	1.327	2089.97	4.61	2.44	5.34	4.44
8 J	1	1.677	2369.39	6.67	5.12	8.49	8.06
	2	1.677	2631.11	6.47	4.47	8.43	7.89
	3	1.668	2443.98	6.88	5.17	8.43	7.98
13 J	7	2.133	2473.06	12.56	12.53	13.84	12.53
	8	2.126	2524.90	13.53	13.53	13.80	13.53

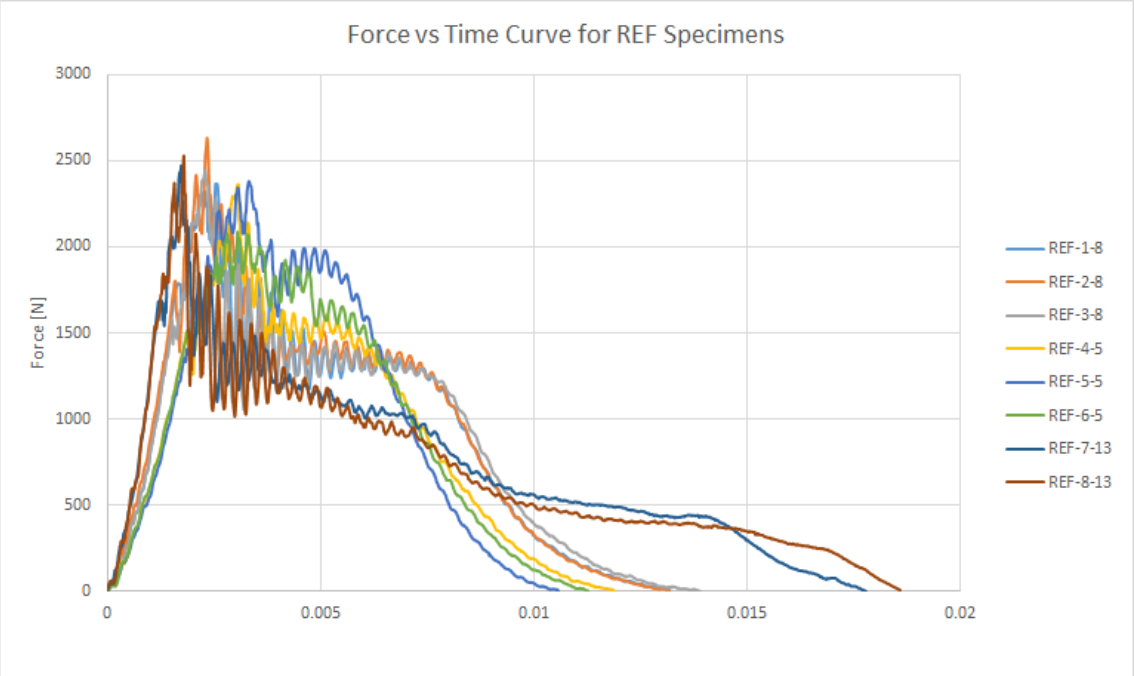


Figure 4.19: Impact's Force vs Time curve REF specimens

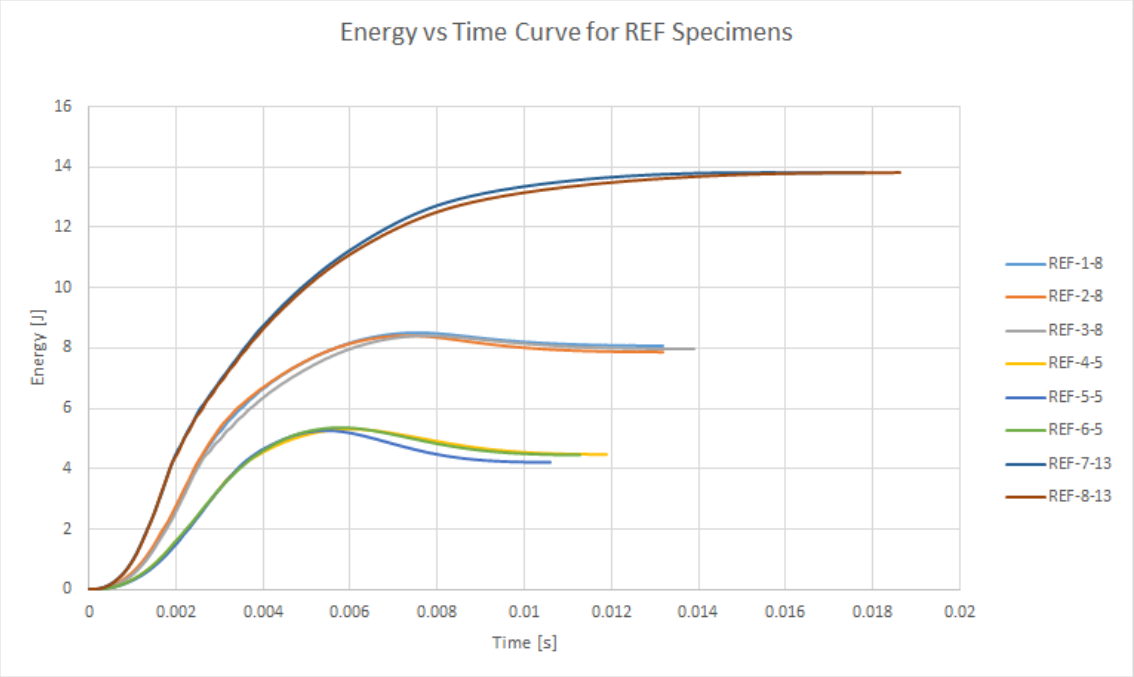


Figure 4.20: Impact's Energy vs Time curve REF specimens

4.2.2 Thin Cork Film - C1

Table 4.16: Values from C1 Specimens' impact test

Impact Energy	Specimen	Impact Velocity	Peak Force [N]	Peak Deflection [mm]	Final Deflection [mm]	Peak Energy [J]	Final Energy [J]
5 J	4	1.317	2885.24	4.13	1.71	5.24	3.44
	5	1.317	2911.79	4.15	1.73	5.24	3.33
	6	1.318	2961.10	4.16	1.70	5.25	3.29
8 J	1	1.667	3198.80	5.85	3.85	8.34	7.69
	2	1.665	3270.87	5.35	3.54	8.30	7.69
	3	1.670	2689.27	5.35	3.99	8.39	7.74
13 J	7	2.121	2796.74	10.01	8.53	13.55	13.34
	8	2.128	3235.46	9.43	8.01	13.60	13.37

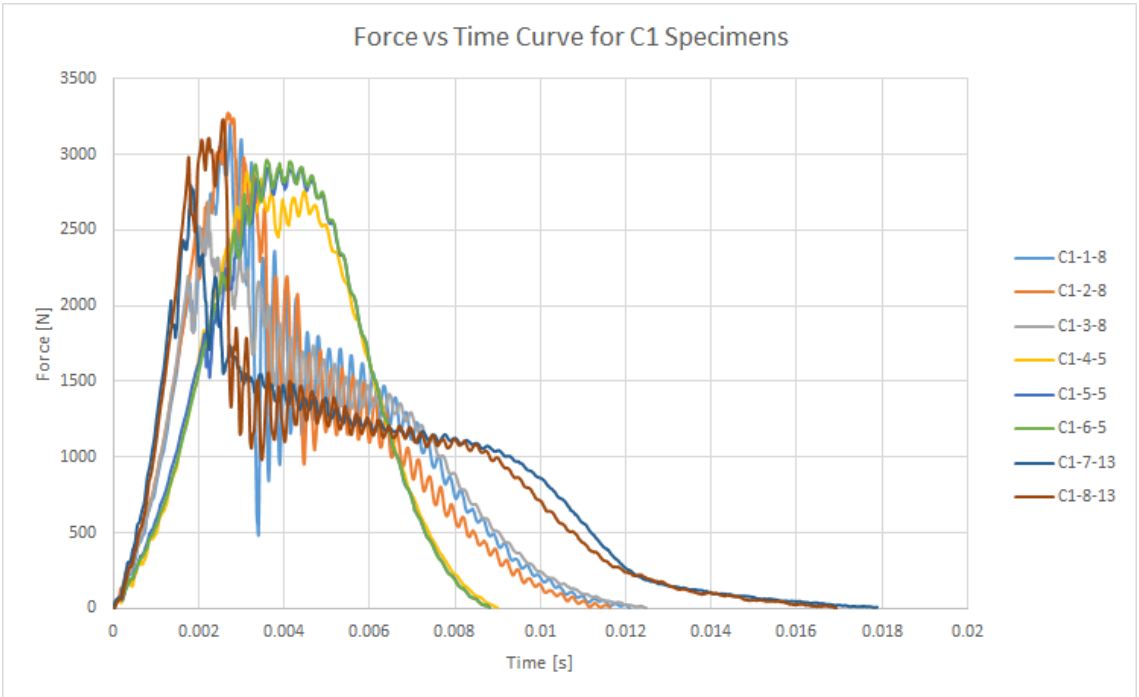


Figure 4.21: Impact's Force vs Time curve C1 specimens

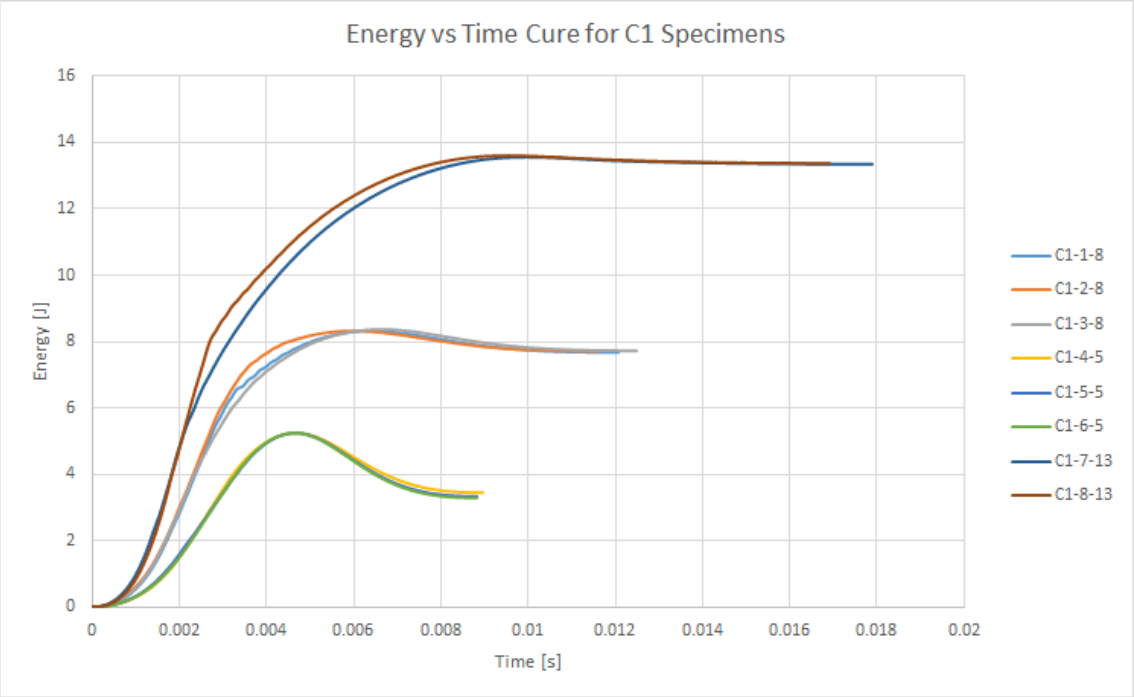


Figure 4.22: Impact's Energy vs Time curve C1 specimens

4.2.3 Thick Cork Film - C2

Table 4.17: Values from C1 Specimens' impact test

Impact Energy	Specimen	Impact Velocity	Peak Force [N]	Peak Deflection [mm]	Final Deflection [mm]	Peak Energy [J]	Final Energy [J]
5 J	4	1.322	3307.53	4.37	2.00	5.29	3.11
	5	1.323	3640.05	4.22	1.63	5.29	2.70
8 J	1	1.674	3732.35	5.38	3.18	8.40	7.20
	2	1.673	3942.23	5.24	3.08	8.37	6.83
	3	1.671	3781.66	5.38	3.46	8.37	7.23
13 J	6	2.144	4126.83	8.08	6.43	13.72	13.30
	7	2.137	3728.56	7.63	5.36	13.61	12.81

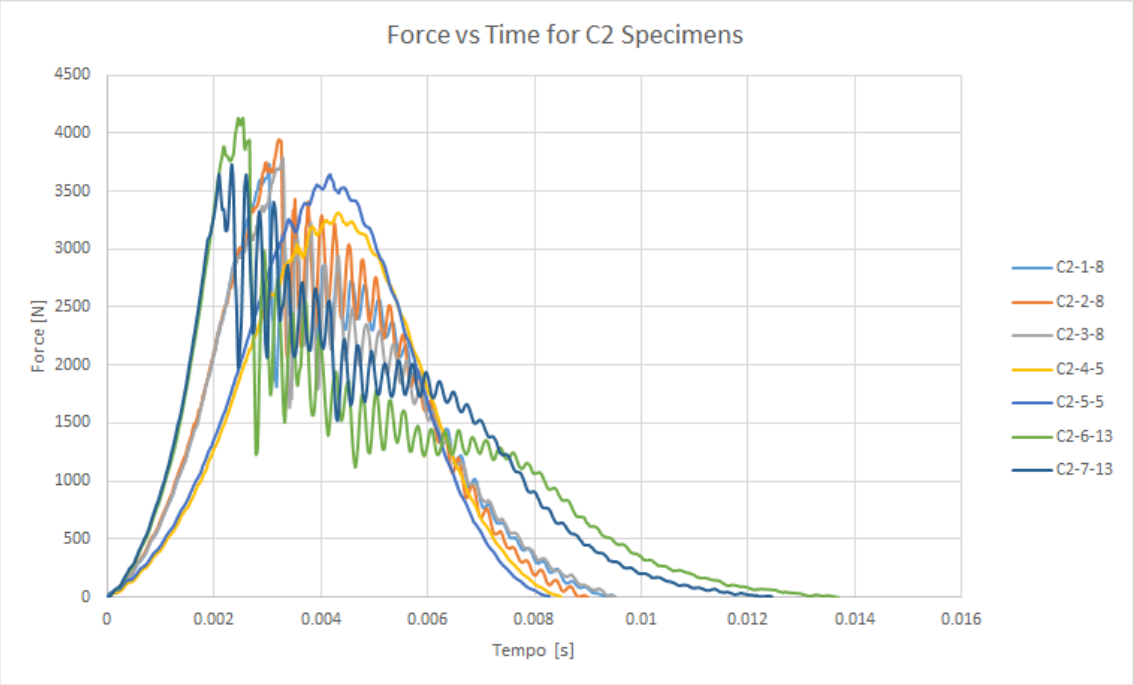


Figure 4.23: Impact's Force vs Time curve C2 specimens

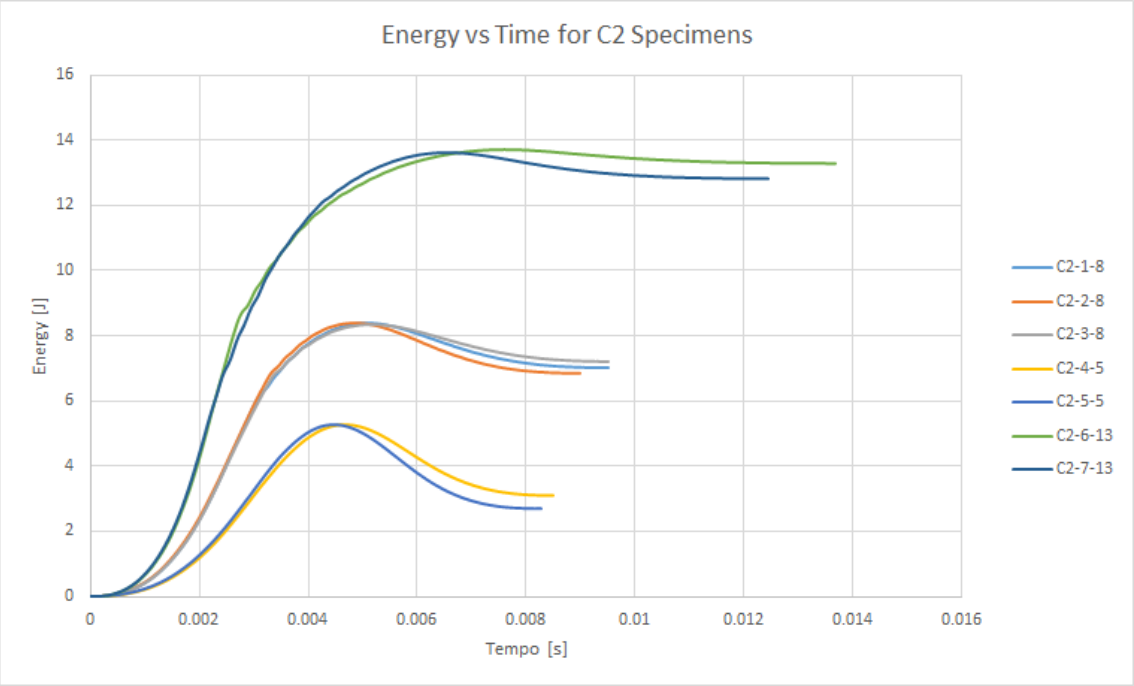


Figure 4.24: Impact's Energy vs Time curve C2 specimens

Table 4.18: Values from K30 Specimens' impact test

Impact Energy	Specimen	Impact Velocity	Peak Force [N]	Peak Deflection [mm]	Final Deflection [mm]	Peak Energy [J]	Final Energy [J]
5 J	4	1.318	2291.00	4.88	2.62	5.29	4.41
	5	1.313	1969.85	4.80	2.40	5.25	4.37
	6	1.319	2236.63	4.82	2.55	5.30	4.45
8 J	1	1.674	2384.56	7.03	5.32	8.48	8.07
	2	1.666	2441.45	6.82	5.02	8.40	7.95
	3	1.656	2129.16	6.92	5.06	8.30	7.23
13 J	7	2.124	2486.97	14.09	14.09	13.80	13.80
	8	2.131	2569.15	12.47	12.41	13.81	13.80

4.2.4 Kraton™ granules 30 g/m^2 - K30

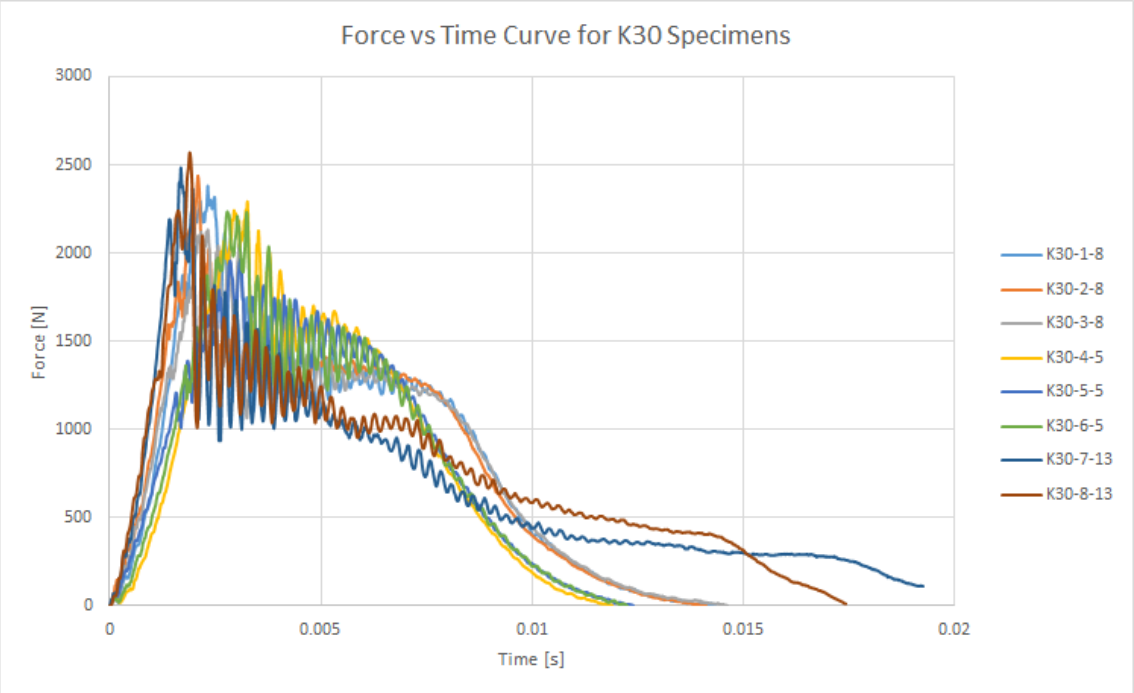


Figure 4.25: Impact's Force vs Time curve K30 specimens

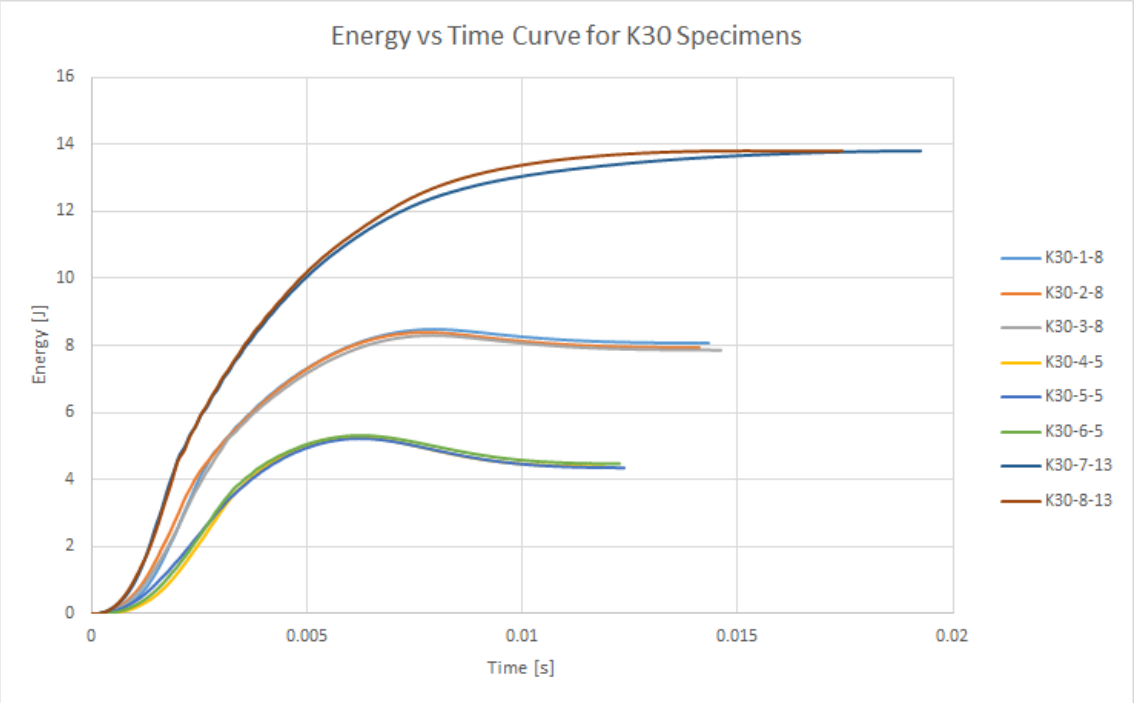


Figure 4.26: Impact's Energy vs Time curve K30 specimens

4.2.5 Kraton™ granules 40 g/m^2 - K40

Table 4.19: Values from K40 Specimens' impact test

Impact Energy	Specimen	Impact Velocity	Peak Force [N]	Peak Deflection [mm]	Final Deflection [mm]	Peak Energy [J]	Final Energy [J]
5 J	4	1.317	2330.19	4.45	2.09	5.26	4.31
	5	1.312	2844.78	4.34	2.28	5.22	4.11
	6	1.317	2909.26	4.10	1.81	5.23	4.05
8 J	1	1.662	2556.51	6.33	4.44	8.33	7.77
	2	1.667	2741.10	6.56	4.71	8.39	7.90
	3	1.672	3018.00	5.92	4.18	8.40	7.80
13 J	7	2.137	2952.25	8.73	7.11	13.67	13.31
	8	2.133	2175.94	10.96	9.58	13.80	13.65

