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Bioinspired approaches for toughening of fibre reinforced polymer composites

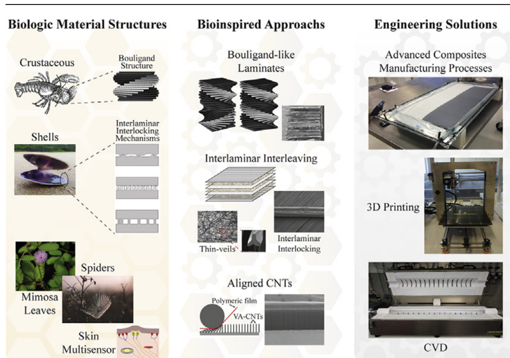
L. Amorim *, A. Santos *, J.P. Nunes, J.C. Viana

IPC - Institute for Polymers and Composites, University of Minho, Guimarães 4800-058, Portugal

HIGHLIGHTS

- Review of structures found in Nature and their main toughening mechanisms, envisaging potential mimicking approaches;
- Summary of engineering manufacturing solution capable to mimic biological material structures;
- Proposal of new bioinspired solutions combining both, Nature solutions and engineering developments to achieve high mechanical performance and multifunctional composite materials.

GRAPHICAL ABSTRACT



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ABSTRACT

In Nature, there are a large range of tough, strong, lightweight and multifunctional structures that can be an inspiration to better performing materials. This work presents a review of structures found in Nature, from biological ceramics and ceramics composites, biological polymers and polymers composites, biological cellular materials, biological elastomers to functional biological materials, and their main toughening mechanisms, envisaging potential mimicking approaches that can be applied in advanced continuous fibre reinforced polymer (FRP) composite structures. For this, the most common engineering composite manufacturing processes and current composite damage mitigation approaches are analysed. This aims at establishing the constraints of biomimetic approaches development as these bioinspired structures are to be manufactured by composite technologies. Combining both Nature approaches and engineering composites developments is a route for the design and manufacturing of high mechanical performance and multifunctional composite structures, therefore new bioinspired solutions are proposed.

Contents

* Corresponding authors.
E-mail addresses: luis.amorim@dep.uminho.pt (L. Amorim), b7525@dep.uminho.pt (A. Santos), jpn@dep.uminho.pt (J.P. Nunes), jcv@dep.uminho.pt (J.C. Viana).

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1. Introduction

Fibre reinforced polymer (FRP) composites are materials with superior properties and performance widely used in high demanding applications where lightweight is also required. Examples of application of FRP composites can be found in high-technology sectors, such as aeronautics, automotive, marine, energy, among others. For these reasons, a lot of research activity has been devoted to these materials. Laminated FRP composites parts are designed, manufactured and optimized for improved mechanical response [1,2]. Understanding their behaviour allows developing better products with more efficient processes and at reduced costs.

Recently, high performance FRP composites have also been looked as multifunctional, exploring its dissimilar materials nature, their hierarchical structure and anisotropic behaviour [3,4]. Besides improved stiffness and strength, superior impact toughness and fatigue resistance are also required. In addition to superior mechanical performance, also enhanced electrical or thermal properties are desirable [5]. The incorporation of nanoparticles into the resin [5,6] or at interfaces [7,8] has proved to be an efficient route to enhance the behaviour of FRP. The use of ultra-thin laminates have been shown to clearly improve the energy absorption capabilities of laminated composites [9,10]. Solutions for smart FRP composites, such as self-sensing [11], self-adaptive (morphing composites) [12] and self-healing [13] have been developed and investigated more recently.

Along the years, Nature has been able to develop high performance, multifunctional materials, which have been manipulated to adapt to the environment and to a given function [14–16]:

1. It is known that the high structural nacreous shells have superior impact toughness in spite of being made of brittle materials (aragonite) [17–19];
2. Seafood carapaces made of a combination of chitin and protein are light and impact resistant due to the structural arrangement of the constituents yielding high shock absorbance and resistance [20–23];
3. The metatarsal lyriform organ of the Central American wandering spider (Cupiennius salei) is a sensitive vibration sensor, able to sense a wide range of vibrations, typically from 0.1 Hz to several kHz [24];
4. All mammals have nerve ends organs that senses thought tactile, thermal, painful, pruritic (itch) and pleasant sensing and relay information to the central nervous system [25,26];
5. Mimosa is a plant that can close quickly their leaves under an external stimuli (e.g. when touched, dropped or jostled) [27];
6. A woodpecker beak is made of a laminated structure: a rhamphotheca wavy layer composed by β-keratin scales, a closed-cell foam inner layer made of mineralized bone, and bony layer made of collagen fibres embedded in a mineral matrix; being able of absorbing shock energy without damaging his body [28].