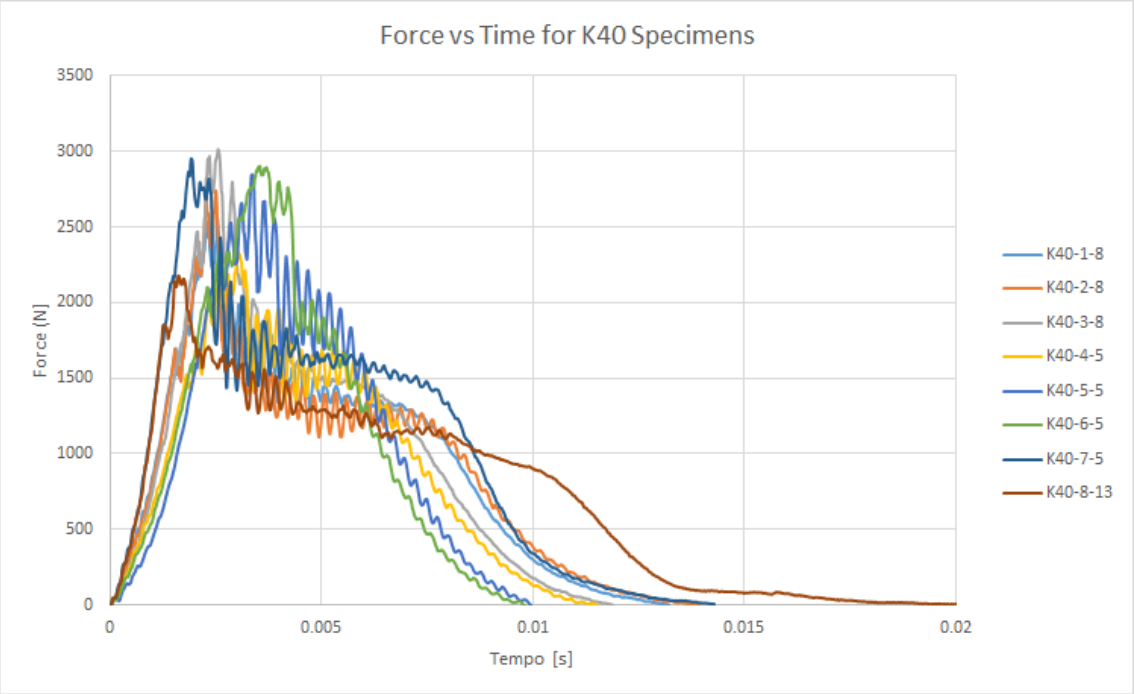


Figure 4.27: Impact's Force vs Time curve K40 specimens

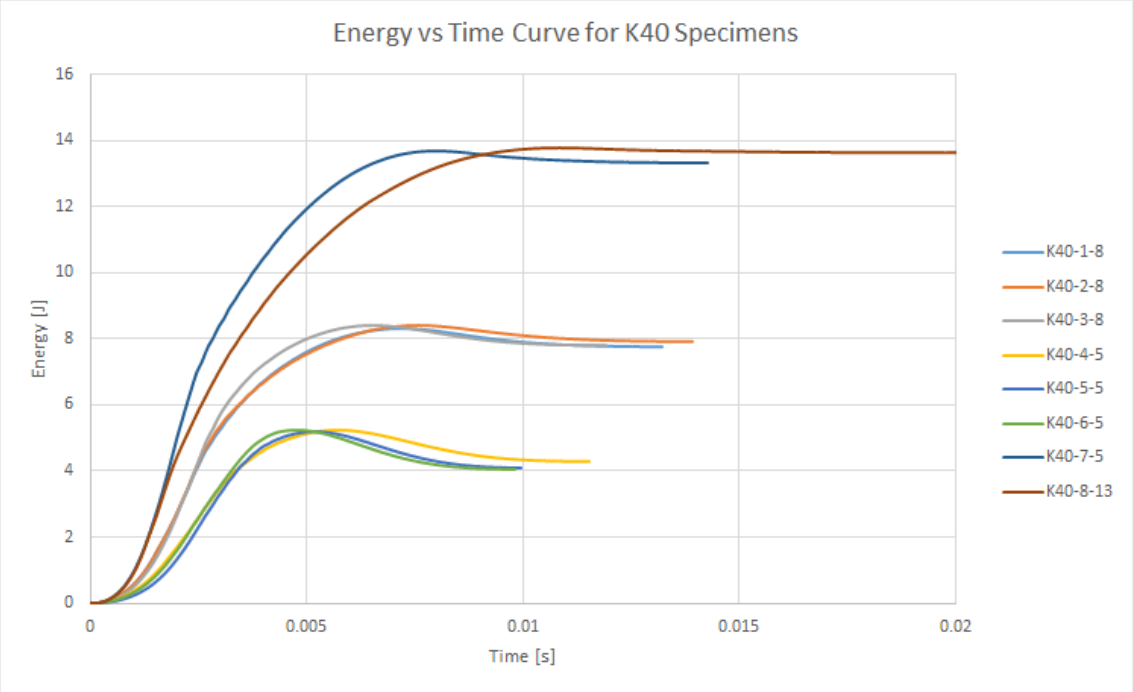


Figure 4.28: Impact's Energy vs Time curve K40 specimens

Table 4.20: Values from K60 Specimens' impact test

Impact Energy	Specimen	Impact Velocity	Peak Force [N]	Peak Deflection [mm]	Final Deflection [mm]	Peak Energy [J]	Final Energy [J]
5 J	4	1.323	2804.32	4.27	1.52	5.26	4.09
	5	1.314	2670.30	4.26	2.05	5.20	4.28
	6	1.316	2500.88	4.38	1.96	5.22	4.26
8 J	1	1.676	2988.92	6.21	4.28	8.38	7.79
	2	1.671	2933.28	6.33	4.48	8.37	7.82
	3	1.672	2407.32	6.33	3.81	8.40	7.73
13 J	7	2.137	2882.71	10.25	9.32	13.67	13.47
	8	2.128	3181.10	9.66	8.18	13.61	13.29

4.2.6 Kraton™ granules 60 g/m^2 - K60

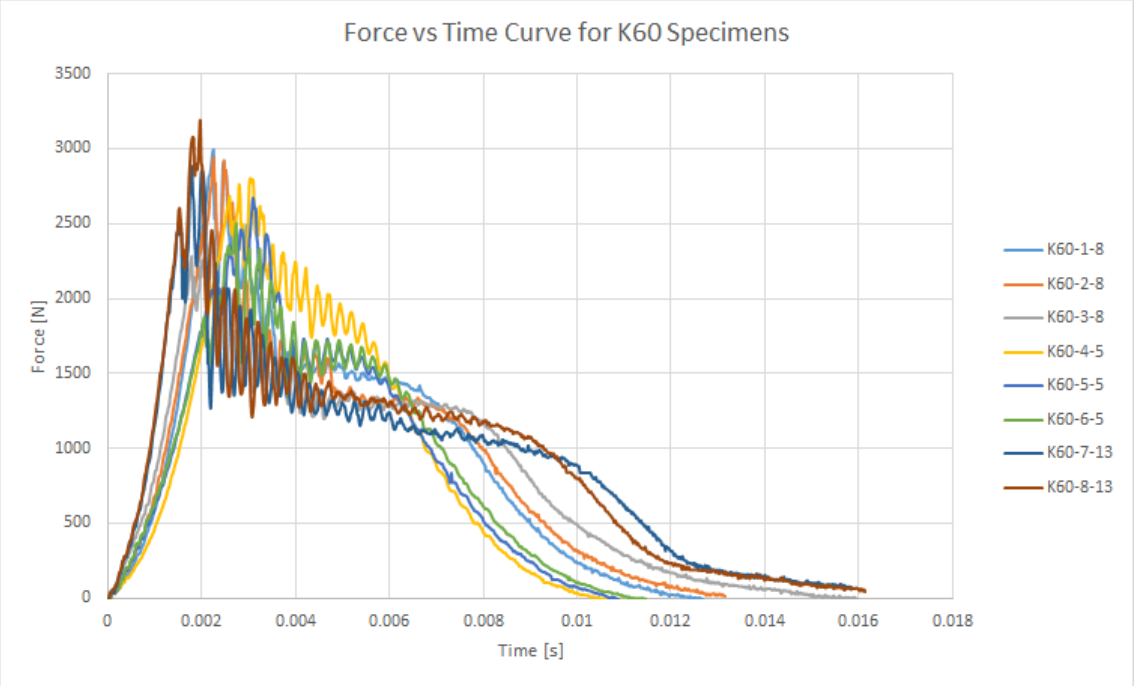


Figure 4.29: Impact's Force vs Time curve K60 specimens

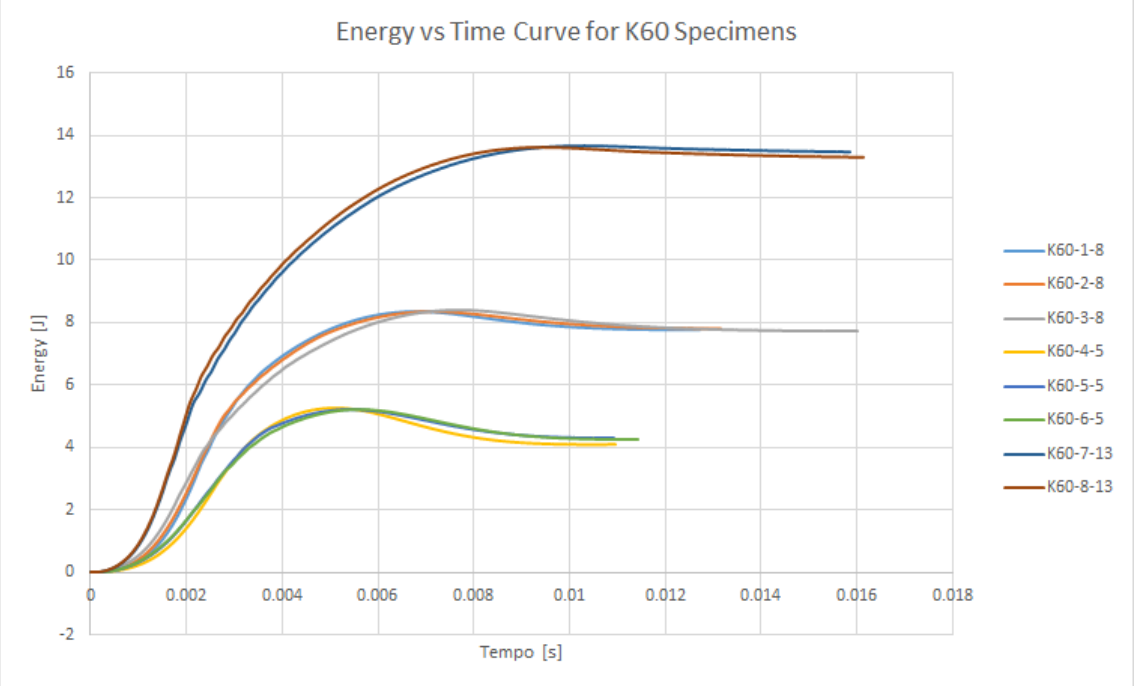


Figure 4.30: Impact's Energy vs Time curve K60 specimens

4.2.7 Expanded cork granules 10 g/m^2 - B10

Table 4.21: Values from B10 Specimens' impact test

Impact Energy	Specimen	Impact Velocity	Peak Force [N]	Peak Deflection [mm]	Final Deflection [mm]	Peak Energy [J]	Final Energy [J]
5 J	4	1.324	2473.06	4.29	1.88	5.27	4.35
	5	1.325	2164.56	4.52	2.04	5.30	4.50
	6	1.321	2397.20	11.25	1.70	5.25	4.32
8 J	1	1.676	2392.14	6.89	4.62	8.45	7.97
	2	1.671	2189.85	6.95	4.76	8.41	7.93
	3	1.675	2296.05	6.96	4.62	8.45	7.93
13 J	7	2.137	2407.32	11.25	10.59	13.74	13.67
	8	2.128	2139.27	11.58	11.12	13.65	13.62

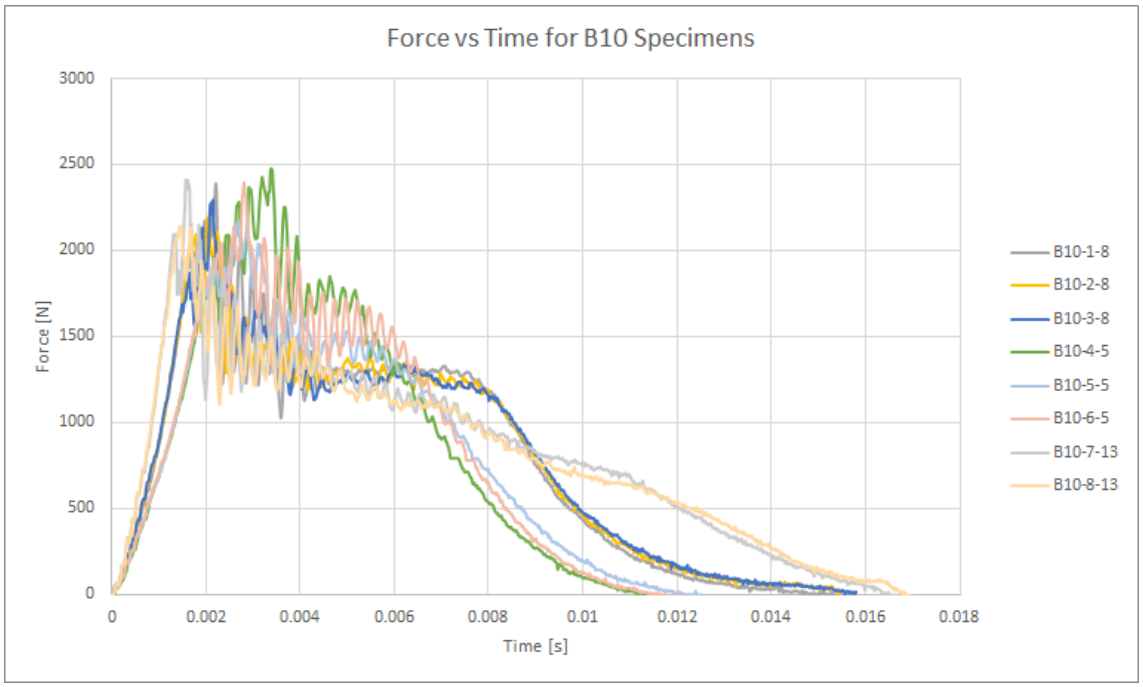


Figure 4.31: Impact's Force vs Time curve B10 specimens

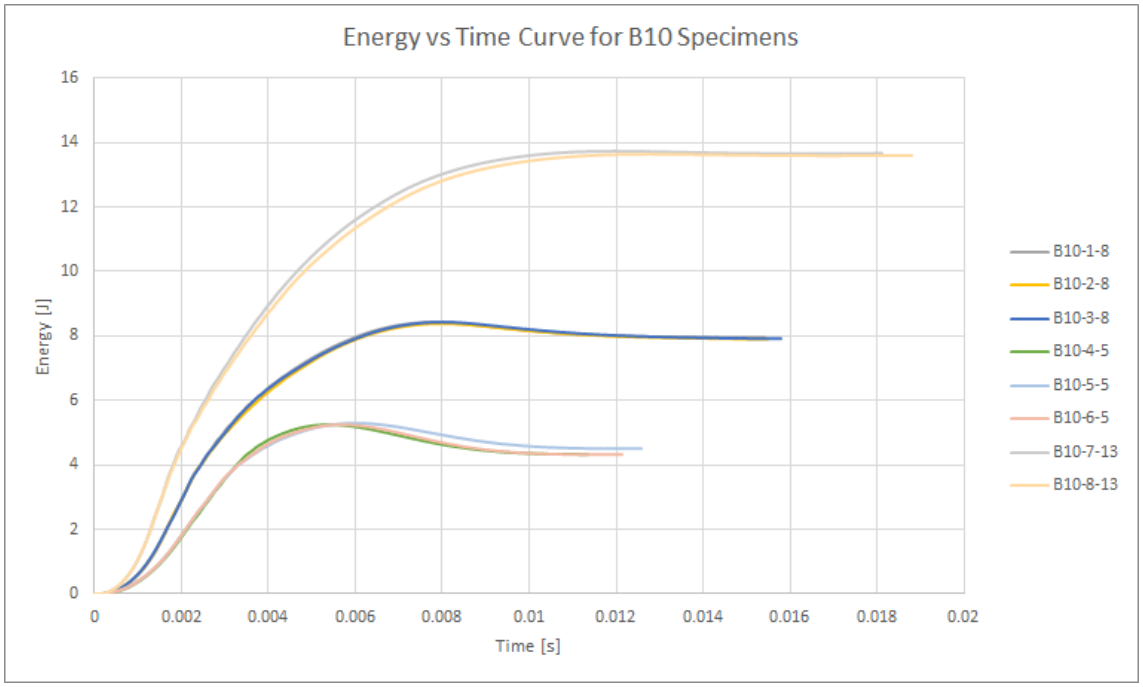


Figure 4.32: Impact's Energy vs Time curve B10 specimens

Table 4.22: Values from B20 Specimens' impact test

Impact Energy	Specimen	Impact Velocity	Peak Force [N]	Peak Deflection [mm]	Final Deflection [mm]	Peak Energy [J]	Final Energy [J]
5 J	4	1.322	2245.48	4.46	2.09	5.27	4.52
	5	1.319	1977.44	4.58	2.07	5.25	4.36
	6	1.319	2361.80	4.28	1.95	5.23	4.38
8 J	1	1.680	2296.05	6.82	4.63	8.48	8.05
	2	1.672	2240.42	7.26	4.71	8.43	8.01
	3	1.672	2073.53	6.98	4.71	8.41	7.93
13 J	7	2.133	2291.00	13.06	13.06	13.81	13.81
	8	2.135	2736.05	13.07	13.07	13.82	13.82

4.2.8 Expanded cork granules 20 g/m^2 - B20

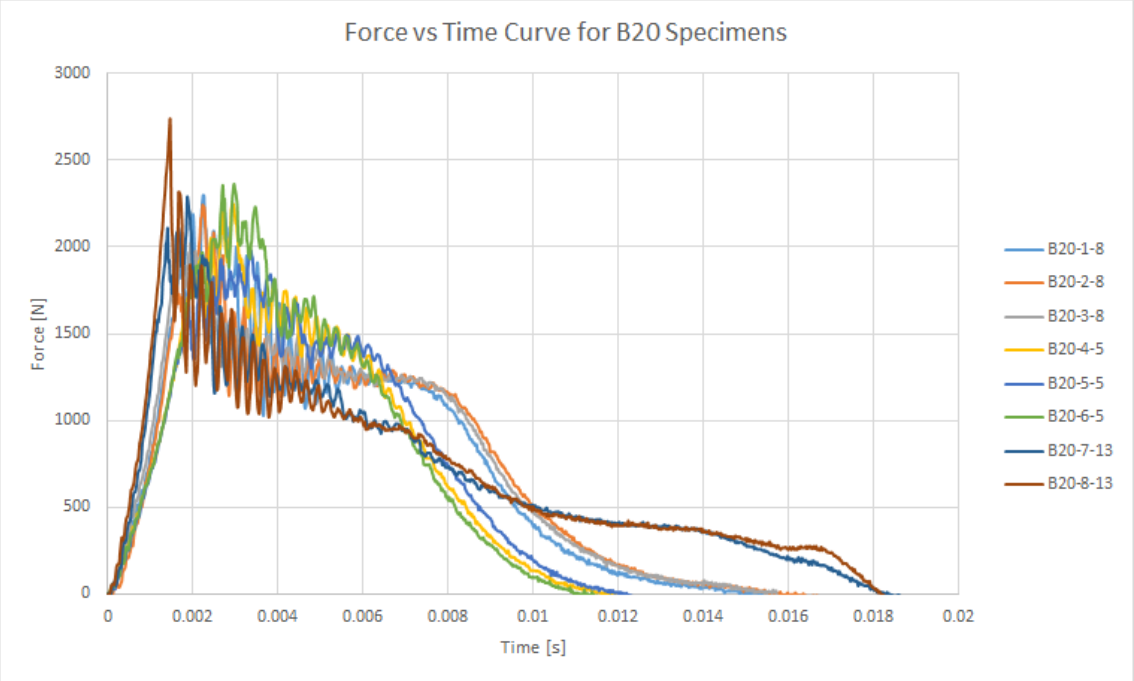


Figure 4.33: Impact's Force vs Time curve B20 specimens

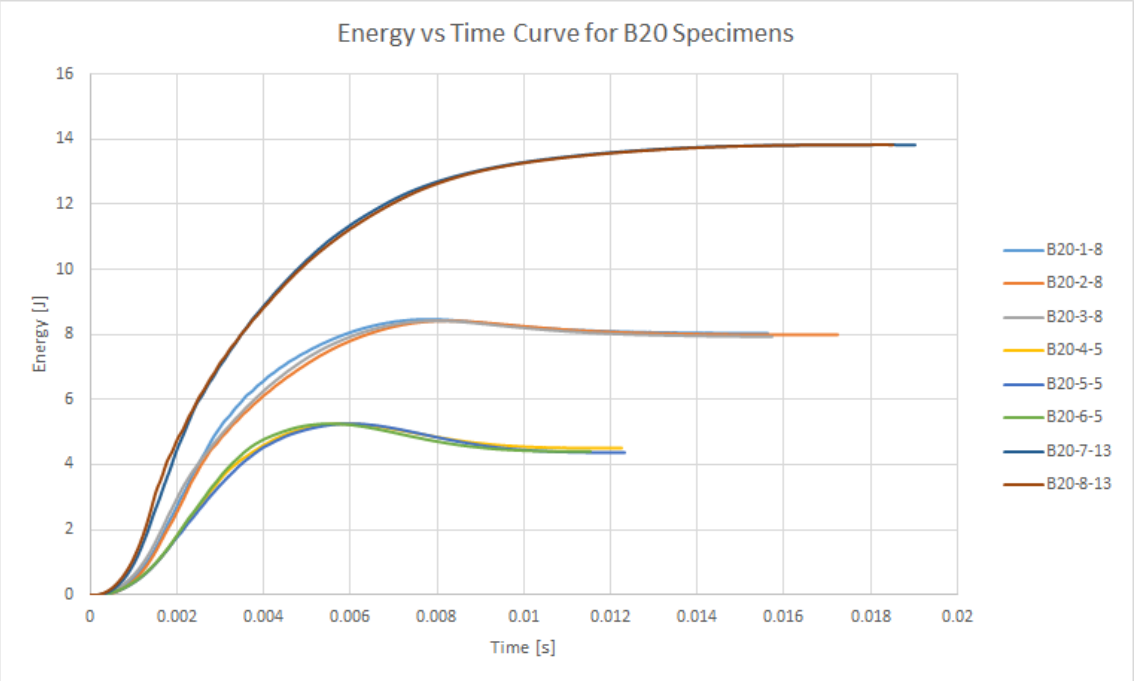


Figure 4.34: Impact's Energy vs Time curve B20 specimens

4.2.9 Expanded cork granules 30 g/m^2 - B30

Table 4.23: Values from B30 Specimens' impact test

Impact Energy	Specimen	Impact Velocity	Peak Force [N]	Peak Deflection [mm]	Final Deflection [mm]	Peak Energy [J]	Final Energy [J]
5 J	4	1.319	2781.56	4.17	1.70	5.23	4.22
	5	1.324	2493.29	4.21	1.84	5.27	4.09
	6	1.320	2523.94	4.24	1.76	5.24	4.30
8 J	1	1.684	2392.14	6.43	4.10	8.50	7.95
	2	1.665	2589.38	6.04	3.42	8.29	7.63
	3	1.668	2493.29	6.19	3.50	8.33	7.67
13 J	7	2.133	2452.83	10.52	9.66	13.65	13.51
	8	2.135	2498.35	10.63	8.92	13.65	13.41