Understanding and exploring these Nature enhanced and multifunctional solutions have becoming an interesting technological-scientific challenge. Biomimicry is defined as the imitation of nature solutions for the purpose of solving complex human problems. The capability of mimicking nature approaches to develop high performance materials have been already explored [3,4,29,30]. The development of self-healing solutions for materials attempted to mimic natural healing through the adoption of several nature approaches [31,32] (e.g., matrix vesicles of bone, human vascular networks [33] and leaf venation systems [34]). However, in what concerns FRP composites, there is still the need for biomimetic approaches to develop novel solutions for improved and multifunctional behaviour.

Despite the efforts, above described, to mimicking natural structures and their multifunctionalities, their production and integration in advanced composites are still at an early stage. It is required first to understand the Nature approaches for materials toughening and multifunctionality by studying several natural examples. Then, bioinspired solutions are to be developed considering FRP composite structures and the constraints of their current manufacturing technologies.

Considering the actual literature, several review articles have been already published with regard to natural structures [14,29,30,35] and engineering approaches to improve mechanical performances [7,8,36–39] of advanced composite materials. However, a lack of conjugation between these subjects still have not been meet. Therefore, it is not intent of this article to give an individual overview of those topics or develop new manufacturing processes, but rather filling this gap by adapting bioinspired solutions to current composite technologies.

In this article, are initially analysed several approaches that have proved to be the relevant contributes to the improvement of mechanical and functional properties of natural products considering a wide range of Nature examples. Then, main composite technologies are revised with the aim of establish an engineering approach that allows the mechanical and functional improvements of composites structures. Only continuous laminated FRP composites are considered due to their application in structural components, requiring enhanced mechanical and multifunctional behaviour. Finally, crossing both Nature solutions and composite technology development, bio-inspired engineering approaches are proposed for mechanical performance improvement and self-sensing of high demanding composite structures.

2. Nature approaches to mechanical and functional improvements

For millions of years, natural materials have been optimizing their structure and composition to achieve outstanding mechanical and multifunctional properties. As first described in the 20th century [3,4], these materials have the ability to assembly weak brittle components at the nanoscale into highly organized macroscale structures with high strength and toughness. Understanding their structural hierarchy and toughening mechanisms throughout multi-length scales from nano- to the macroscale is the key to build high performance materials. In this section are reviewed the nature approaches of some materials, such as shells, arthropod exoskeletons, sponge spicules, birds' beaks, molluscs' byssus threads, bone, wood and bamboo, that lead to their superior mechanical performance and multifunctionality [14,15,45,16,29,30,40–44]. Based on Wegst and Ashby classification [46,47], the materials were assorted into: Biological ceramics and ceramic composites, Biological polymers and polymer composites, Biological elastomers and Cellular materials.

A schematic description of this chapter may be seen in Fig. 1.

2.1. Biological ceramics and ceramic composites

Biological ceramics and ceramic composites are composed of a high percentage of a brittle material and a minor content of a softer component. Several organisms, namely nacreous shells, conch shell, brachiopod shell, deep-sea mollusc shell, ammonite shell, shrimp club, had developed a biological ceramic body armour with a high tough structure [14,29,41]. Others, as sponge spicules, had overcome the fragility of its components resulting in a highly flexible skeleton [14,48,49]. In this section is presented a review of their structure, mechanical properties and respective synthetic biomimetic approaches.

2.1.1. Nacreous shells (abalone shell)

In the last decades, abalone shell has been the most studied organic-inorganic nanocomposite material. By a bio-mineralization process, aragonite ($CaCO_3$) platelets grow throughout a biopolymer matrix resulting in the brick-mortar like structure [50–59], as shown in Fig. 2. These aragonite bricks, which are approximately 95% percent of the composite, yield stiffness and hardness to the structure [60–66], while the interaction mechanisms between them and the viscoelastic mortar-like matrix are crucial to the toughness behaviour of the material. As others have highlighted [27,31,49,50,56–61,62–64], several toughening mechanisms enable a higher energy dissipation, namely by pull-out with interfacial hardening of the platelets and interlocking mechanisms between their surface. In a greater detail, under stress it can be observed local transverse elastic deformation of the aragonite platelets core region, followed by longitudinal sliding of their surface, which yields interfacial hardening, as schematized in Fig. 3. The ensuing interlock of adjacent bricks occurs on the overlap regions, absorbing energy by inelastic deformation.