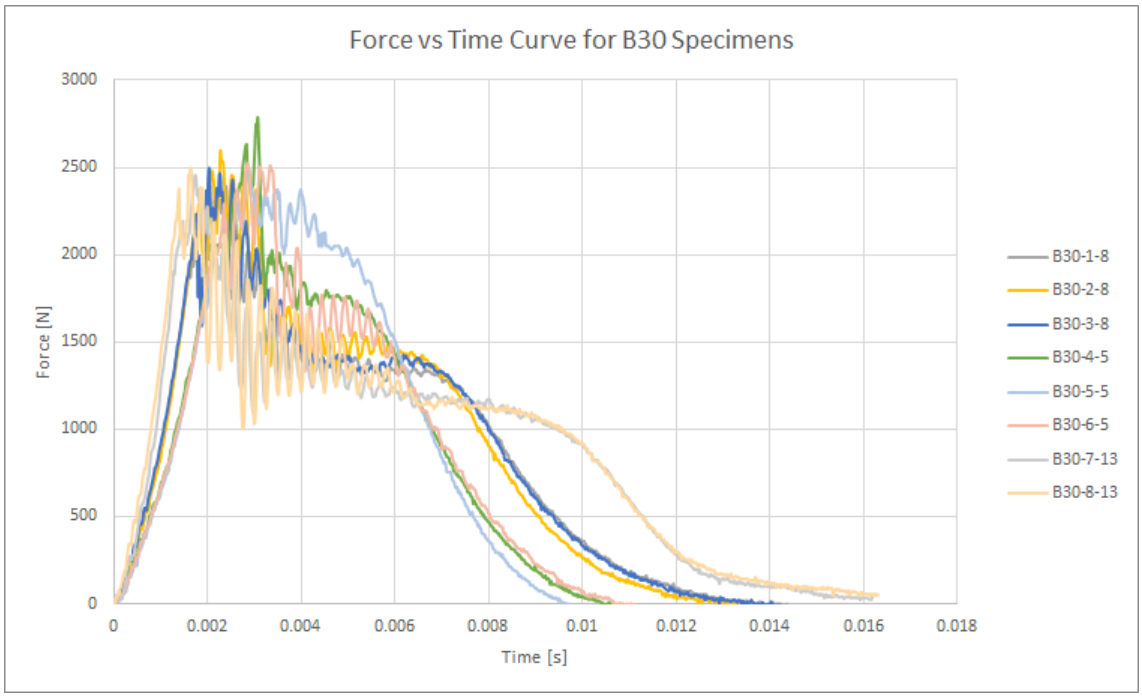


Figure 4.35: Impact's Force vs Time curve B30 specimens

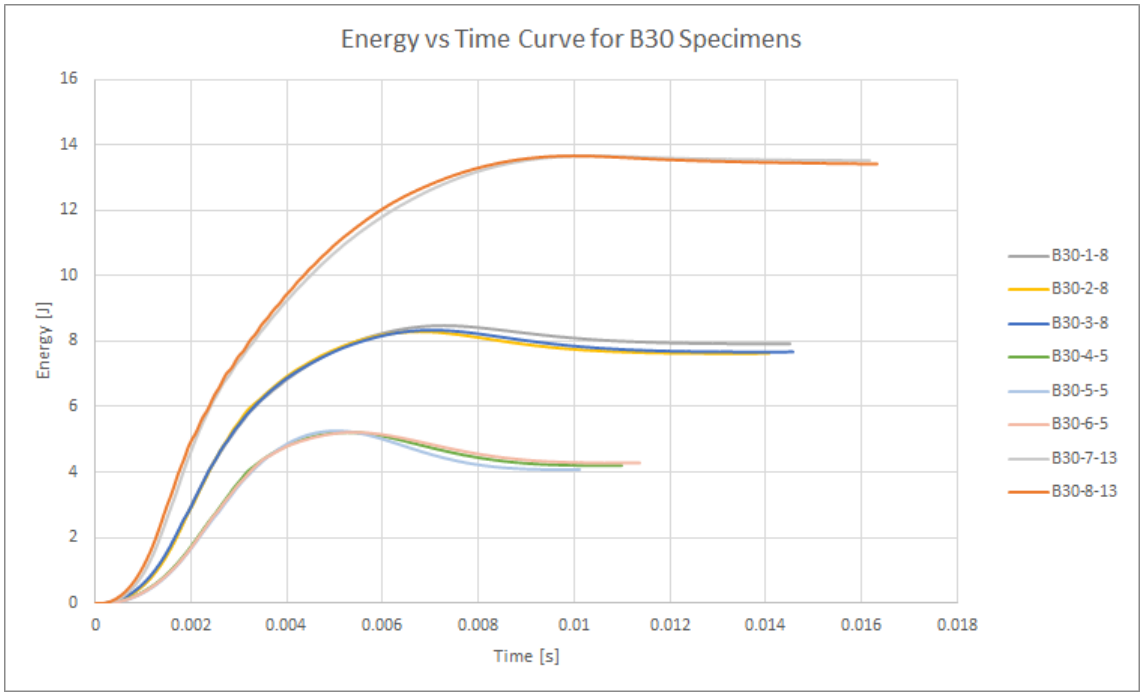


Figure 4.36: Impact's Energy vs Time curve B30 specimens

Table 4.24: Values from B40 Specimens' impact test

Impact Energy	Specimen	Impact Velocity	Peak Force [N]	Peak Deflection [mm]	Final Deflection [mm]	Peak Energy [J]	Final Energy [J]
5 J	4	1.320	2452.83	4.38	1.82	5.25	4.38
	5	1.317	2255.59	4.79	2.36	5.24	4.57
	6	1.321	2326.40	4.39	2.30	5.26	4.54
8 J	1	1.670	2098.82	7.02	5.06	8.40	7.99
	2	1.671	2402.26	6.59	4.20	8.38	7.89
	3	1.675	2063.41	6.95	4.90	8.45	8.09
13 J	7	2.135	2103.87	12.40	12.30	13.78	13.78
	8	2.136	2225.25	11.96	11.67	13.76	13.73

4.2.10 Expanded cork granules 40 g/m^2 - B40

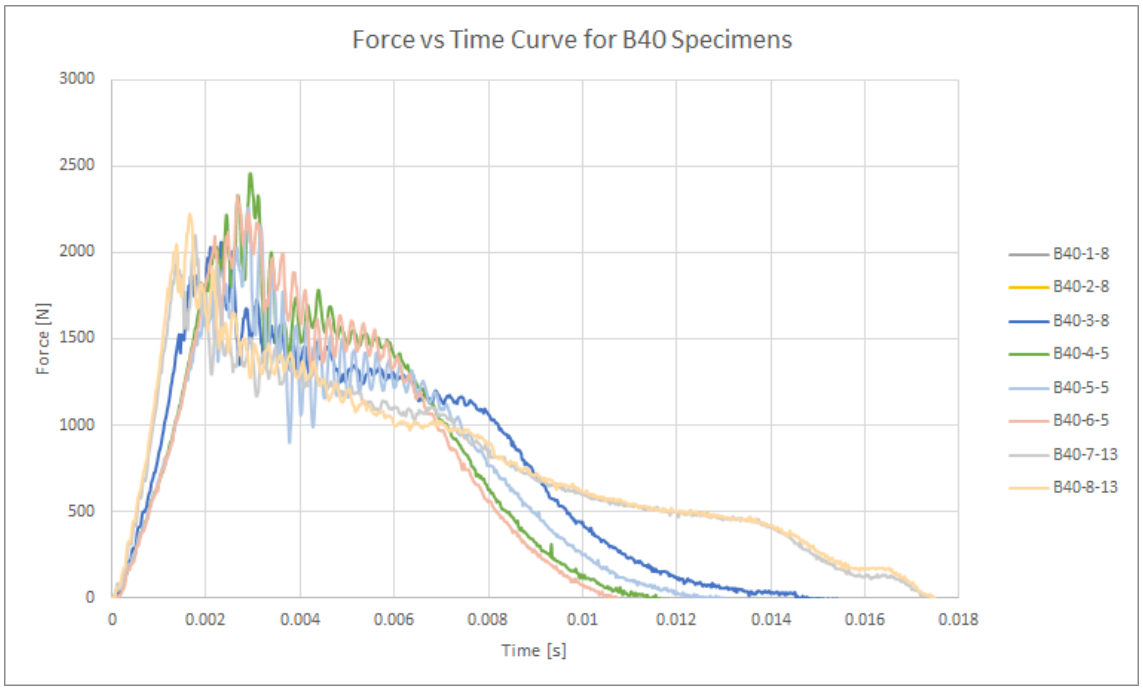


Figure 4.37: Impact's Force vs Time curve B40 specimens

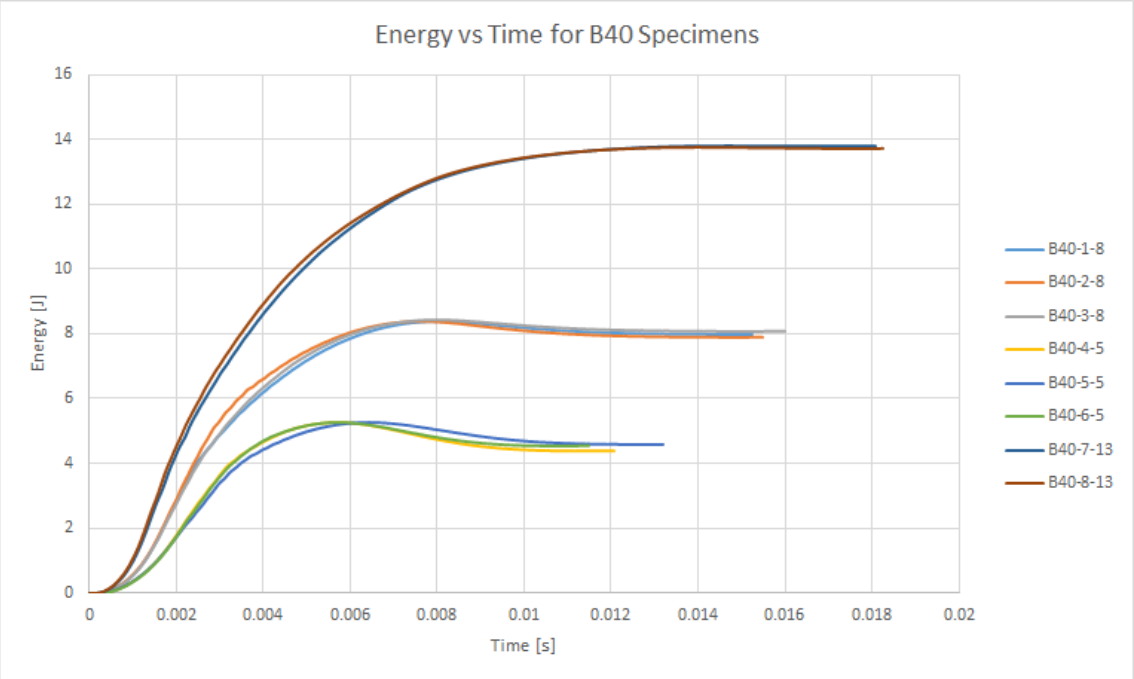


Figure 4.38: Impact's Energy vs Time curve B40 specimens

4.2.11 Analysis of Results

Looking at the reference laminates' specimens (Fig. 4.39), it is possible to see by figures 4.19 and 4.20, that some specimens were severely damaged. In the force-time curve (Fig. 4.19), all the graphs presented a drop after their peak force was reached, and when the impact energy was 13 J, both specimens even got pierced.

On the other hand, looking for instance at the graphs of C1 (Fig. 4.21 and 4.22) and C2 (Fig. 4.23 and 4.24), it is possible to see that with the lower impact energy (5 J), the force-time curves, although showing a drop after the peak force, the decreases are slower, and only present some a slight wave-type irregularity that can be due to some small delaminations in the impacted zone.

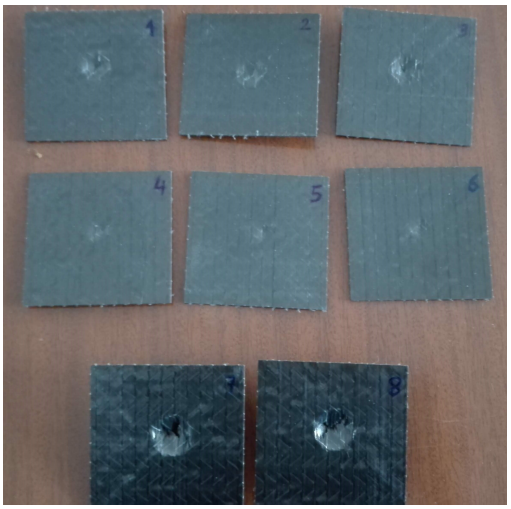


Figure 4.39: REF Impacted specimens

Table 4.25: Average values from all specimens' impact test

Laminate	**Energy [J]**	**Peak Force [N]**	**Peak Deflection [mm]**	**Final Deflection [mm]**	**Peak Energy [J]**	**Final Energy [J]**	**Energy Recover [%]**
REF	5 J	2277.93	4.56	2.36	5.31	4.38	18%
	8 J	2481.49	6.67	4.98	8.45	7.98	6%
	13 J	2498.98	13.05	13.03	13.82	13.82	0%
C1	5 J	2919.38	4.15	1.71	5.24	3.35	36%
	8 J	3053.93	5.49	3.79	8.34	7.71	8%
	13 J	3016.10	9.72	8.27	13.58	13.36	2%
C2	5 J	3473.79	4.29	1.82	5.29	2.91	45%
	8 J	3818.75	5.33	3.24	8.38	7.03	16%
	13 J	3927.69	7.86	5.90	13.67	13.67	4%
K30	5 J	2165.83	4.83	2.52	5.28	4.41	16%
	8 J	2318.39	6.92	5.13	8.40	7.96	5%
	13 J	2528.06	13.28	13.25	13.80	13.80	0%
K40	5 J	2694.74	4.30	2.06	5.24	4.16	21%
	8 J	2771.87	6.27	4.44	8.37	7.82	7%
	13 J	2564.10	9.85	8.34	13.73	13.48	2%
K60	5 J	2658.50	4.30	1.84	5.23	4.21	19%
	8 J	2776.51	6.29	4.19	8.38	7.78	7%
	13 J	3031.90	9.96	8.75	13.64	13.38	2%
B10	5 J	2344.94	6.68	1.87	5.27	4.39	17%
	8 J	2292.68	6.93	4.67	8.43	7.94	6%
	13 J	2273.30	11.42	10.85	13.70	13.65	0%
B20	5 J	2194.91	4.44	2.04	5.25	4.42	16%
	8 J	2203.33	7.02	4.68	8.44	8.00	5%
	13 J	2513.52	13.07	13.07	13.81	13.81	0%
B30	5 J	2599.50	4.21	1.77	5.25	4.20	20%
	8 J	2491.61	6.22	3.67	8.37	7.75	7%
	13 J	2475.59	10.34	9.29	13.65	13.46	1%
B40	5 J	2344.94	4.50	2.16	5.25	4.49	14%
	8 J	2188.16	6.85	4.72	8.41	7.99	5%
	13 J	2164.56	12.18	11.98	13.77	13.75	0%

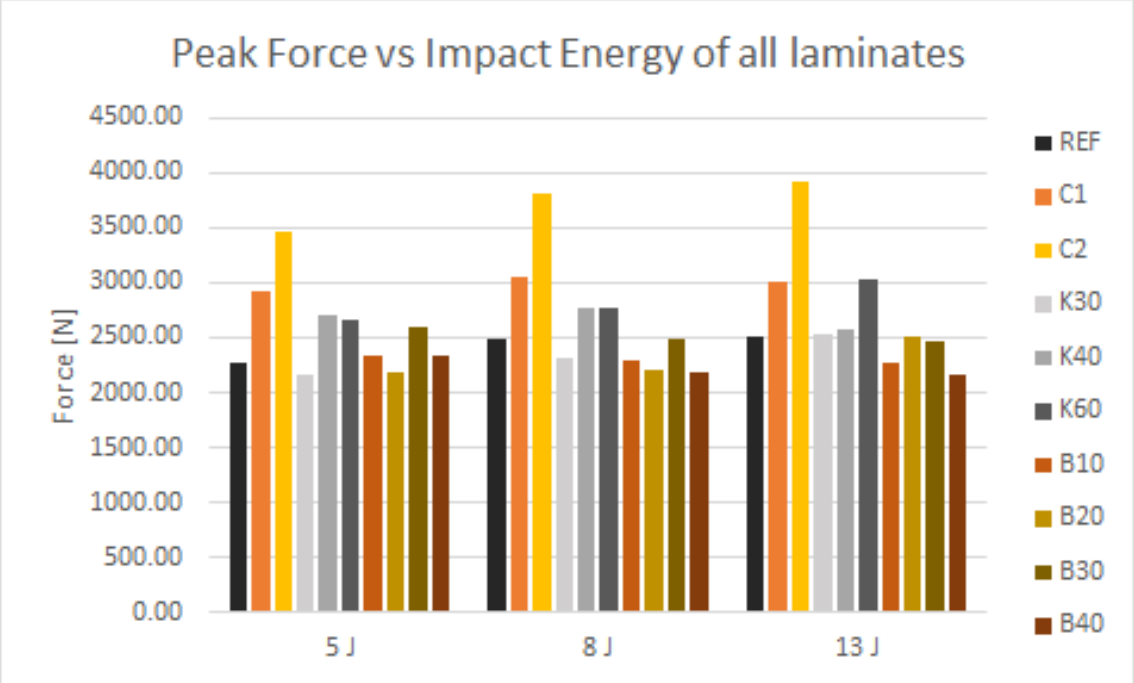


Figure 4.40: Peak force vs impact energy of all laminates

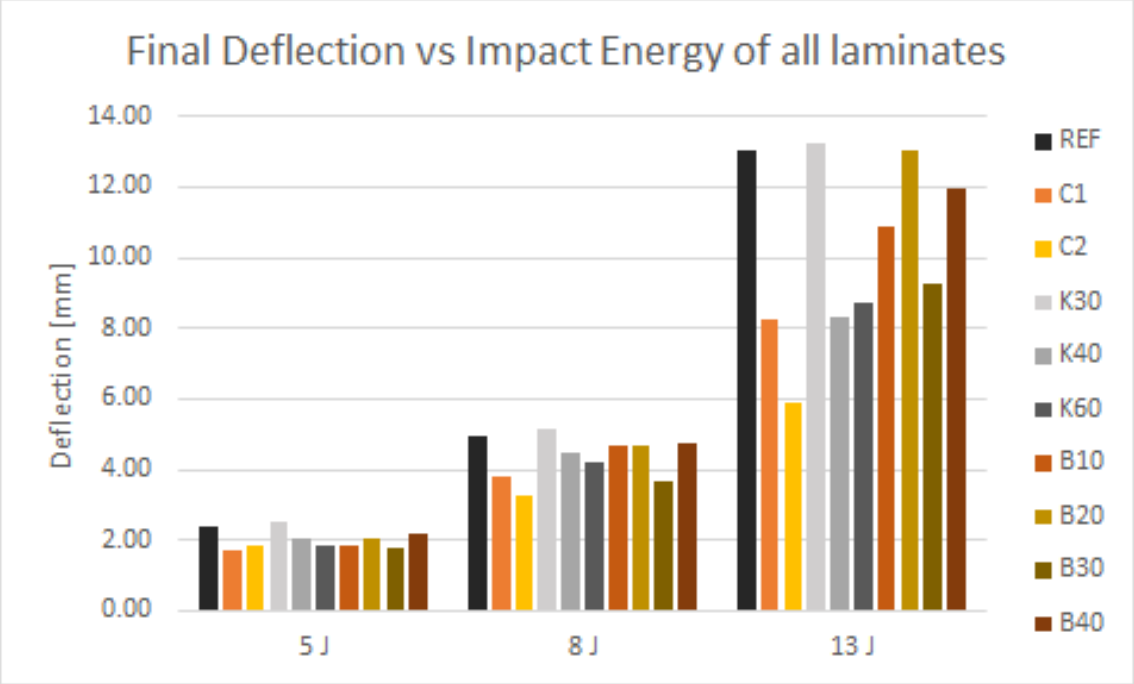


Figure 4.41: Final deflection vs impact energy of all laminates

The graph in figure 4.40 shows the average values of the specimens for each laminate and each impact energy. The result that automatically stand out is C2, which by far has the highest peak force, followed by C1, showing that having a cork film allows to reach higher force values.

After the cork film interlayers, K40 and K60 are also capable of reaching higher values, when comparing with the reference. Regarding the "B group", the results are somewhat dispersed, not allowing to have a global comparison with the reference. This might be due to the granules dispersion that, since it was not homogeneous, lead to some areas of the laminate to have higher granules concentration than others.

The discussion about the final deflection (Fig. 4.41) becomes a bit more complicated, due to the fact that the values are quite similar in both 5 and 8 J, but as the energy increases to 13 J, the differences among the laminates increase as well. The global idea taken is that cork films can decrease the final deflection, making also the damage not so easy to detect.

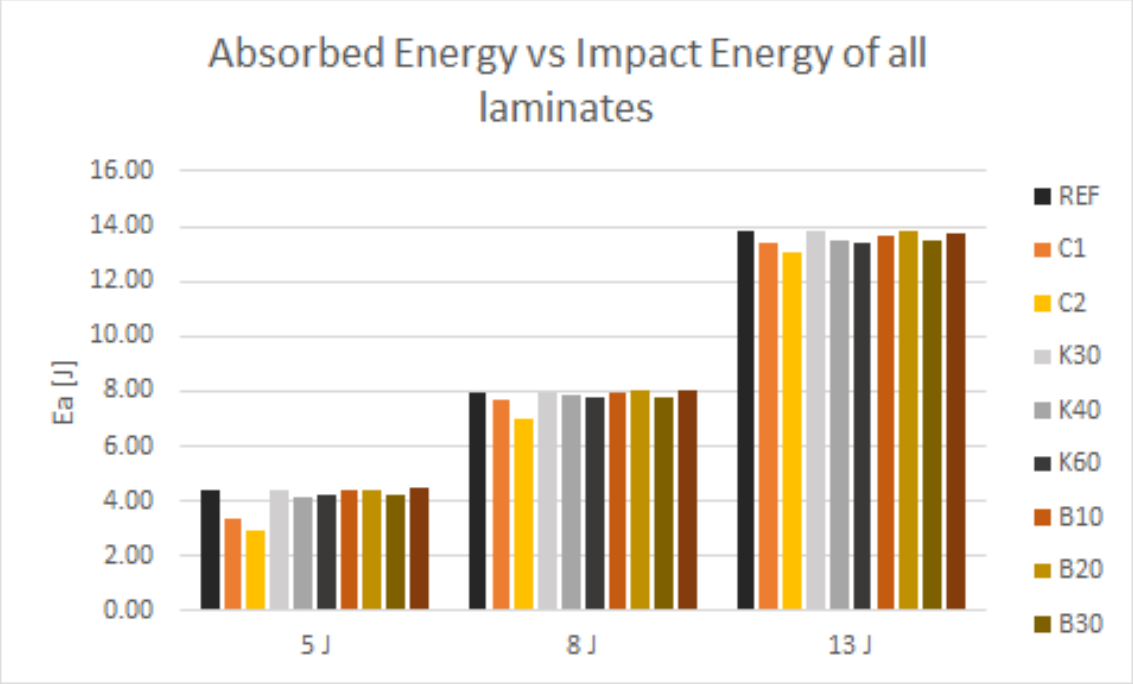


Figure 4.42: Absorbed energy vs impact energy of all laminates

The absorbed energy value ends up meaning the amount of damage that the component suffered, since it is the remaining of energy that was elastically dissipated. Looking at figure 4.42, it is possible to see that all the values are similar for each energy level, but the global tendency is that, when the thickness/concentration increases, the absorbed energy decreases. Exception made for the expanded cork granules that behave differently, but the pattern repeats itself for every impact energy level.

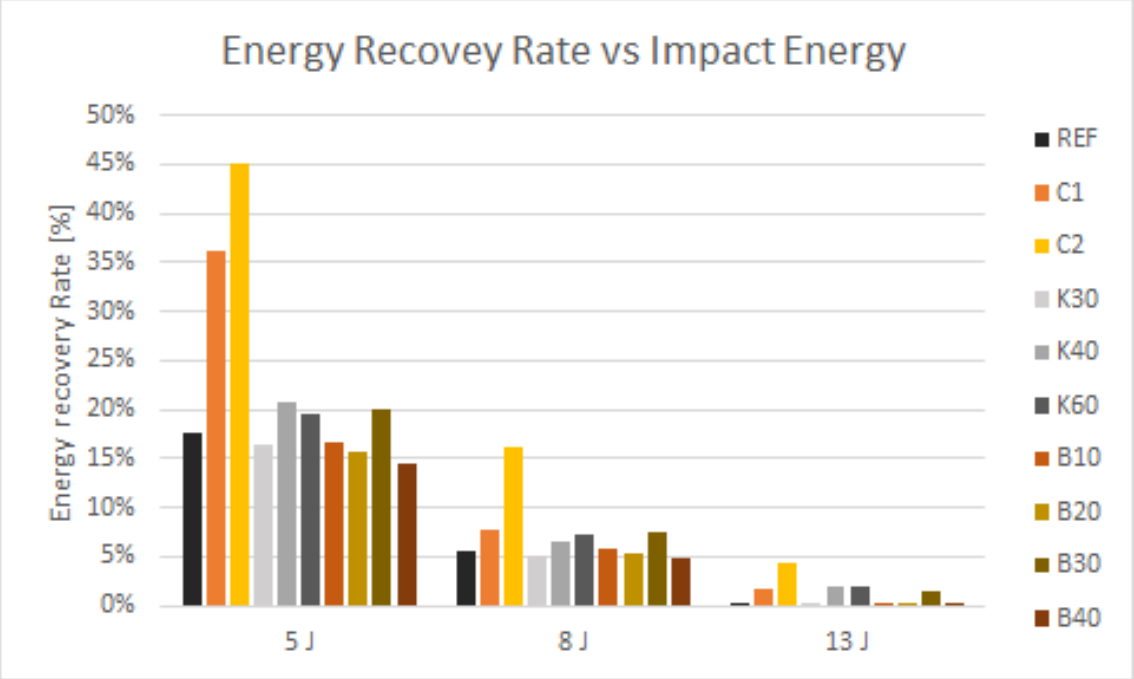


Figure 4.43: Energy recovery rate for each impact energy and all laminates

The energy recovery rate is the most interesting analysis to be made under the context of this study, and concerning impact tests, as it was mentioned before. Figure 4.43 shows the average values for each laminate and each impact energy of the energy recovery rate.

Once again, cork films stand out, showing good properties, especially the thicker film used, that managed to show the higher rate for every impact energy. Other good results that managed to overcome or equal the reference material were K40, K60 and B30, which gives the impression that having more concentration (i.e higher toughness), increases the energy recovery rate, which was the hypothesis that the study was based on: adding a tough material as interlayer to increase the damage tolerance.

4.3 Tensile After Impact Tests

Tensile after impact test (TAI) is of extreme importance for this study, since it's one important way to assess the residual properties after impact, and thus assess the damage tolerance, by measuring how a composite is affected by an impact load through a tensile test.

The results obtained here, can be compared with the tensile test results in order to calculate the reduction of mechanical properties, such as the ultimate tensile strength (UTS) and the Young's modulus (E).

This section begins by presenting the stress-strain curve of every specimen that was subjected to an impact load (Table 4.26) and a table where the main properties and measurements are indicated. After, a comparison will be made between the reference laminate and each group of interlayer solution, and in the very end a comparison between all the laminates.

Table 4.26 shows the amount of impact energy that each specimen has suffered.

Table 4.26: Specimens for each laminate and their respective impact energy

Specimen	Impact Energy [J]
A1	2.5
A2	2.5
A3	5.0
A4	5.0
A5	3.5
A6	3.5

4.3.1 Reference - REF

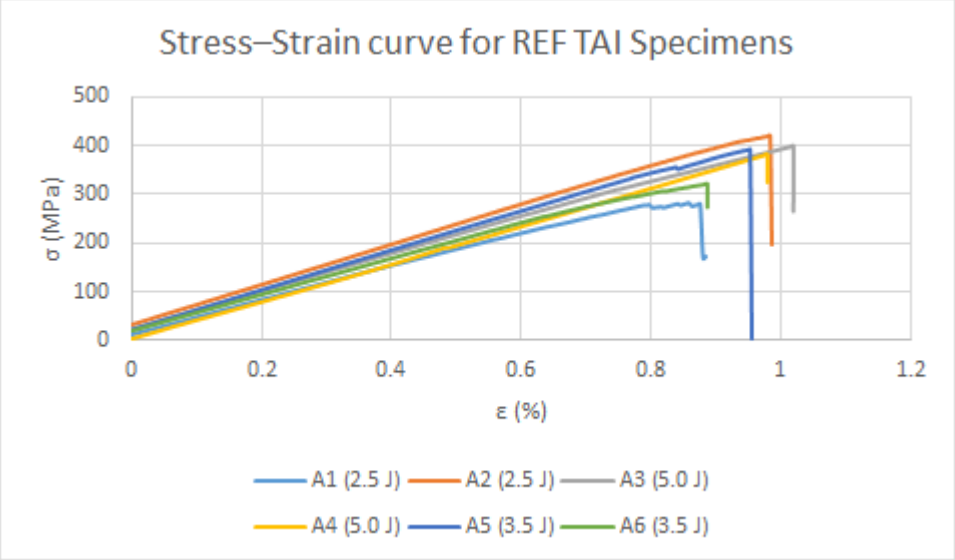


Figure 4.44: Stress–Strain curve for TAI REF Specimens

Table 4.27: Dimensions and mechanical properties of REF tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus [GPa]	Energy [J]	UTS Average [MPa]	Young's Modulus Average [GPa]
A1	24.53	1.15	282	36.2	2.5	353	38.8
A2	24.53	1.18	423	41.5			
A3	24.85	1.13	398	38.8	5.0	390	38.0
A4	24.72	1.15	381	37.2			
A5	23.05	1.20	394	41.4	3.5	357	39.3
A6	24.50	1.25	320	37.2			

4.3.2 Thin Cork Film - C1

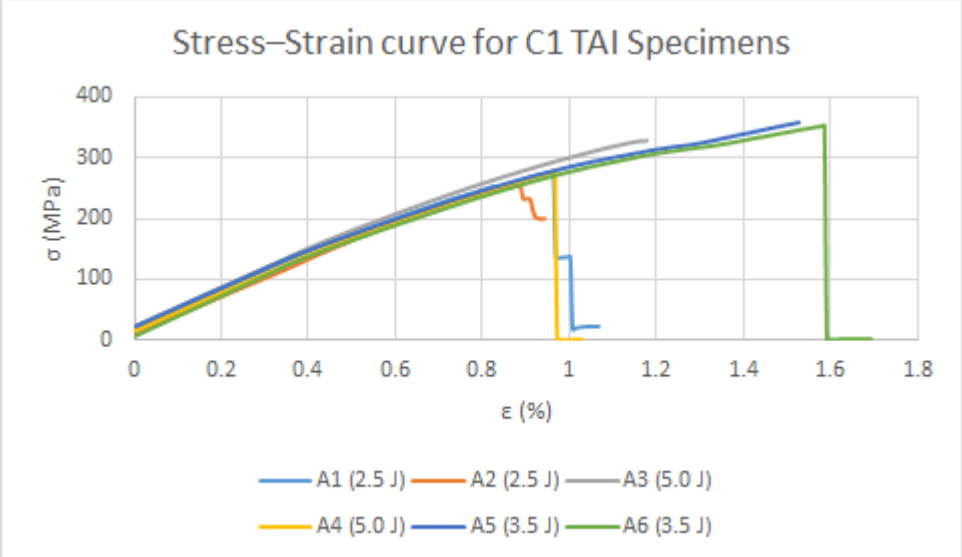


Figure 4.45: Stress–Strain curve for TAI C1 Specimens

Table 4.28: Dimensions and mechanical properties of C1 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus [GPa]	Energy [J]	UTS Average [MPa]	Young's Modulus Average [GPa]
A1	24.58	1.35	279	32.1	2.5	267	31.3
A2	24.60	1.35	254	30.5			
A3	24.87	1.38	329	32.2	5.0	303	31.8
A4	24.78	1.43	278	31.4			
A5	24.98	1.47	358	32.0	3.5	355	32.6
A6	25.02	1.48	352	33.2			

4.3.3 Thick Cork Film - C2

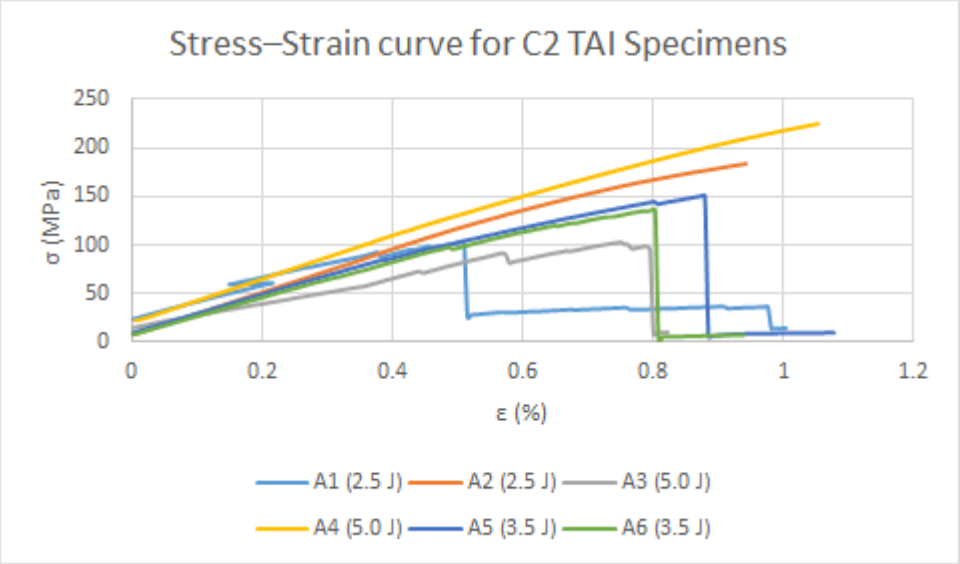


Figure 4.46: Stress–Strain curve for TAI C2 Specimens

Table 4.29: Dimensions and mechanical properties of C2 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus [GPa]	Energy [J]	UTS Average [MPa]	Young's Modulus Average [GPa]
A1	23.23	1.93	103	21.0	2.5	166.10	21.38
A2	24.57	1.97	229	21.8			
A3	24.68	1.85	102	11.8	5.0	172	17.0
A4	24.88	1.92	242	22.3			
A5	24.98	2.08	151	19.9	3.5	144	19.6
A6	24.07	2.07	137	19.4			

4.3.4 Kraton™ granules 30 g/m^2 - K30

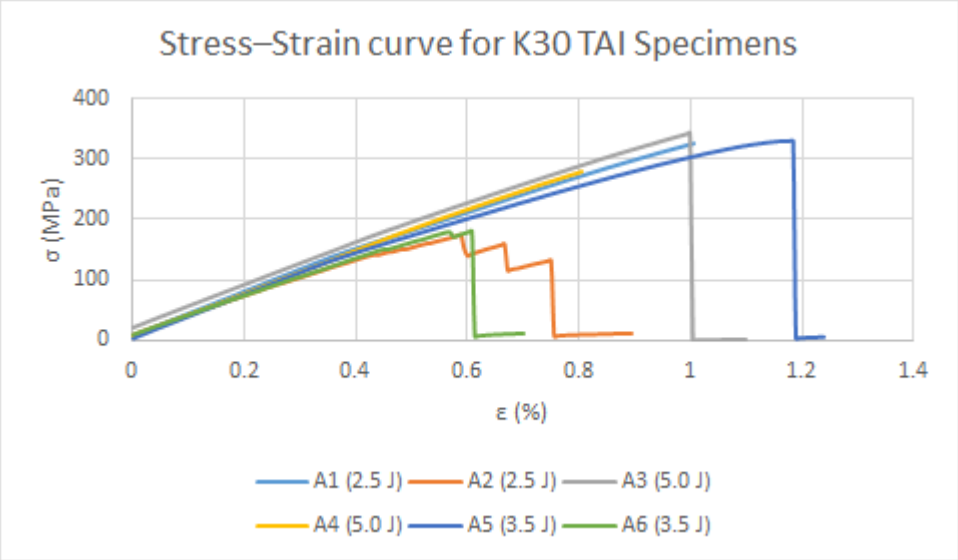


Figure 4.47: Stress–Strain curve for TAI K30 Specimens

Table 4.30: Dimensions and mechanical properties of K30 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus [GPa]	Energy [J]	UTS Average [MPa]	Young's Modulus Average [GPa]
A1	24.58	1.30	327	36.8	2.5	250	34.3
A2	24.53	1.28	174	31.8			
A3	24.83	1.28	341	35.1	5.0	311	35.4
A4	24.93	1.28	280	35.8			
A5	25.03	1.28	330	36.2	3.5	255	33.9
A6	25.02	1.28	181	31.6			

4.3.5 Kraton™ granules 40 g/m^2 - K40

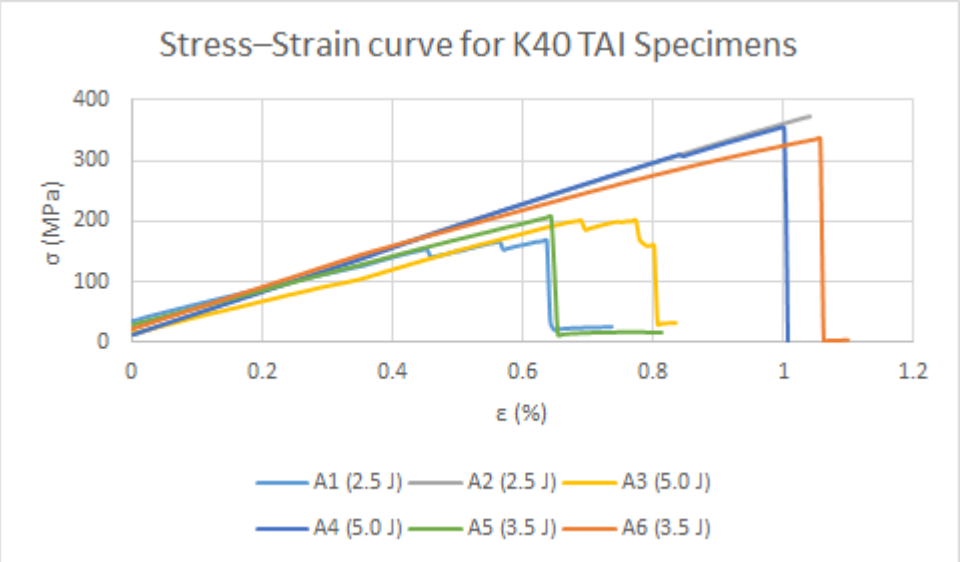


Figure 4.48: Stress–Strain curve for TAI K40 Specimens

Table 4.31: Dimensions and mechanical properties of K40 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus [GPa]	Energy [J]	UTS Average [MPa]	Young's Modulus Average [GPa]
A1	24.55	1.20	170	26.3	2.5	271	31.8
A2	24.57	1.25	372	37.4			
A3	24.82	1.20	202	25.9	5.0	279	30.7
A4	24.82	1.25	356	35.6			
A5	25.08	1.32	208	28.8	3.5	272	32
A6	25.07	1.33	336	34.7			

4.3.6 Kraton™ granules 60 g/m^2 - K60

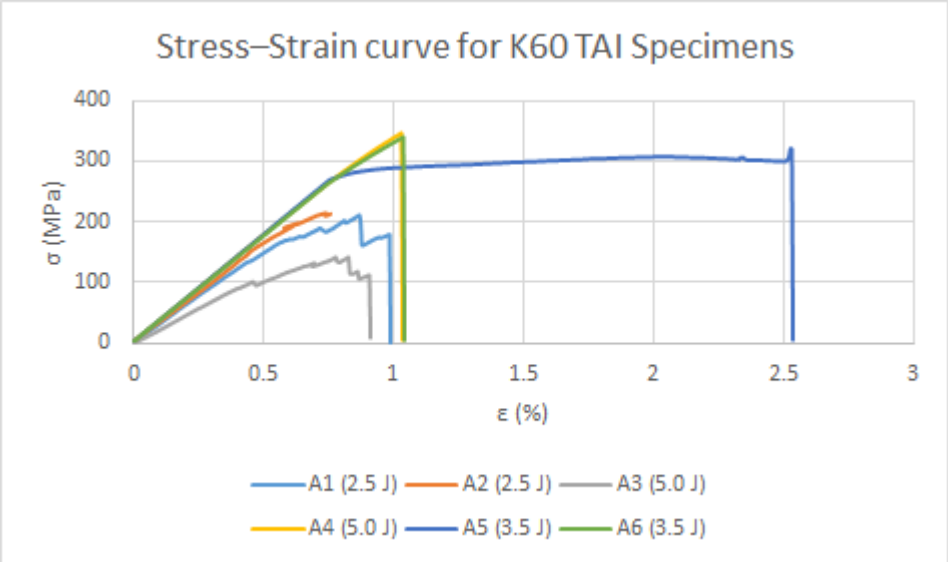


Figure 4.49: Stress–Strain curve for TAI K60 Specimens

Table 4.32: Dimensions and mechanical properties of K60 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus [GPa]	Energy [J]	UTS Average [MPa]	Young's Modulus Average [GPa]
A1	24.98	1.22	209	31.1	2.5	212	31.7
A2	25.02	1.25	216	32.2			
A3	25.22	1.25	141	23.3	5.0	244	29.3
A4	25.04	1.28	347	35.2			
A5	25.22	1.35	323	35.5	3.5	332	35.2
A6	25.23	1.35	341	34.9			

4.3.7 Expanded cork granules 10 g/m^2 - B10

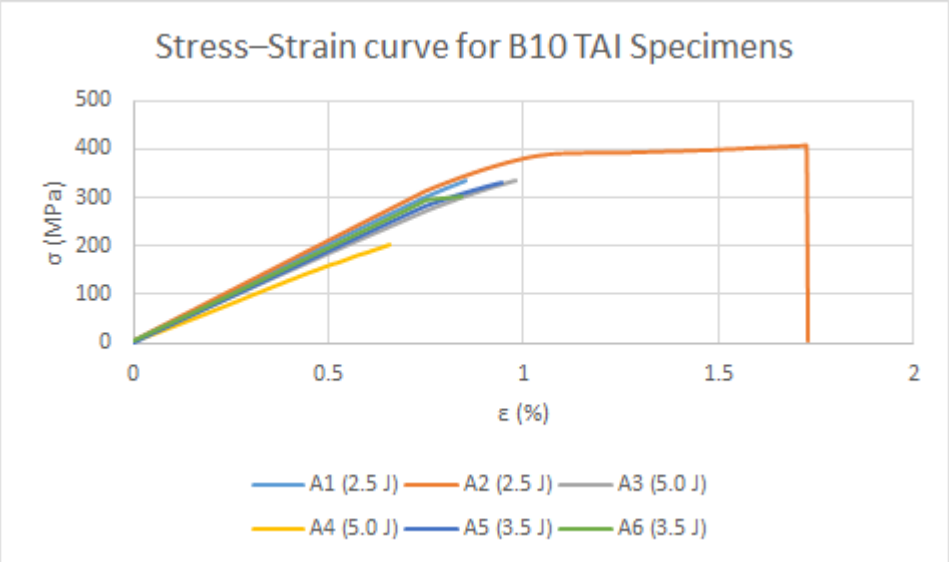


Figure 4.50: Stress–Strain curve for TAI B10 Specimens

Table 4.33: Dimensions and mechanical properties of B10 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus [GPa]	Energy [J]	UTS Average [MPa]	Young's Modulus Average [GPa]
A1	25.00	1.20	429	40.5	2.5	419	41.1
A2	24.95	1.18	408	41.7			
A3	25.08	1.23	335	36.4	5.0	268	34.0
A4	25.17	1.23	201	31.7			
A5	25.18	1.25	329	37.5	3.5	360	38
A6	25.23	1.27	391	38.3			

4.3.8 Expanded cork granules 20 g/m^2 - B20

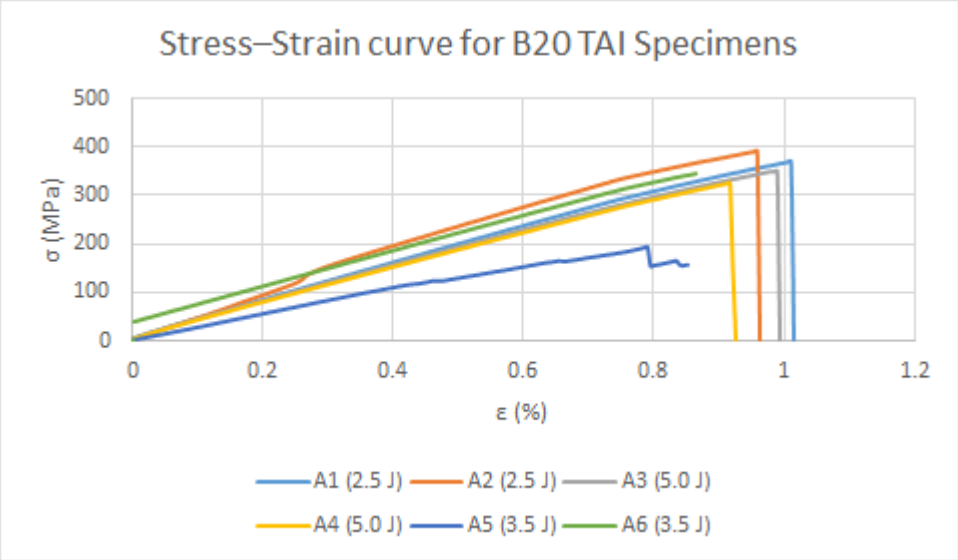


Figure 4.51: Stress–Strain curve for TAI B20 Specimens

Table 4.34: Dimensions and mechanical properties of B20 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus [GPa]	Energy [J]	UTS Average [MPa]	Young's Modulus Average [GPa]
A1	24.82	1.22	369	39.0	2.5	380	42.8
A2	25.00	1.22	390	46.6			
A3	25.15	1.20	352	38.0	5.0	339	37.4
A4	25.12	1.23	325	36.8			
A5	25.27	1.30	192	27.4	3.5	269	32.1
A6	25.18	1.28	345	36.8			

4.3.9 Expanded cork granules 30 g/m^2 - B30

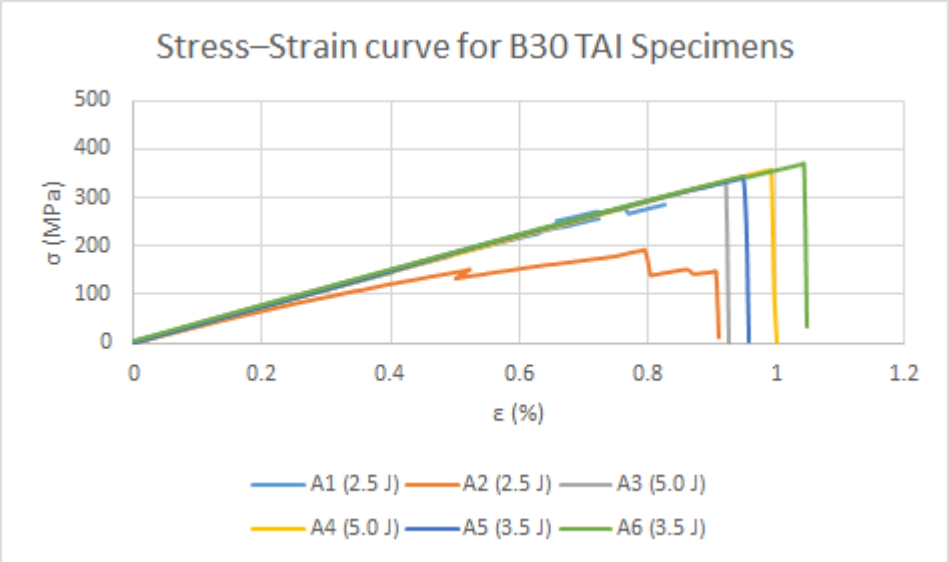


Figure 4.52: Stress–Strain curve for TAI B30 Specimens

Table 4.35: Dimensions and mechanical properties of B30 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus [GPa]	Energy [J]	UTS Average [MPa]	Young's Modulus Average [GPa]
A1	24.52	1.22	285	35.8	2.5	238	33.9
A2	24.43	1.22	192	32.0			
A3	24.81	1.22	335	38.3	5.0	346	37.4
A4	24.72	1.25	356	36.5			
A5	24.80	1.30	339	36.4	3.5	355	36.8
A6	24.93	1.30	372	37.1			

4.3.10 Expanded cork granules 40 g/m^2 - B40

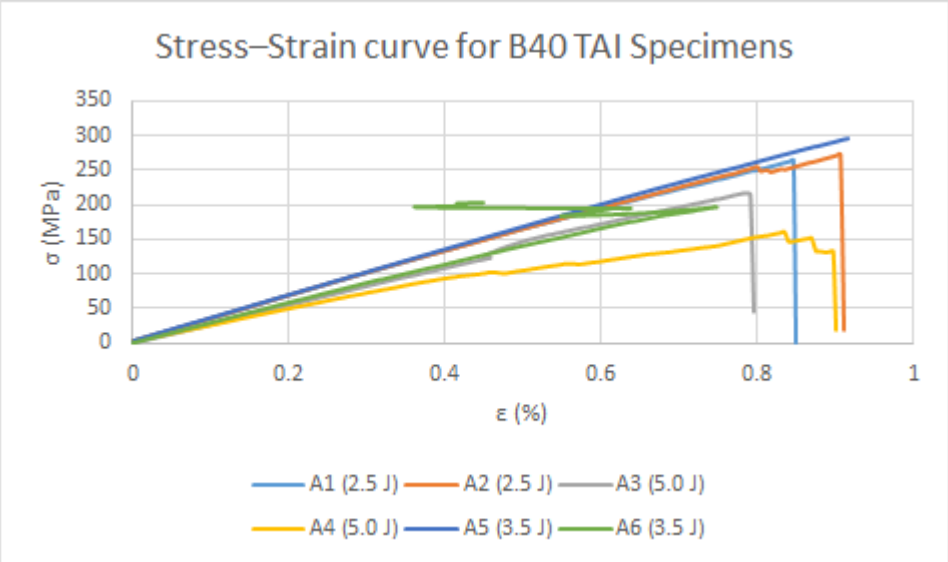


Figure 4.53: Stress–Strain curve for TAI B40 Specimens

Table 4.36: Dimensions and mechanical properties of B40 tensile tests' specimens

Specimen	Width b [mm]	Thickness h [mm]	UTS [MPa]	Young's Modulus [GPa]	Energy [J]	UTS Average [MPa]	Young's Modulus Average [GPa]
A1	24.40	1.32	265	33.0	2.5	269.27	31.31
A2	24.62	1.30	274	33.6			
A3	24.95	1.30	217	28.0	5.0	188.68	25.91
A4	24.75	1.35	160	23.9			
A5	24.80	1.37	296	33.3	3.5	249.08	31.07
A6	24.77	1.38	202	28.8			

4.3.11 Analysis of Results

Before the comparisons are made, some comments should be done concerning the individual tests. As it possible to see, in almost all the different laminates, the values obtained from the specimens of the same laminate are quite spread. This might have been due to the fact that the impact made caused different types of damage: along the specimens axis or transversal to it. The tensile test results are highly affected by this direction. It would be interesting to separate the specimens among the damage type, but since each laminate had only two specimens for each energy level, it was chosen not to do that study.

Comparison between C1, C2 and REF

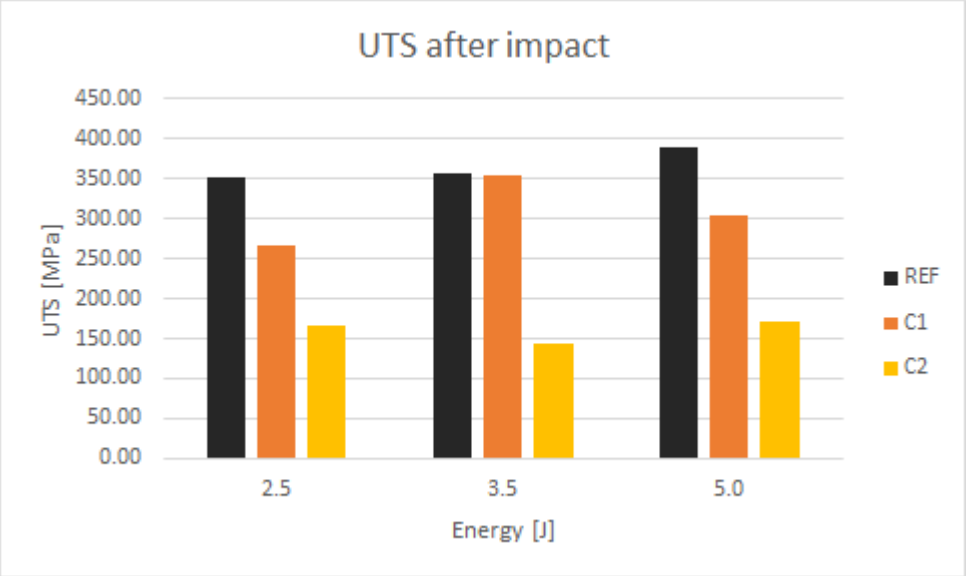


Figure 4.54: Ultimate Tensile Strength after impact comparison between C1, C2 and REF

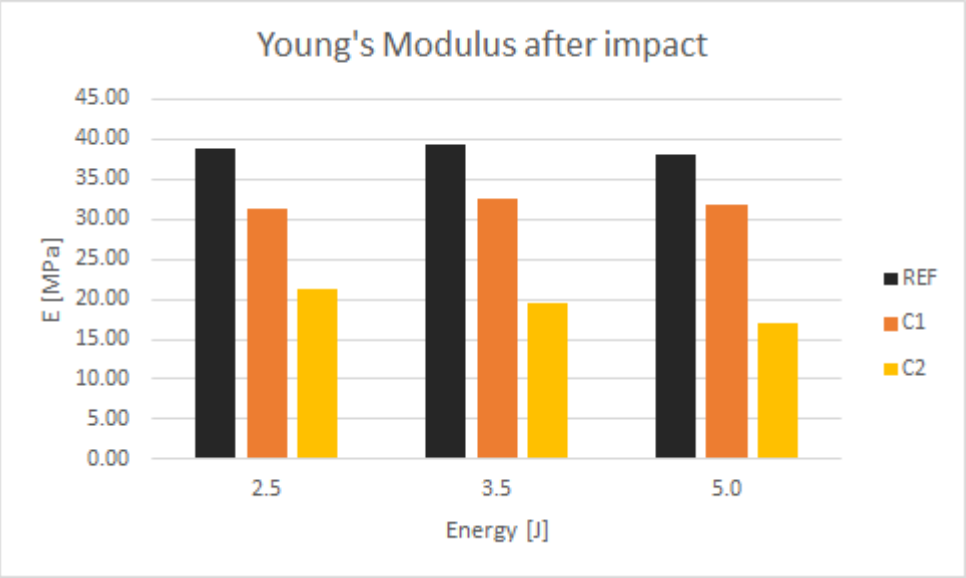


Figure 4.55: Young's modulus after impact comparison between C1, C2 and REF

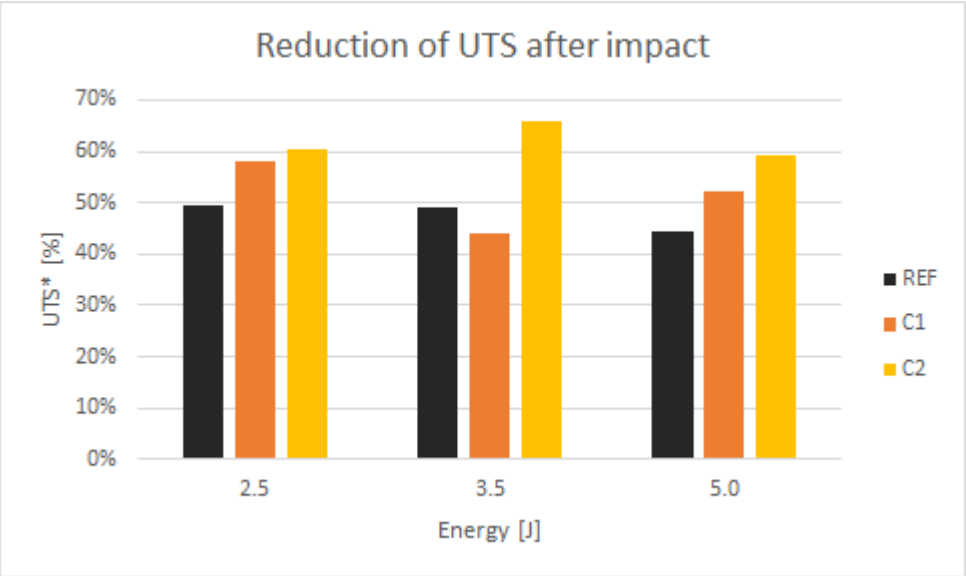


Figure 4.56: Reduction of Ultimate Tensile Strength after impact comparison between C1, C2 and REF

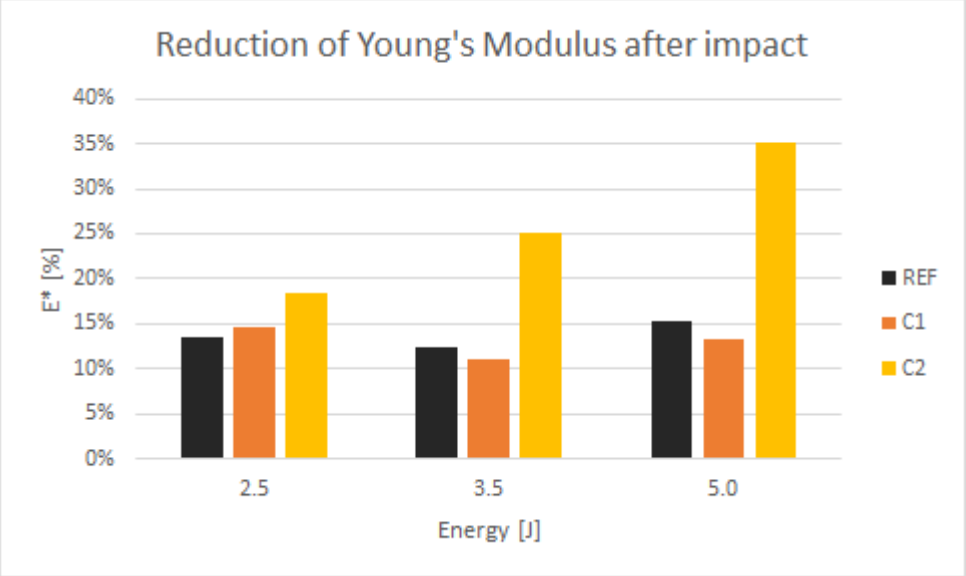


Figure 4.57: Reduction of Young's modulus after impact comparison between C1, C2 and REF

Table 4.37: Mechanical properties of REF, C1 and C2 and their reduction after impact

Laminate	Energy	Impacted		Not Impacted		Reduction	
		UTS	E	UTS	E	UTS	E
	[J]	[MPa]	[GPa]	[MPa]	[GPa]	[%]	[%]
REF	2.5	253	38.8	699	44.9	50	13
	3.5	357	39.3			49	12
	5.0	390	38.0			44	15
C1	2.5	267	31.3	634	36.7	58	15
	3.5	355	32.6			44	11
	5.0	303	31.8			52	13
C2	2.5	166	21.4	422	26.2	61	18
	3.5	144	19.6			66	25
	5.0	172	17.0			59	35

Figure 4.54, 4.55 and table 4.37, show that the reference laminate is the one, among the laminates presented here, that have the best mechanical properties (Ultimate Tensile Strength and Young's Modulus) after going through the impact energies used. Following the reference laminate, the best one is C1 (that in case of an impact of 3.5 J had almost the same properties of REF), and lastly comes C2. This verification follows the same pattern as the tensile tests mentioned in the previous section.

Regarding the properties' reduction, C2 is clearly the one that was the most affected by all the impacts. Regarding the difference between REF and C1, in case of UTS, C1 is capable of showing a lower reduction, for impacts of 3.5 J. About the Young's modulus, C1 only "loses" against REF for an impact of 2.5 J.

Comparison between K30, K40, K60 and REF

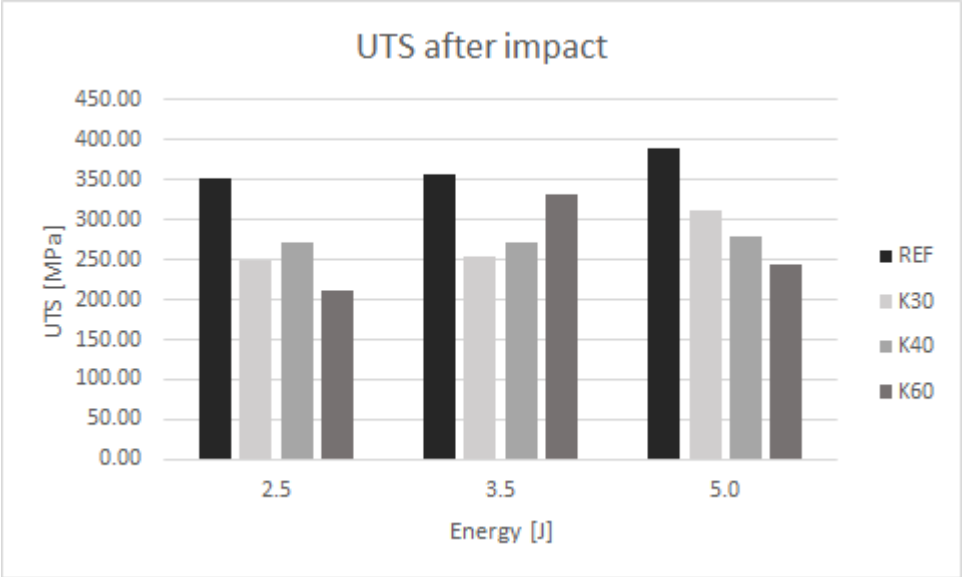


Figure 4.58: Ultimate Tensile Strength after impact comparison between K30, K40, K60 and REF

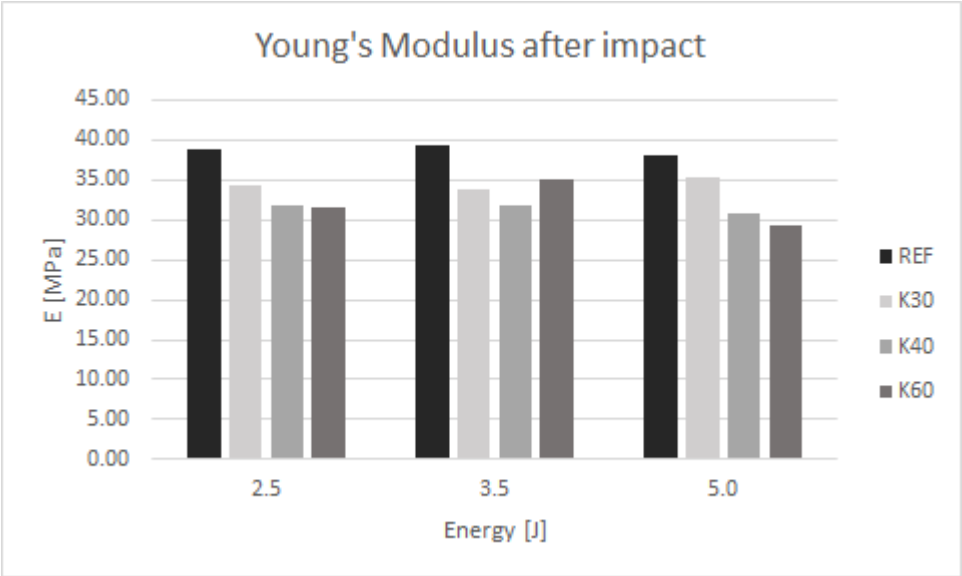


Figure 4.59: Young's modulus after impact comparison between K30, K40, K60 and REF

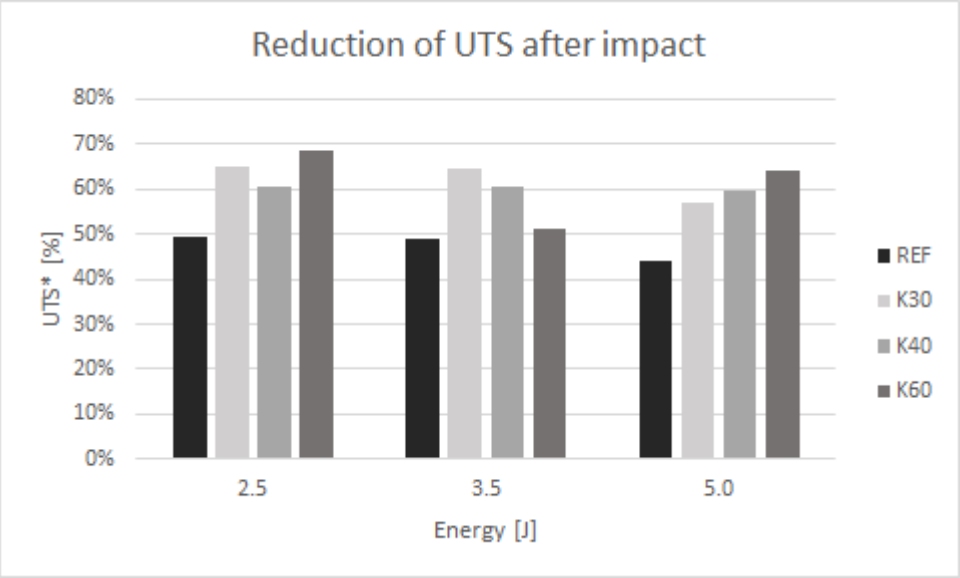


Figure 4.60: Reduction of Ultimate Tensile Strength after impact comparison between K30, K40, K60 and REF

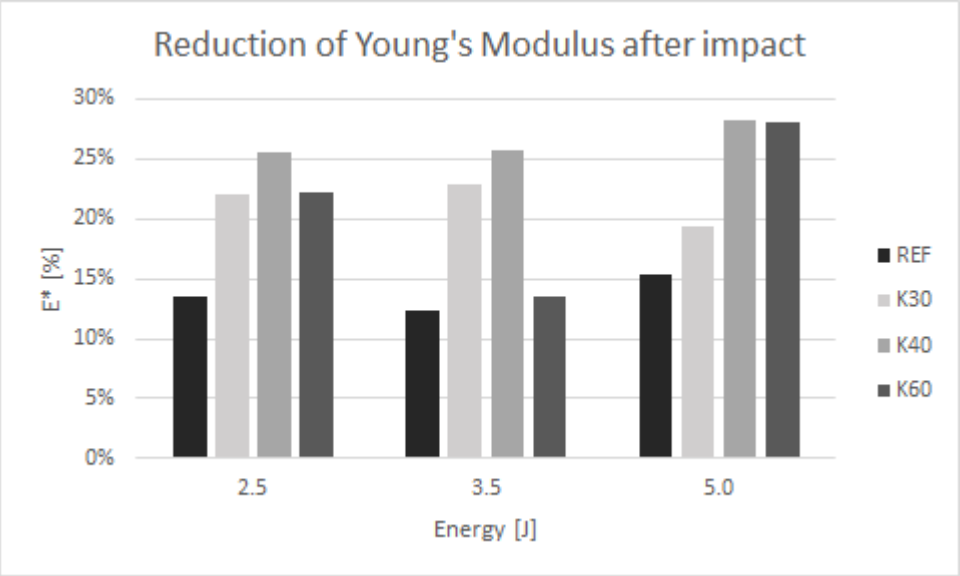


Figure 4.61: Reduction of Young's modulus after impact comparison between K30, K40, K60 and REF

Analysing figures 4.58 and 4.59, it is possible to verify that the reference laminate, after impacts of 2.5, 3.5 and 5 J, present higher UTS and Young's Modulus, even though K30 had higher UTS when analysing the without impact situations. It is not possible to take any conclusion about the influence of the Kraton™ granules' concentration, since it seems that there is no rule that can be modeled with these results.

Looking now at the reduction of the mechanical properties under study, the composite reference was capable of showing lower reduction comparing with the Kraton™ laminates. Again,

Table 4.38: Mechanical properties of REF, K30, K40 and K60 and their reduction after impact

Laminate	Energy	Impacted		Not Impacted		Reduction	
		UTS	E	UTS	E	UTS	E
	[J]	[MPa]	[GPa]	[MPa]	[GPa]	[%]	[%]
REF	2.5	253	38.8	699	44.9	50	13
	3.5	357	39.3			49	12
	5.0	390	38.0			44	15
K30	2.5	250	34.3	720	44	65	22
	3.5	255	33.9			65	23
	5.0	311	35.4			57	19
K40	2.5	271	31.8	690	42.8	61	26
	3.5	272	31.8			61	26
	5.0	279	36.7			60	28
K60	2.5	213	31.7	677	40.7	69	22
	3.5	312	35.2			51	14
	5.0	244	29.3			64	28

the results do not allow to establish a model between the granules concentration and the residual properties.

Comparison between B10, B20, B30, B40 and REF

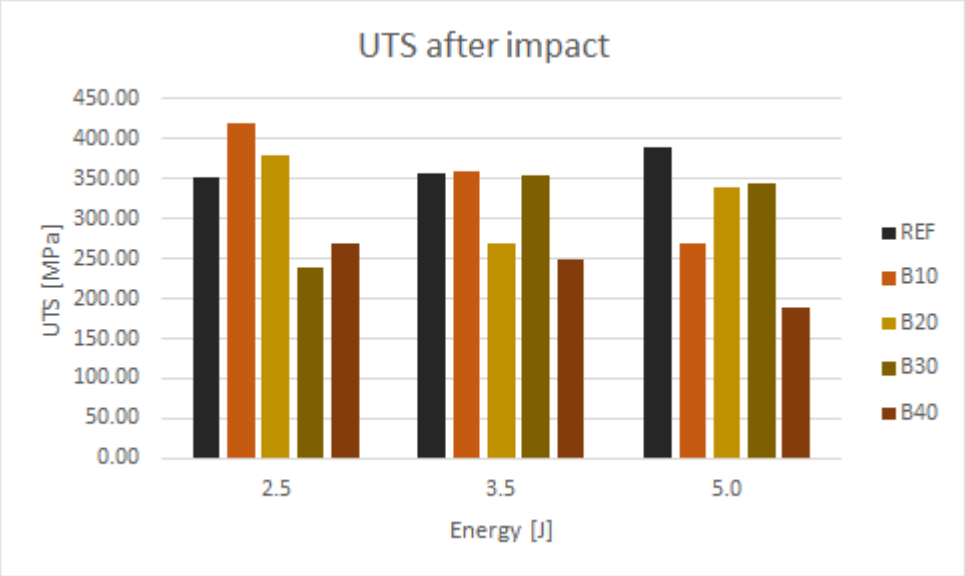


Figure 4.62: Ultimate Tensile Strength after impact comparison between B10, B20, B30, B40 and REF

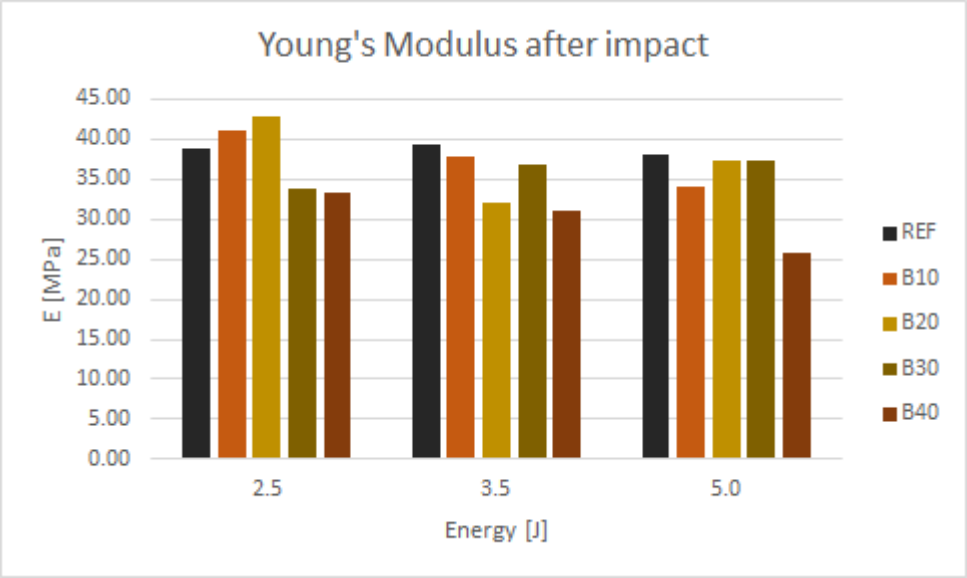


Figure 4.63: Young's modulus after impact comparison between B10, B20, B30, B40 and REF

The results from expanded cork granules are more exciting, in the sense that in some situations, the properties of the laminates with cork granules as interlayer managed to be higher than the reference, unlike what happened with the previous laminate groups.

Looking at figure 4.13 (UTS after impact), it is possible to see that B10 and B20 have a higher value for 2.5 J impact and, concerning 3.5 J of energy, B10 and B30 had similar values, but as the energy increases to 5.0 J, cork granules are not competitive enough to overcome the reference's results.

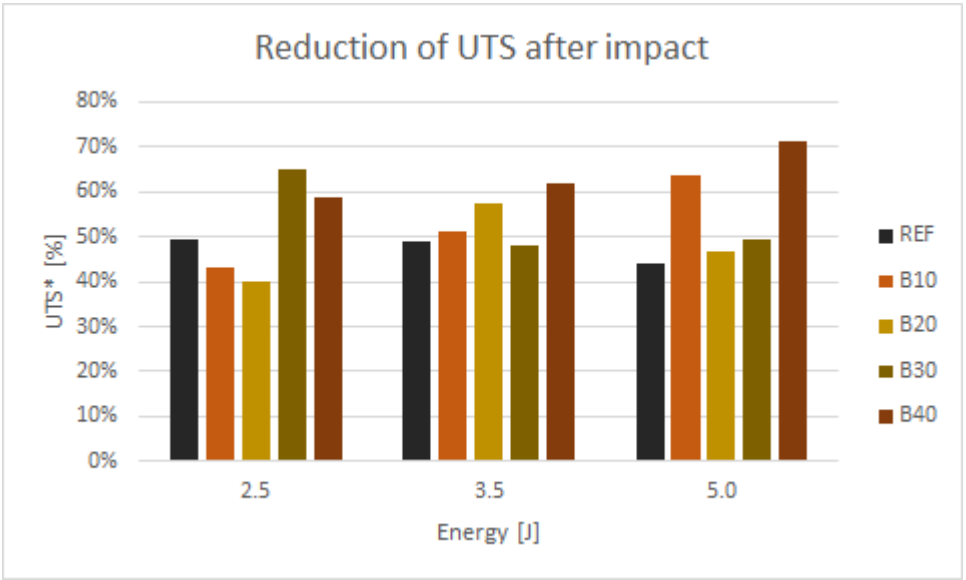


Figure 4.64: Reduction of Ultimate Tensile Strength after impact comparison between B10, B20, B30, B40 and REF

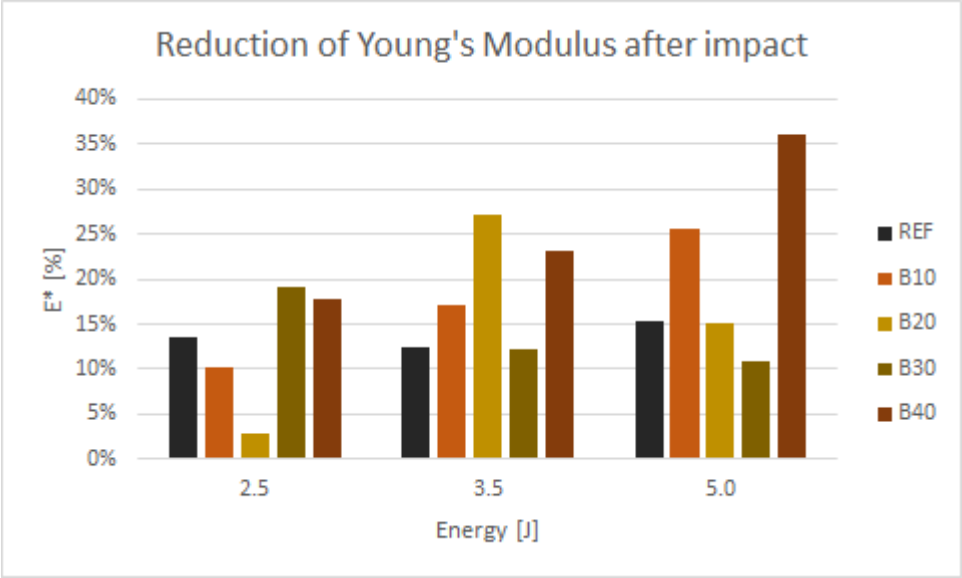


Figure 4.65: Reduction of Young's modulus after impact comparison between B10, B20, B30, B40 and REF

Table 4.39: Mechanical properties of REF, B10, B20, B30 and B40 and their reduction after impact

Laminate	Energy [J]	Impacted UTS [MPa]	Impacted E [GPa]	Not Impacted UTS [MPa]	Not Impacted E [GPa]	Reduction UTS [%]	Reduction E [%]
REF	2.5	253	38.8	699	44.9	50	13
	3.5	357	39.3			49	12
	5.0	390	38.0			44	15
B10	2.5	419	41.1	739	45.8	43	10
	3.5	360	37.9			51	17
	5.0	268	34.0			64	26
B20	2.5	380	42.8	634	44.1	40	3
	3.5	269	32.1			58	27
	5.0	389	37.4			47	15
B30	2.5	238	33.9	683	41.9	65	19
	3.5	355	36.8			48	12
	5.0	346	37.4			49	11
B40	2.5	269	33.3	654	40.5	59	18
	3.5	249	31.1			62	23
	5.0	189	25.9			71	36

Regarding the tensile strength, the reference laminate showed lower values when comparing with the other cork granules' laminates. It was only overcomed by B10 and B20 on 2.5 J of impact energy and slightly on 3.5 J by B30. Concerning the reduction of Young's Modulus, the qualitative results were quite similar. This time, B30 managed to have a considerable reduction for 5 J of impact energy.

Comparison between all

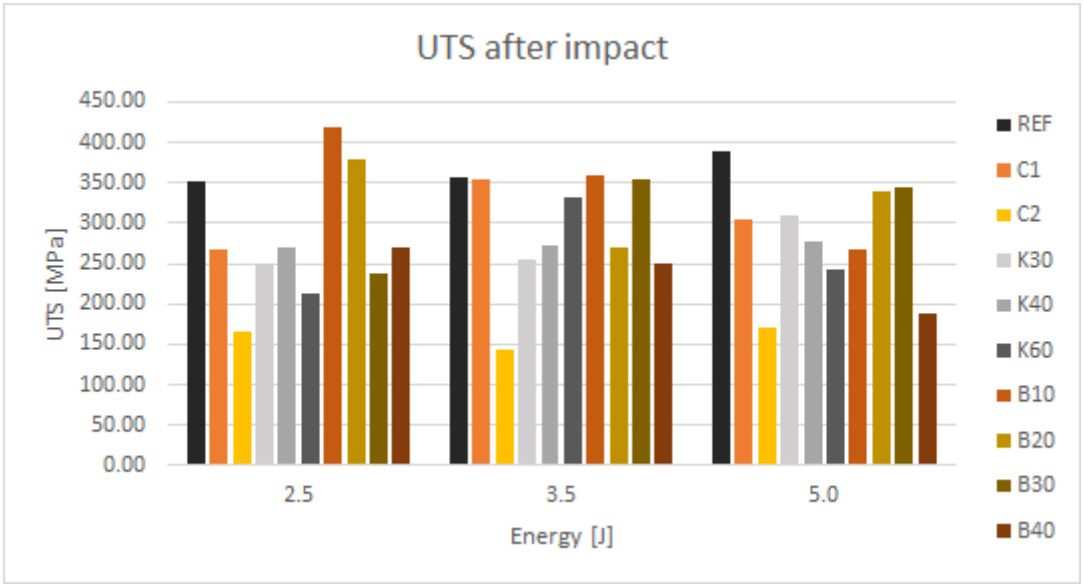


Figure 4.66: Ultimate Tensile Strength after impact comparison between all laminates

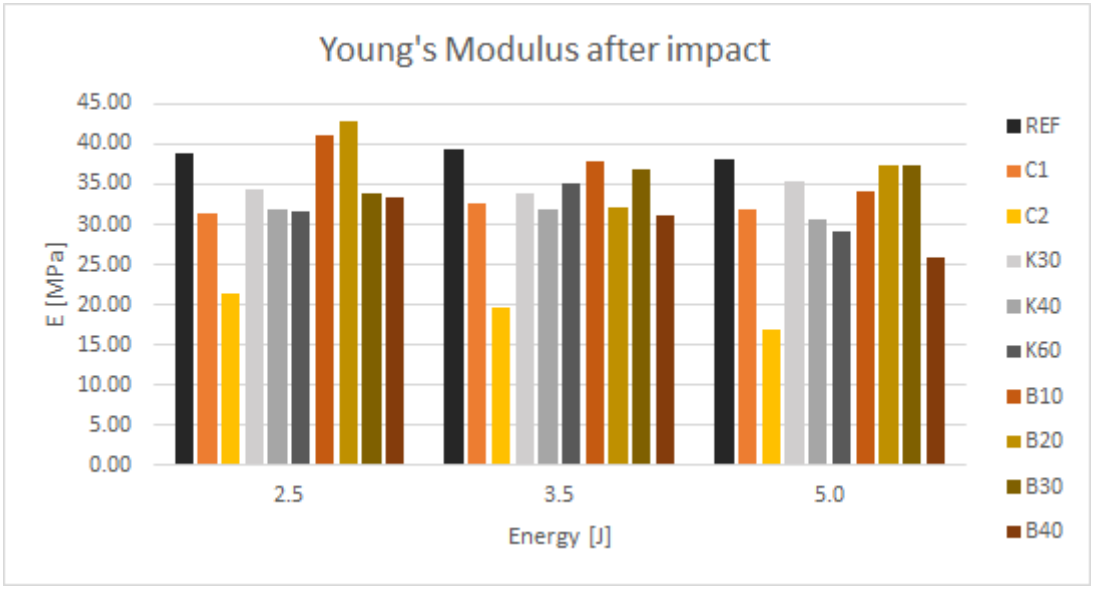


Figure 4.67: Young's modulus after impact comparison between all laminates

It is now presented a global comparison between all the laminates. Starting with the tensile strength after impact (Fig. 4.66), it is possible to see that the reference laminate has the ability to keep a good ultimate tensile strength, despite the fact that is was overcomed by some other laminates. In case of the specimens that suffered an impact of 2.5 J of energy, B10 was the one that showed the higher value. After B10, it was B20 and then REF, still with a good advantage over the following. In the case of impacts of 3.5 J, B10 keeps being the most resistant to tensile

stresses, but this time with a lower advantage over REF, B30 and C1 (by descending order), despite all of them having close values from each other. Analysing now the 5 J impacted specimens, REF was capable of keeping a wide advantage over the rest, where B30 and B20 were able to approach the value practiced by the reference laminate. It is also important to point out that C2 laminate had the lowest tensile strength of all the laminates.

Looking now at the Young's modulus after impact results (Fig. 4.67), it is verified that the reference composite keeps being, of all the composites studied, the one that present the highest value for this property. In case of impacts of 2.5 J, B10 and B20 were capable of overcoming the property of the reference laminate. For the 3.5 and 5.0 J impact, none of the other solutions studied was able to match the reference, despite B10 (for 3.5 J), B20 and B30 (for 5.0 J) being relatively close.

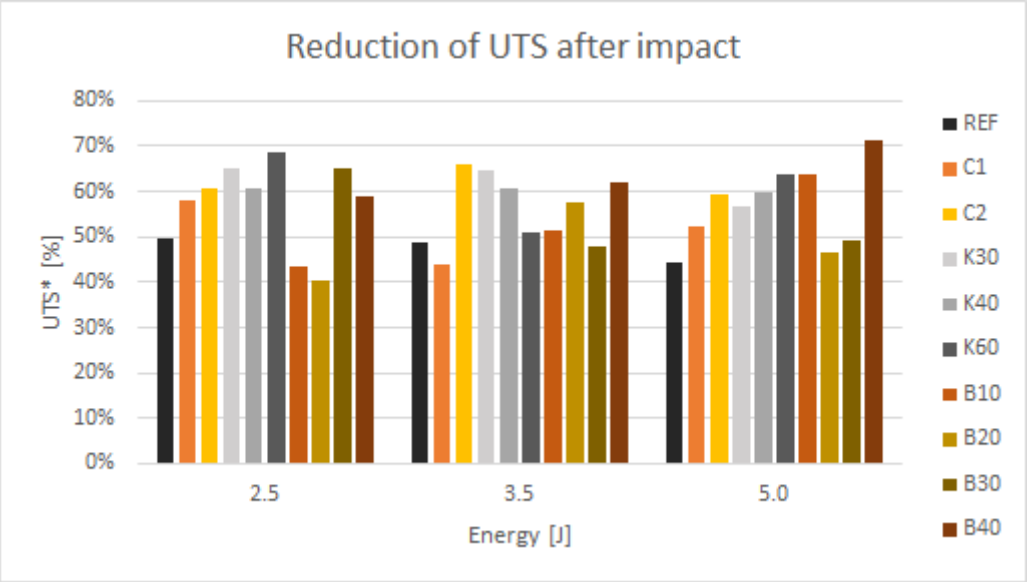


Figure 4.68: Reduction of Ultimate Tensile Strength after impact comparison between all laminates

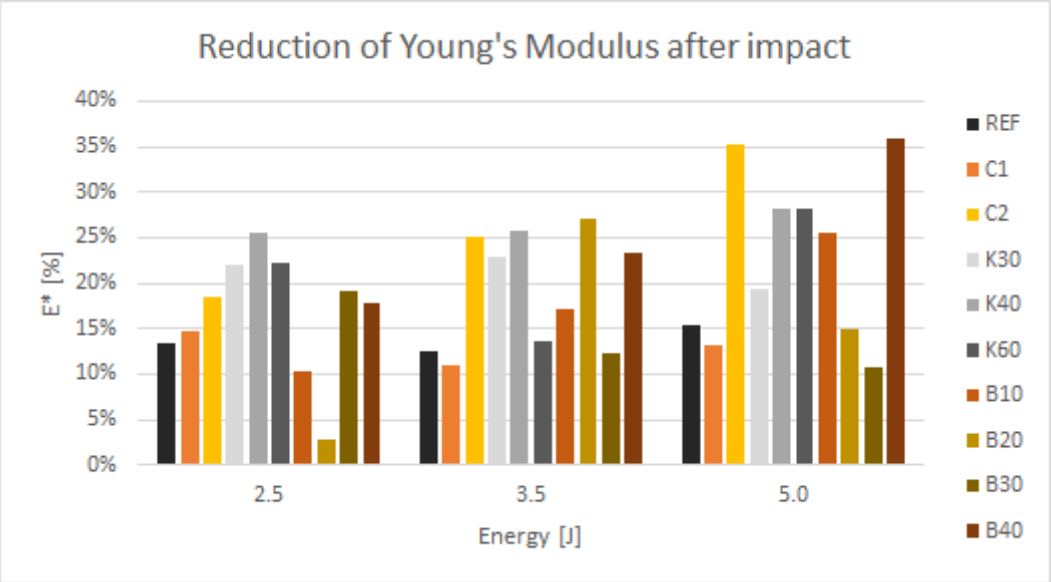


Figure 4.69: Reduction of Young's modulus after impact comparison between B10, B20, B30, B40 and REF

Table 4.40: Reduction of the mechanical properties with an impact of 2.5 J

2.5 J	**Laminate**									
	REF	C1	C2	K30	K40	K60	B10	B20	B30	B40
UTS (%)	50	58	61	65	61	69	43	40	65	59
E (%)	13	15	18	22	26	22	10	3	19	18

Table 4.41: Reduction of the mechanical properties with an impact of 3.5 J

3.5 J	**Laminate**									
	REF	C1	C2	K30	K40	K60	B10	B20	B30	B40
UTS (%)	49	44	66	65	61	51	51	58	48	62
E (%)	12	11	25	23	26	14	17	27	12	23

Table 4.42: Reduction of the mechanical properties with an impact of 5.0 J

5.0 J	**Laminate**									
	REF	C1	C2	K30	K40	K60	B10	B20	B30	B40
UTS (%)	44	52	59	57	60	64	64	47	49	71
E (%)	15	13	35	19	28	28	26	15	11	36

The reduction of Young's modulus and ultimate tensile strength with an impact can be consulted on the figures 4.68 and 4.69 and on tables 4.40, 4.41 and 4.42.

When the impact is of 2.5 J, only B10 and B20 present smaller reductions than the reference, implying that the addition of cork granules, until a certain concentration, can not just improve the mechanical properties, but also improve the residual properties in low energy impacts.

The tensile test after a 3.5 J impact, lead to just C1 laminate, among all studied, to be the one to have a lower reduction than the 49% of UTS without impact reference laminate. On what Young's modulus is concerned, C1 laminates stand out again with just 11% reduction, following B30 with just 12%.

Lastly, for 5.0 J impact, the reference laminate kept presenting lower values of reduction, with 44% for UTS, despite the fact that B20 and B30 show near values. For the Young's modulus, B30 is able to present the smallest percentage, followed by B20 and then C1, that presented smaller percentages than the reference, showing again that by adding cork granules can improve this residual property.

As a final comment, one important constatation to be made by analysing table 4.43, is that Young's modulus is not as affected by the impact as the ultimate tensile strength.

Altough a comparison between the values was made, it is not possible to take conclusions from this test for the reasons aforementioned. A way to solve this problem is by producing way more specimens and divide them according with the damage type. Another comment can be made regarding improvement points, which is the usage of lower impact energies. For the energies used, the damage might have reached the interlayers, which might have propagated transversally.

Table 4.43: Mechanical properties of the laminates and their reduction after impact

Laminate	Energy	Impacted		Not Impacted		Reduction	
		UTS	E	UTS	E	UTS	E
	[J]	[MPa]	[GPa]	[MPa]	[GPa]	[%]	[%]
	2.5	253	38.8			50	13
REF	3.5	357	39.3	699	44.9	49	12
	5.0	390	38.0			44	15
	2.5	267	31.3			58	15
C1	3.5	355	32.6	634	36.7	44	11
	5.0	303	31.8			52	13
	2.5	166	21.4			61	18
C2	3.5	144	19.6	422	26.2	66	25
	5.0	171	17.0			59	35
	2.5	250	34.3			65	22
K30	3.5	255	33.9	720	44.0	65	23
	5.0	311	35.4			57	19
	2.5	271	31.8			61	26
K40	3.5	272	31.8	689.7	42.8	61	26
	5.0	279	36.7			60	28
	2.5	213	31.7			69	22
K60	3.5	312	35.2	677	40.7	51	14
	5.0	244	29.3			64	28
	2.5	419	41.1			43	10
B10	3.5	360	37.9	739	45.8	51	17
	5.0	268	34.0			64	26
	2.5	380	42.8			40	3
B20	3.5	269	32.1	634	44.1	58	27
	5.0	389	37.4			47	15
	2.5	238	33.9			65	19
B30	3.5	355	36.8	683	41.9	48	12
	5.0	346	37.4			49	11
	2.5	269	33.3			59	18
B40	3.5	249	31.1	654	40.5	62	23
	5.0	189	25.9			71	36

4.4 Identation Tests

As it was mentioned before, this test consists in measuring the indentation of the impacted specimen right after impact, one day after, one week after and one month after. Due to some time

constraints it was not possible to measure all the specimens one month after the impact. In these situations there is no reference to any measurement.

By measuring these values, it is possible to understand how the indentation varies with respect to the impact energy and the interlayer material used. By measuring its evolution over time, it is possible to evaluate its recovery as the time goes by.

This test has also some constraints. The values might have an associated error both because of the system used to measure and the fact that the energies were high for this type of test, ending up causing a lot of damage to the specimen and making it hard to take measurements. Most of the specimens impacted with 13 J got pierced, making it impossible and not interesting to measure.

4.4.1 Results

4.4.1.1 Reference - REF

Table 4.44: Indentation results for REF specimens

Time	Energy	Specimen	Average 0 Level	Center	Identation	Average
After Impact	5 J	4	16.99	16.23	0.76	0.75
		5	16.91	16.09	0.82	
		6	16.97	16.29	0.68	
	8 J	1	15.38	13.16	2.22	2.07
		2	15.39	13.57	1.82	
		3	15.36	13.19	2.17	
	13 J	7	pierced			
		8	pierced			
1 Day after	5 J	4	9.48	8.62	0.86	0.86
		5	9.42	8.51	0.91	
		6	9.50	8.70	0.80	
	8 J	1	9.46	7.47	1.99	1.91
		2	9.47	7.47	1.67	
		3	9.46	7.80	2.06	
	13 J	7	pierced			
		8	pierced			
1 Week after	5 J	4	23.02	22.25	0.77	0.75
		5	22.95	22.15	0.80	
		6	22.99	22.30	0.69	
	8 J	1	22.96	20.92	2.04	1.87
		2	23.01	21.38	1.63	
		3	22.96	21.01	1.95	
	13 J	7	pierced			
		8	pierced			
1 Month after	5 J	4	8.23	7.52	0.71	1.32
		5	8.88	7.22	1.66	
		6	8.94	7.34	1.60	
	8 J	1	8.13	6.53	1.60	1.63
		2	8.23	6.77	1.49	
		3	8.16	6.35	1.81	
	13 J	7	pierced			
		8	pierced			

Table 4.45: Indentation evolution for REF specimens

Energy	Indentation Evolution				Percentage		
	After impact	1 Day after	1 Week after	1 Month after	1 Day after	1 Week after	1 Month after
5	0.85	0.86	0.753	0.752	-0.98%	11.20%	11.38%
8	2.07	1.91	1.87	1.63	7.74%	9.56%	20.94%

4.4.1.2 Thin Cork Film - C1

Table 4.46: Indentation results for C1 specimens

Time	Energy	Specimen	Average 0 Level	Center	Identation	Average
After Impact	5 J	4	15.88	15.19	0.69	0.67
		5	15.88	15.17	0.71	
		6	16.43	15.81	0.62	
	8 J	1	15.65	14.11	1.54	1.32
		2	15.77	14.35	1.42	
		3	15.84	14.84	1.00	
	13 J	7	pierced			
		8	pierced			
1 Day after	5 J	4	10.04	9.36	0.68	0.63
		5	10.02	9.40	0.62	
		6	10.07	9.46	0.61	
	8 J	1	9.87	8.42	1.45	1.19
		2	9.97	8.77	1.20	
		3	10.01	9.08	0.93	
	13 J	7	pierced			
		8	pierced			
1 Week after	5 J	4	22.88	22.28	0.60	0.57
		5	22.87	22.28	0.59	
		6	22.94	22.42	0.51	
	8 J	1	23.12	21.80	1.32	1.40
		2	23.22	22.11	1.11	
		3	23.19	21.41	1.78	
	13 J	7	pierced			
		8	pierced			
1 Month after	5 J	4	8.31	7.81	0.50	0.50
		5	8.30	7.79	0.51	
		6	8.34	7.85	0.49	
	8 J	1	8.14	6.92	1.22	0.97
		2	8.21	7.34	0.88	
		3	8.28	7.46	0.82	
	13 J	7	pierced			
		8	pierced			

Table 4.47: Indentation evolution for C1 specimens

Energy	Indentation Evolution				Percentage		
	After impact	1 Day after	1 Week after	1 Month after	1 Day after	1 Week after	1 Month after
5	0.67	0.63	0.57	0.50	6.06%	15.64%	26.02%
8	1.32	1.19	1.40	0.97	9.80%	-6.48%	26.29%

4.4.1.3 Thick Cork Film - C2

Table 4.48: Indentation results for C2 specimens

Time	Energy	Specimen	Average 0 Level	Center	Identation	Average
After Impact	5 J	4	16.43	15.78	0.65	0.60
		5	16.34	15.78	0.56	
	8 J	1	16.93	15.73	1.20	1.14
		2	16.96	15.90	1.06	
		3	17.11	15.94	1.17	
	13 J	6	17.08	14.40	2.68	2.41
		7	17.10	14.95	2.15	
		8	invalid			
1 Day After	5 J	4	10.69	10.07	0.62	0.53
		5	10.46	10.02	0.44	
	8 J	1	10.61	9.53	1.08	1.05
		2	10.63	9.68	0.95	
		3	10.64	9.50	1.14	
	13 J	6	9.96	7.25	2.71	2.41
		7	10.03	8.06	1.91	
		8	invalid			
1 Week After	5 J	4	23.54	22.95	0.59	0.49
		5	23.45	23.05	0.40	
	8 J	1	23.47	22.39	1.08	1.04
		2	23.44	22.52	0.92	
		3	23.46	22.35	1.11	
	13 J	6	23.57	20.98	2.59	2.25
		7	23.58	21.67	1.91	
		8	invalid			
1 Month After	5 J	4	8.98	8.42	0.56	0.52
		5	8.90	8.41	0.49	
	8 J	1	8.88	7.95	0.93	0.84
		2	8.93	8.25	0.68	
		3	8.92	8.01	0.91	
	13 J	6	8.95	6.37	2.58	2.16
		7	8.97	7.23	1.74	
		8	invalid			

Table 4.49: Indentation evolution for C2 specimens

Energy	Indentation Evolution				Percentage		
	After impact	1 Day after	1 Week after	1 Month after	1 Day after	1 Week after	1 Month after
5	0.60	0.53	0.49	0.52	16.13%	11.06%	8.10%
8	1.14	1.05	1.04	0.84	8.92%	10.42%	30.33%
13	2.41	2.34	2.25	2.16	7.00%	16.50%	25.03%

4.4.1.4 Kraton™ granules 30 g/m^2 - K30

Table 4.50: Indentation results for K30 specimens

Time	Energy	Specimen	Average 0 Level	Center	Identation	Average
After Impact	5 J	4	15.71	14.57	1.14	1.02
		5	15.46	14.38	1.08	
		6	15.65	14.80	0.85	
	8 J	1	15.37	13.02	2.35	2.22
		2	15.50	13.22	2.28	
		3	15.46	13.44	2.02	
	13 J	7	pierced			
		8	pierced			
1 Day after	5 J	4	9.78	8.61	1.17	1.00
		5	9.58	8.59	0.99	
		6	9.75	8.90	0.85	
	8 J	1	9.64	7.35	2.11	1.97
		2	9.64	7.49	2.15	
		3	9.58	7.92	1.66	
	13 J	7	pierced			
		8	pierced			
1 Week after	5 J	4	23.32	22.52	0.80	0.84
		5	23.10	22.15	0.95	
		6	23.28	22.50	0.78	
	8 J	1	23.00	21.06	1.94	1.40
		2	23.15	21.17	1.98	
		3	23.08	21.49	1.59	
	13 J	7	pierced			
		8	pierced			
1 Month after	5 J	4	8.38	7.67	0.71	0.74
		5	8.38	7.25	1.13	
		6	8.13	7.73	0.40	
	8 J	1	22.50	20.75	1.75	1.46
		2	22.97	21.92	1.06	
		3	22.93	21.35	1.58	
	13 J	7	pierced			
		8	pierced			

Table 4.51: Indentation evolution for K30 specimens

Energy	Indentation Evolution				Percentage		
	After impact	1 Day after	1 Week after	1 Month after	1 Day after	1 Week after	1 Month after
5	1.02	1.00	0.84	0.74	2.04%	17.75%	27.30%
8	2.22	1.97	1.83	1.46	10.98%	17.26%	14.17%

4.4.1.5 Kraton™ granules 40 g/m^2 - K40

Table 4.52: Indentation results for K40 specimens

Time	Energy	Specimen	Average 0 Level	Center	Identation	Average
After Impact	5 J	4	15.81	14.86	0.95	0.88
		5	15.70	14.79	0.91	
		6	15.79	15.01	0.78	
	8 J	1	15.58	13.80	1.78	1.83
		2	15.75	13.83	1.92	
		3	15.72	13.93	1.79	
	13 J	7	15.52	13.01	2.51	2.51
		8	pierced			
1 Day after	5 J	4	9.85	9.04	0.81	0.73
		5	10.10	9.23	0.87	
		6	10.20	9.39	0.81	
	8 J	1	9.56	7.90	1.66	1.63
		2	9.65	7.95	1.70	
		3	9.66	8.13	1.53	
	13 J	7	10.10	8.88	1.22	1.22
		8	pierced			
1 Week after	5 J	4	23.12	22.29	0.83	0.83
		5	23.04	22.03	1.01	
		6	23.07	22.50	0.57	
	8 J	1	23.06	21.57	1.49	1.51
		2	23.06	21.52	1.54	
		3	23.07	21.56	1.51	
	13 J	7	23.35	21.33	2.02	2.02
		8	pierced			
1 Month after	5 J	4	8.18	7.46	0.72	0.66
		5	8.09	7.32	0.77	
		6	8.16	7.65	0.51	
	8 J	1	8.10	6.65	1.45	1.46
		2	8.32	6.72	1.59	
		3	8.11	6.78	1.33	
	13 J	7	pierced			
		8	pierced			

Table 4.53: Indentation evolution for K40 specimens

Energy	Indentation Evolution				Percentage		
	After impact	1 Day after	1 Week after	1 Month after	1 Day after	1 Week after	1 Month after
5	0.88	0.83	0.80	0.66	5.50%	8.72%	24.40%
8	1.83	1.63	1.51	1.46	11.07%	17.35%	20.33%

4.4.1.6 Kraton™ granules 60 g/m^2 - K60

Table 4.54: Indentation results for K60 specimens

Time	Energy	Specimen	Average 0 Level	Center	Identation	Average
After Impact	5 J	4	17.86	17.09	0.77	0.80
		5	17.85	17.06	0.79	
		6	17.88	17.05	0.83	
	8 J	1	17.77	15.92	1.85	2.15
		2	17.83	15.75	2.05	
		3	17.87	15.36	2.51	
	13 J	7	pierced			
		8	pierced			
1 Day after	5 J	4	12.57	11.89	0.67	0.70
		5	12.59	11.89	0.70	
		6	12.59	11.86	0.73	
	8 J	1	12.21	10.71	1.50	1.92
		2	11.54	9.59	1.95	
		3	12.58	10.26	2.32	
	13 J	7	pierced			
		8	pierced			
1 Week after	5 J	4	10.45	9.83	0.62	0.64
		5	10.49	9.86	0.63	
		6	10.47	9.81	0.66	
	8 J	1	10.36	8.85	1.51	1.85
		2	10.37	8.47	1.90	
		3	9.05	6.90	2.15	
	13 J	7	pierced			
		8	pierced			

Table 4.55: Indentation evolution for K60 specimens

Energy	Indentation Evolution			Percentage	
	After impact	1 Day after	1 Week after	1 Day after	1 Week after
5	0.80	0.70	0.64	12.27%	20.18%
8	2.15	1.92	1.85	10.53%	13.73%

4.4.1.7 Expanded cork granules 10 g/m^2 - B10

Table 4.56: Indentation results for B10 specimens

Time	Energy	Specimen	Average 0 Level	Center	Identation	Average
After Impact	5 J	4	17.76	16.96	0.77	0.80
		5	17.74	16.75	0.99	
		6	17.78	16.93	0.85	
	8 J	1	17.70	15.58	2.12	2.25
		2	17.73	15.49	2.24	
		3	17.74	15.34	2.40	
	13 J	7	pierced			
		8	pierced			
1 Day after	5 J	4	12.47	11.81	0.66	0.78
		5	12.89	12.01	0.88	
		6	12.93	12.41	0.79	
	8 J	1	12.39	10.62	1.77	1.92
		2	12.44	10.39	2.05	
		3	12.44	10.47	1.97	
	13 J	7	pierced			
		8	pierced			
1 Week after	5 J	4	10.44	9.76	0.68	0.73
		5	10.41	9.66	0.75	
		6	10.44	9.67	0.77	
	8 J	1	10.36	8.60	1.76	1.89
		2	10.38	8.40	1.98	
		3	10.40	8.47	1.93	
	13 J	7	pierced			
		8	pierced			

Table 4.57: Indentation evolution for B10 specimens

Energy	Indentation Evolution			Percentage	
	After impact	1 Day after	1 Week after	1 Day after	1 Week after
5	0.88	0.78	0.73	11.65%	17.18%
8	2.25	1.93	1.89	14.31%	16.17%

4.4.2 Expanded cork granules 20 g/m^2 - B20

Table 4.58: Indentation results for B20 specimens

Time	Energy	Specimen	Average 0 Level	Center	Identation	Average
After Impact	5 J	4	17.79	16.94	0.85	0.85
		5	17.78	16.97	0.81	
		6	17.82	16.92	0.90	
	8 J	1	17.82	15.42	2.40	2.55
		2	17.74	15.00	2.74	
		3	17.76	15.25	2.51	
	13 J	7	pierced			
		8	pierced			
1 Day after	5 J	4	12.92	12.14	0.78	0.75
		5	12.91	12.24	0.67	
		6	12.98	12.17	0.81	
	8 J	1	12.93	10.72	2.21	2.22
		2	12.91	10.42	2.49	
		3	12.92	10.97	1.94	
	13 J	7	pierced			
		8	pierced			
1 Week after	5 J	4	10.38	9.63	0.75	0.73
		5	10.36	9.68	0.68	
		6	10.42	9.67	0.75	
	8 J	1	10.36	8.19	2.17	1.84
		2	10.34	8.83	1.51	
		3	10.36	8.50	1.86	
	13 J	7	pierced			
		8	pierced			

Table 4.59: Indentation evolution for B20 specimens

Energy	Indentation Evolution			Percentage	
	After impact	1 Day after	1 Week after	1 Day after	1 Week after
5	0.85	0.75	0.73	11.48%	14.73%
8	2.55	2.22	1.84	13.15%	27.68%

4.4.2.1 Expanded cork granules 30 g/m^2 - B30

Table 4.60: Indentation results for B30 specimens

Time	Energy	Specimen	Average 0 Level	Center	Identation	Average
After Impact	5 J	4	17.81	17.05	0.76	0.73
		5	17.83	17.11	0.72	
		6	17.87	17.17	0.70	
	8 J	1	17.87	15.60	2.27	2.09
		2	17.85	16.10	1.75	
		3	17.83	15.58	2.25	
	13 J	7	pierced			
		8	pierced			
1 Day after	5 J	4	12.99	12.30	0.69	0.64
		5	12.99	12.34	0.65	
		6	13.03	12.43	0.60	
	8 J	1	13.02	10.94	2.08	1.95
		2	13.01	11.41	1.60	
		3	13.02	10.86	2.16	
	13 J	7	pierced			
		8	pierced			
1 Week after	5 J	4	10.41	9.78	0.63	0.64
		5	10.40	9.76	0.64	
		6	10.45	9.80	0.65	
	8 J	1	10.42	8.43	1.99	1.87
		2	10.40	8.87	1.53	
		3	10.46	8.36	2.10	
	13 J	7	pierced			
		8	pierced			

Table 4.61: Indentation evolution for B30 specimens

Energy	Indentation Evolution			Percentage	
	After impact	1 Day after	1 Week after	1 Day after	1 Week after
5	0.73	0.65	0.64	11.39%	12.07%
8	2.09	1.95	1.87	6.90%	10.62%

4.4.2.2 Expanded cork granules 40 g/m^2 - B40

Table 4.62: Indentation results for B40 specimens

Time	Energy	Specimen	Average 0 Level	Center	Identation	Average
After Impact	5 J	4	17.83	16.94	0.89	0.85
		5	17.84	16.92	0.92	
		6	17.84	17.09	0.75	
	8 J	1	17.79	15.41	2.37	2.38
		2	17.89	15.66	2.23	
		3	17.82	15.30	2.52	
	13 J	7	pierced			
		8	pierced			
1 Day after	5 J	4	12.94	12.23	0.71	0.76
		5	12.96	12.10	0.86	
		6	12.98	12.27	0.71	
	8 J	1	12.93	10.73	2.20	2.22
		2	13.07	11.01	2.06	
		3	13.02	10.62	2.40	
	13 J	7	pierced			
		8	pierced			
1 Week after	5 J	4	11.36	10.65	0.71	0.71
		5	11.33.39	10.72	0.67	
	8 J	1	11.29	9.08	2.21	2.14
		2	11.36	9.46	1.91	
		3	11.35	9.05	2.30	
	13 J	7	pierced			
		8	pierced			

Table 4.63: Indentation evolution for B40 specimens

Energy	Indentation Evolution			Percentage	
	After impact	1 Day after	1 Week after	1 Day after	1 Week after
5	0.85	0.76	0.69	10.37%	19.17%
8	2.38	2.22	2.14	6.27%	10.02%

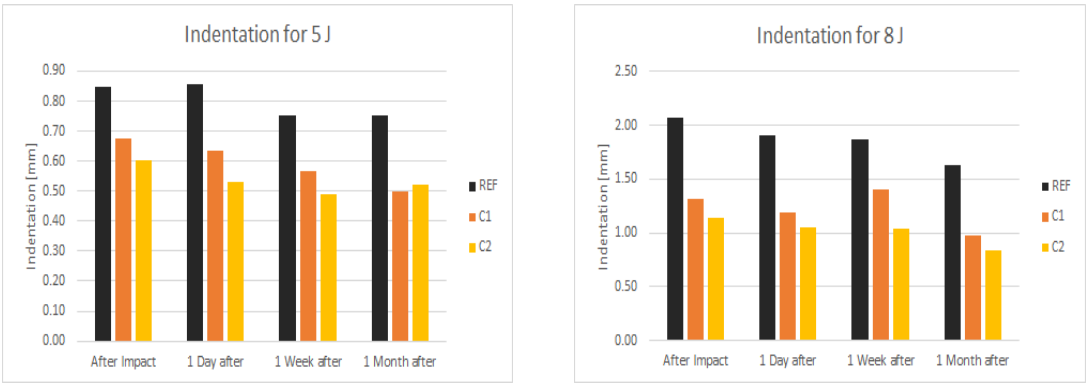
4.4.3 Analysis of Results

Comparison between C1, C2 and REF

Regarding the indentation for impact energies of 5 and 8 J (Fig. 4.70), REF has the biggest value, followed by C1 and then C2. This sequence repeats over time, and thus the results are the same with permanent indentation. By having the lowest indentation value, C2 shows less superficial damage, but on the other hand, REF has the advantage of making it easier to identify damage on

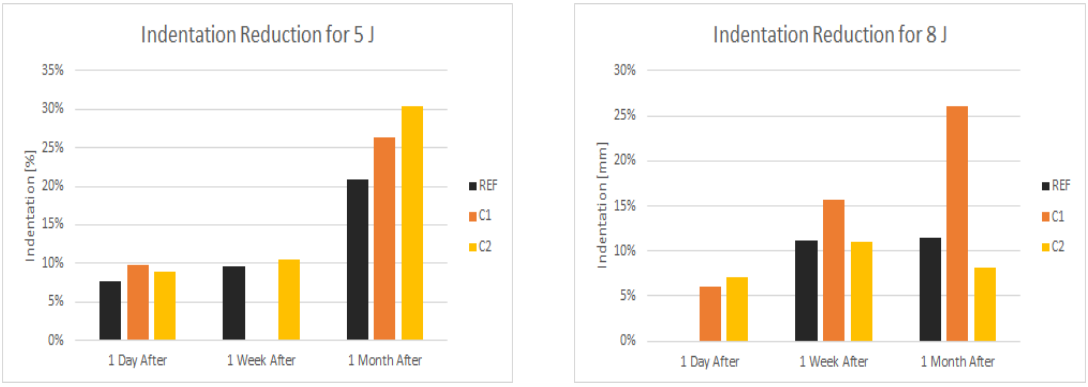
the surface. Also, the results show that by having a thicker cork film, the indentation is lower. Since there is only two thicknesses, it is not possible to reach an optimum thickness value, since it might be over the thickness of C2 or somewhere between C1 and C2.

Concerning the restitution (Fig. 4.71b), it is not possible to reach a final model for the results, but by adding the cork film, the recovery of part of the indentation is higher. By looking the the Figs 4.71, it is possible to see that this is not true only in a couple of situations.



(a) Indentation for 5 J impact energy (b) Indentation for 8 J impact energy

Figure 4.70: Indentation for C1, C2 and REF over time



(a) Indentation reduction for 5 J impact energy (b) Indentation reduction for 8 J impact energy

Figure 4.71: Indentation reduction for C1, C2 and REF over time

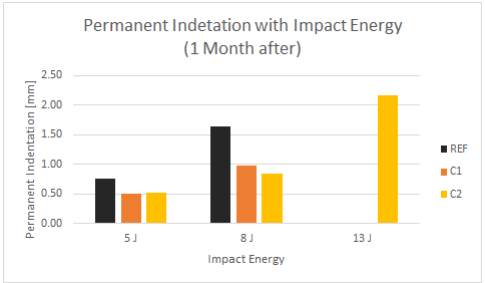
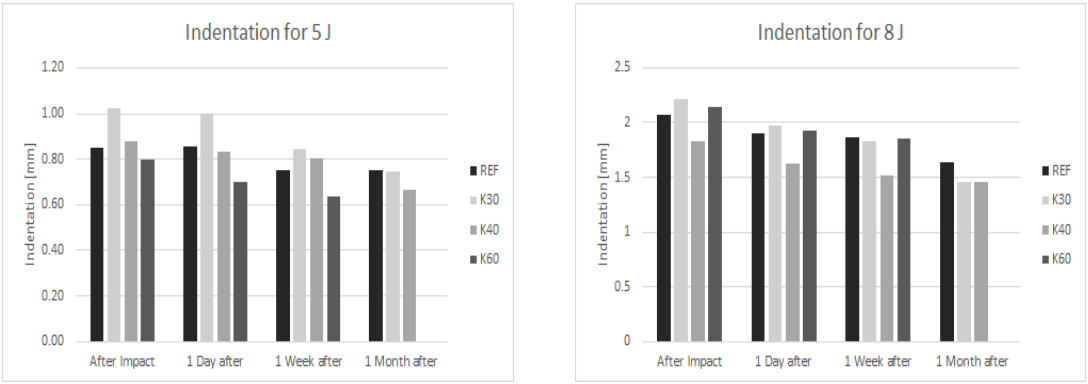


Figure 4.72: Permanent indentation for C1, C2 and REF

Comparison between K30, K40, K60 and REF

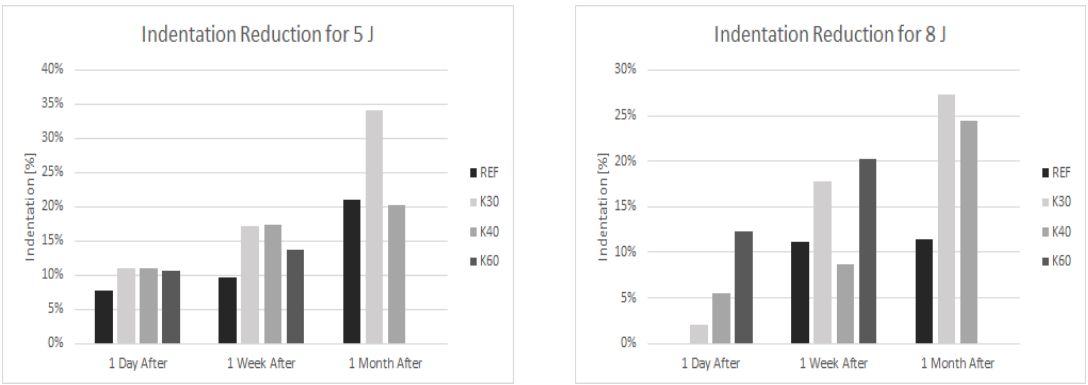
Analysing the figure 4.73, what stands out is the fact that K30 is the only one that have higher values of indentation comparing to REF in almost all of the cenarios. Another analysis is that K60 is the one with lower indentation for 5 J impact energy and K40 for 8 J. Due to the fact that the values do not follow any trend, it hard to take a conclusion out of the rest of the variables.

Globally, REF specimens show smaller values of recovery (Fig. 4.74), but it is also difficult to find a pattern to reach a final model due to the fact that the values vary.



(a) Indentation for 5 J impact energy (b) Indentation for 8 J impact energy

Figure 4.73: Indentation for K30, K40, K60 and REF over time



(a) Indentation reduction for 5 J impact energy (b) Indentation reduction for 8 J impact energy

Figure 4.74: Indentation reduction for K30, K40, K60 and REF over time

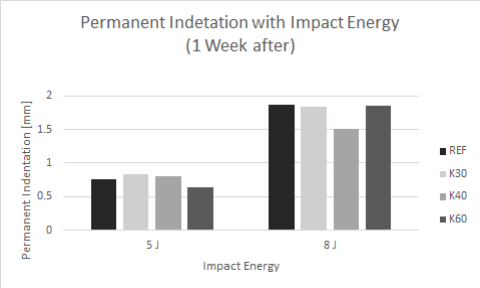
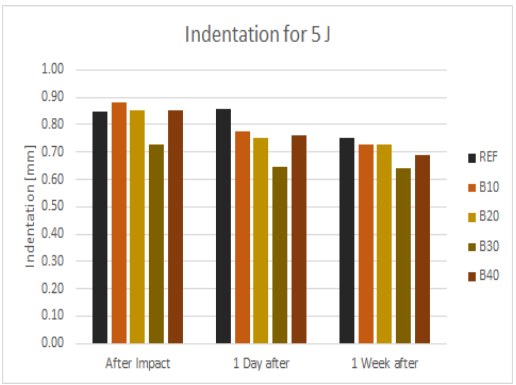


Figure 4.75: Permanent indentation for K30, K40, K60 and REF

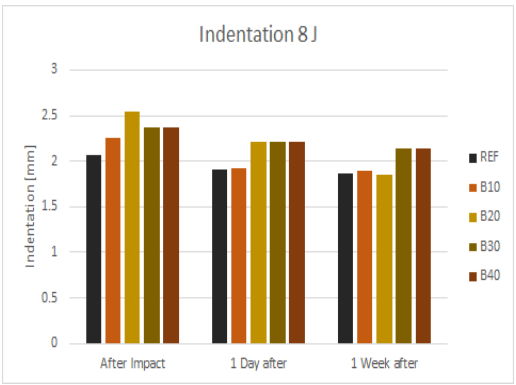
Comparison between B10, B20, B30, B40 and REF

The indentation values (Fig. 4.76)are quite similar for 5 J energy, but it's possible to see that K30 has lower values, which keeps going as the time goes by. For 8 J, REF average value is lower comparing with the expanded cork granules solutions, and higher value for B20.

On what restitution is concerned (Fig. 4.77), B10 has a high recovery value both for 5 and 8 J, but the difference starts to decrease with time for other laminates with expanded cork granules, specially for 8 J impact energy.

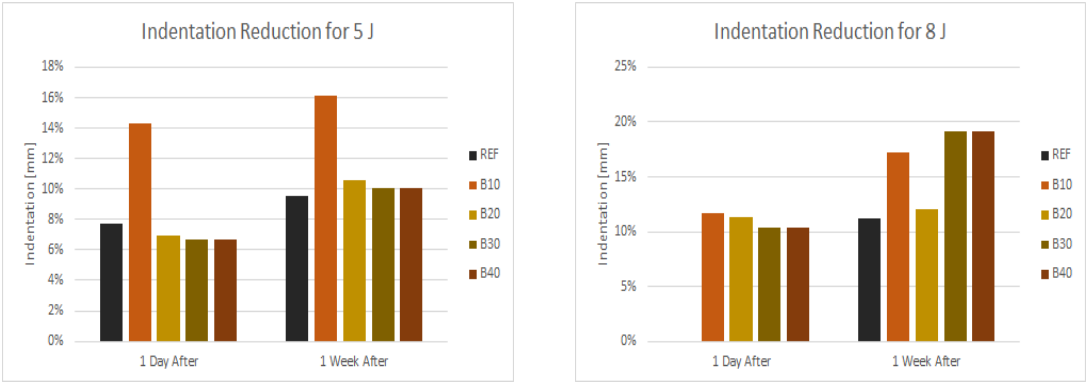


(a) Indentation for 5 J impact energy



(b) Indentation for 8 J impact energy

Figure 4.76: Indentation for B10, B20, B30, B40 and REF over time



(a) Indentation reduction for 5 J impact energy (b) Indentation reduction for 8 J impact energy

Figure 4.77: Indentation reduction for B10, B20, B30, B40 and REF over time

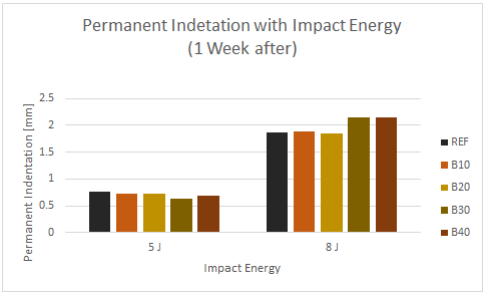


Figure 4.78: Permanent indentation for B10, B20, B30, B40 and REF over time

Comparison between all

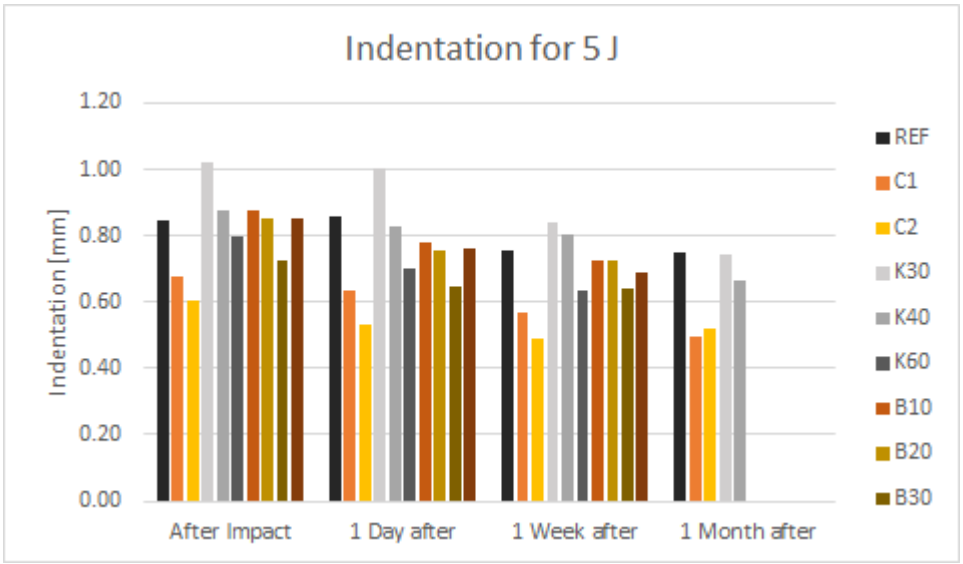


Figure 4.79: Indentation for 5 J impact energy

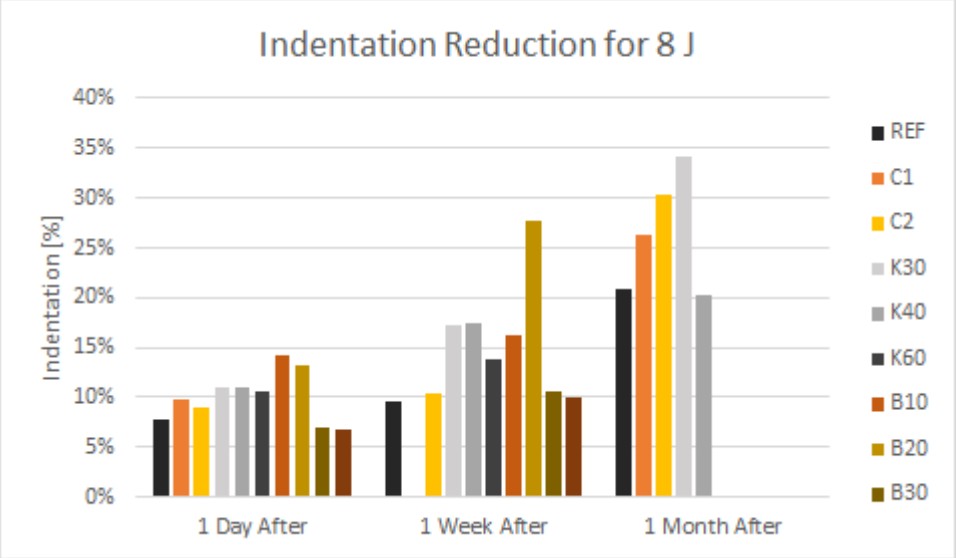


Figure 4.80: Indentation for 8 J impact energy

Analysing the whole group and looking first at the indentation values for 5 J impact energy (Fig. 4.79), the highest value goes for K30, followed by K40 and REF. On the other hand, C2 and C1 have the lowest indentation values. Outside of the "C group", B30 also shows low values, followed by K60. Looking at the whole picture, it is possible to verify that the pattern is kept over time, except some exceptions.

When looking at the results of 8 J impact energy level (Fig. 4.80), B20 has the highest indentation value, followed by B40. The "C group" have again the lowest values of indentation. Once again, the pattern is almost kept over time.

Although it is not on the graphs, by the tables 4.48 and 4.49, it is possible to see that there was one specimen that stood an impact energy of 13 J without being pierced.

Figure 4.83 shows the permanent indentation. The values shown are only with respect to one week after, to have a more fair comparison, although these values are most probably not the final permanent indentation values. C2 keeps having the lowest indentation values, followed by C1, both for 5 and 8 J of energy. K30 and K40 are the ones with higher indentation values for 5 J, but when looking at the 8 J group, the "B group" shows higher indentation values.

Regarding the recovery of some indentation values (Fig. 4.81 and Fig. 4.82), they are quite dispersed. Looking at one day after the impact, K30 and K40 have the lowest restitution values, but on the other hand, K60 has the highest percentage, not far away from B10, B20, B30 and B40. 1 week after, K60 leads the group with more than 20% of indentation recovery, where the "B group" also has interesting values, followed by K30 and C1. It is also possible to see that K40 continues to be one of the lowest. On the one month after group, not all the specimens are present as it was mentioned before, but K30, K40 and C1 show the highest values not far from each other, in opposition with C2, which is the lowest.

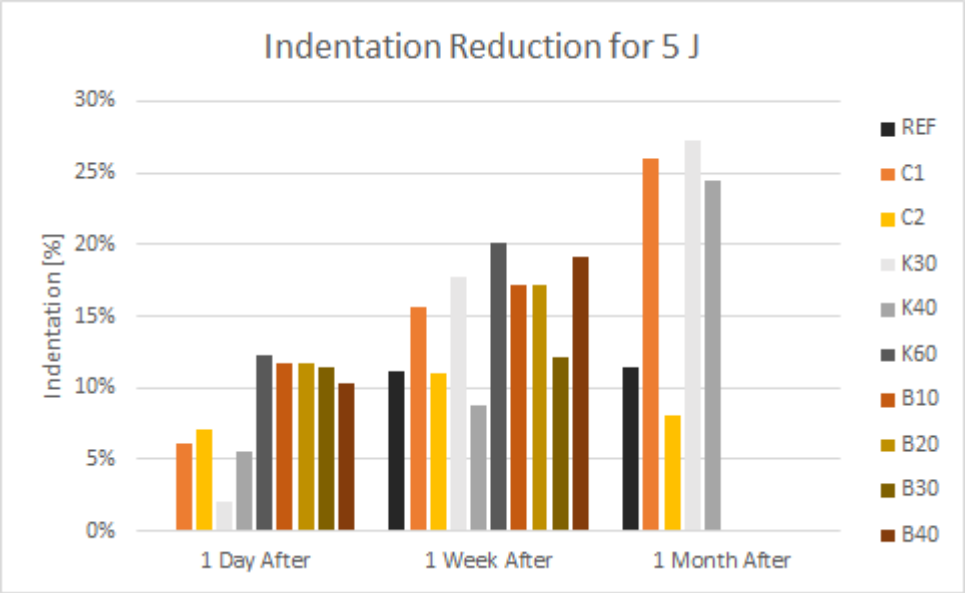


Figure 4.81: Indentation reduction for 5 J impact energy

From figures 4.81 and 4.82, it is also possible to see that almost all interlayer solutions show higher results, when compared to the reference.

Still about the restitution, but this time regarding 8 J impacted specimens, the values for one day after are quite similar with each other, with B10 and B20 showing the highest values and B30 and B40 having the lowests. As the time increases to one week, B20 jumps to the highest value, leaving B30 and B40 on the bottom.

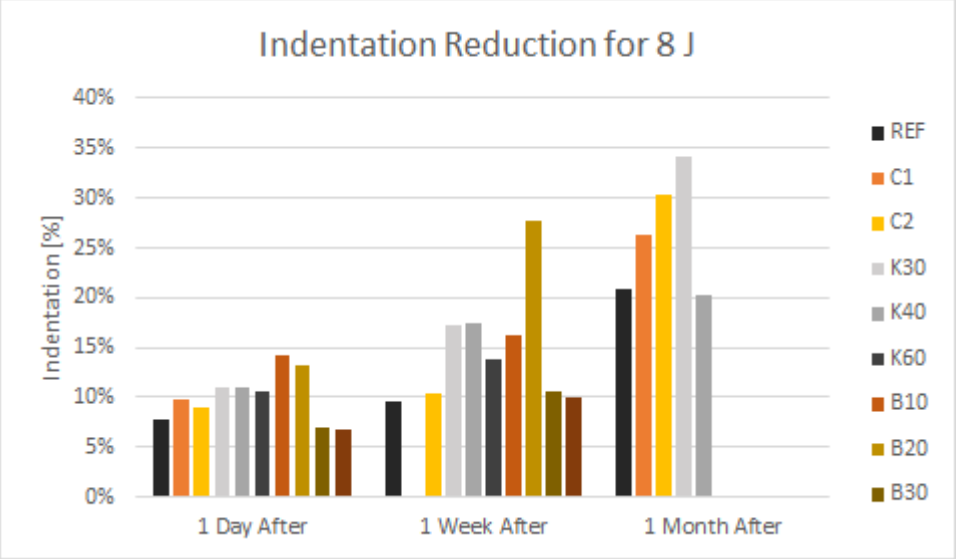


Figure 4.82: Indentation reduction for 8 J impact energy

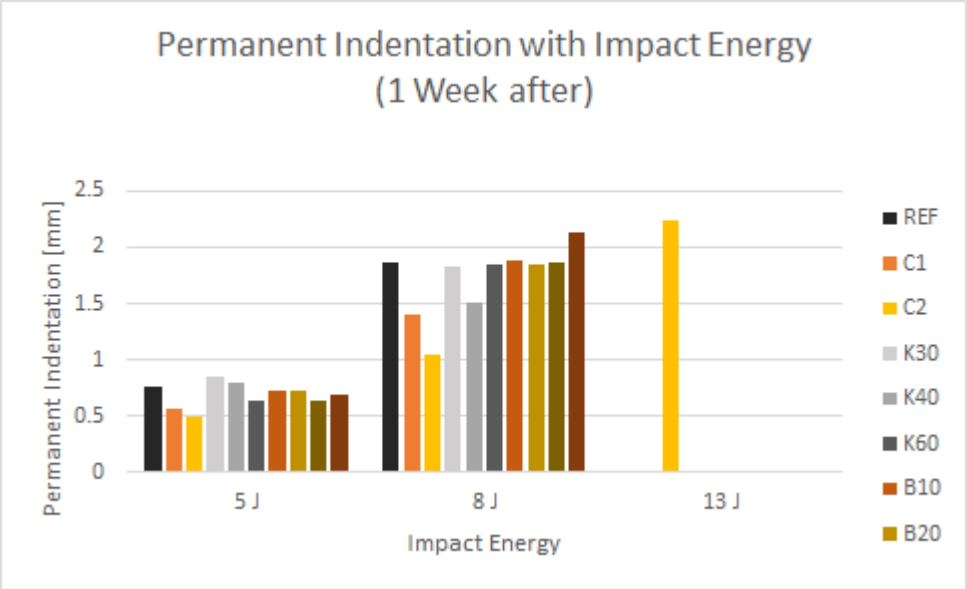


Figure 4.83: Permanent indentation

Chapter 5

Conclusion and Future Work

This dissertation consisted in the comparison of a carbon fibre prepreg with and without a interlayer material (reference) to assess the damage tolerance capabilities. As interlayer material, it was used cork and Kraton ™, in order to compare cork with another available solution. The usage of cork was also divided into two different formats: expanded cork granules and cork filmes. Two different thicknesses of films and four different concentrations of granules were used to evaluate how their difference behaves on what the topic of this thesis is concerned.

The experimental part consisted in subjecting the specimens to tensile tests, impact tests and tensile after impact tests (TAI). The indentation of the specimens subjected to impact tests was also measured after the impact and over time.

Tensile tests showed that in this situation, there was not a considerable difference in the direction of which the specimens were taken from the laminate, since the laminate was balanced. Tensile tests revealed also not considerable difference in the fabrication, since both reference laminates presented nearly the same mechanical properties. The tests for the solutions with cork films revealed a huge loss of mechanical properties, specially C2, since it made the laminate lose almost half of its properties. On the other hand, B10, laminate with the lowest concentration of expanded cork granules, revealed an improvement of the mechanical properties and K30, laminate with the lowest concentration of Kraton™ granules, revealed also a slight improvement of the Young's modulus. It is also possible to conclude that the higher the concentration of both Kraton™ and cork granules, the higher is the reduction of these properties. It is also important to mention that the granules of both materials just caused a small reduction of the properties, making these materials interesting candidates to be added as interlayer materials for this or other purposes.

Regarding the impact tests, the reference material showed one of the highest deflection and among the laminates studied and "stayed in the middle" on what peak force is concerned. Cork films, specially C2, managed to show the best values in all components of this study: peak force, deflection and energy recovery rate. An important remark to be made from this test is that although, cork films presented higher results, their thickness might have had a slight effect of impact resistance improvement. Besides cork films, Kraton™ granules also managed to have a good impact behaviour, sometimes even overcoming the reference material. Some examples were their

peak force (which was always higher), small deflection values (in the case of lower concentration), consistently high energy recovery rate (in case of the higher concentrations).

The tensile after impact test revealed itself quite inconsistent, due to the fact that the results presented a high variability, not being able to show an understandable behaviour, but as a whole, their performance was slightly better than the reference. This test aimed to essentially assess the residual properties of the laminates, after being subjected to low impact energies. High importance was taken to the reduction of the properties, by comparing the results from tensile tests of the impacted specimens with the results from tensile tests of not impacted specimens. Unfortunatly, it is not possible to take conclusions from this test due to the fact that the impact damaged provoked on the specimens sometimes happened along the specimen axis, other times it happened transversal to it, not being able to make a fair comparison amoung all. For these type of tests, it would be interesting to have more specimens and use lower impact energies.

On a more general way, it was not possible to completely understand, under the context of this study, if cork can be a good solution to enhance the damage tolerance in composite systems. By using the same prepreg and keeping the same stacking sequence, the only variable was the interlayer material. Cork films, such as the ones used, that had conditions to be used with both the prepreg and the resin chosen, until a certain thickness, showed good impact behaviour results, specially on the energy recovery rate, overcoming any other solutions studied, including the reference material, but the laminate ended up losing a considerable amount of its mechanical properties. Expanded cork granules showed quite some variating results on impact and tensile after impact tests. In some situations it managed to show better properties than the reference, but they were not exactly consistent, so no global conclusion can be taken. This might have happened due to the spreading technique that was used to apply the granules on the laminate, that not allowed the creation of a homogeneous layer, and thus, not homogeneous properties.

Future Work

After the whole experimental procedure, data analysis and results discussion, some hypotheses can be done about this topic and the work done so far. To improve the results, some suggestions can be made:

- Since one of the main limitations to take conclusions from TAI tests was the dispersity of the results between the laminates and in the specimens of the same laminate, due to mainly the damage occured, more specimens from each laminate would enable to differentiate the damage types and groups them accordingly. Since it was also concluded that the energies used for the TAI tests were too high, it would be important to use lower energies.

- Explore, under the same conditions, different cork film thicknesses and/or cork concentrations, in order to find the threshold until where the addition of this material enhances the damage tolerance, trying to find the “optimum” solution. It is also important to improve the

technique used to spread the granules along the laminates' surface to create a homogeneous layer.

- Perform similar studies (or with lower energy levels, if possible), but using compression and CAI (compression after impact) tests, since some of the materials used (cork, for instance) have different behaviours when their are subjected to tensile or compression loads. Besides, compression tests show a higher sensitivity to lower damages.

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