Furthermore, recent studies [17,30,41,61,71,76] underlined the role of platelets surface topology (e.g., design and roughness) in these toughening processes, as well as in the arrest of possible resulting cracks. Three types of interfacial interactions were considered: a) asperities; b) organic interface; and c) mineral bridges; as sketched in Fig. 4a-c. Asperities interlocking (Fig. 4a) leads to morphological anisotropy and high energy dissipation (inelastic deformation) yielding high resistance to crack propagation. In addition, the organic matrix itself can act as a viscoelastic reinforcement between the rigid bricks (Fig. 4b), leading to stress redistribution and crack path deflection by extrinsic mechanisms, such as crack bridging or microcracking [30,41,43,71,76]. Other possible surface interaction is interlocking by mineral bridges (fully-grown throughout the organic matrix, Fig. 4c), which break during pull-out and act as asperities [30,41,43,71,76]. However, since these interlock mechanisms cannot induce the platelets transverse expansion level observed in nacreous shells, some authors have described a cooperative toughening mechanism [18,71]. The bricks surface waviness (Fig. 4d) leads to progressive interlocking, yielding slide resistance and ensuring high interfacial hardening, while the

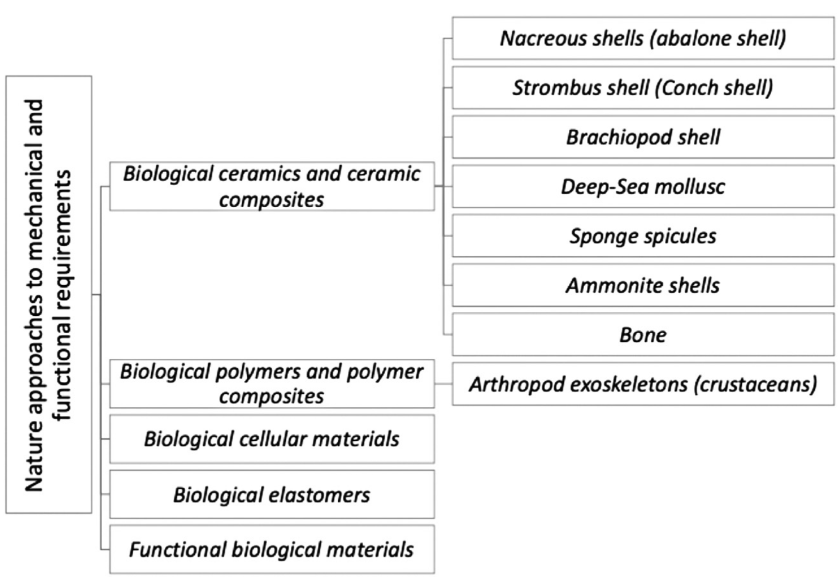


Fig. 1. Structural scheme of Chapter 2 - Nature approaches to mechanical and functional improvements

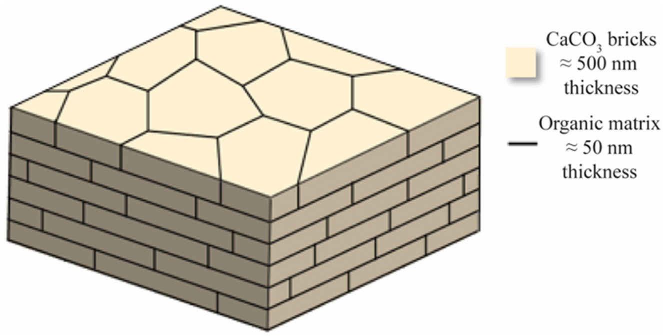


Fig. 2. Sketch of the brick-mortar nanostructure of nacre.

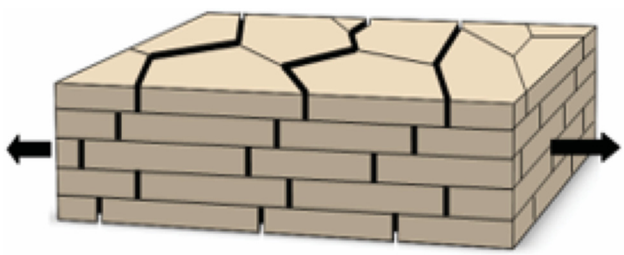


Fig. 3. Schematics of the platelets' pull-out during tension.

mineral bridges and organic matrix fasten adjacent aragonite bricks. It should be noted that this interfacial waviness effect can be replaced with a dovetail structure [17,68], as exemplified in Fig. 4d,e, which promotes high interfacial hardening and crack path deflection. Further studies are being conducted to fully understand the mechanisms that yield hardness, toughness and crack propagation resistance of this complex nacreous material.

In order to mimic the natural structure of nacreous shell and its high mechanical performance, several manufacturing techniques had been proposed (Fig. 5a-c): Layer by layer deposition (LBL); Ice-templation; 3D-printing, among others.

LBL method is the most widely used to fabricate nacre-like composites. *Tang et al.* [77], poly(diallydimethylammonium) chloride (PDDA) and montmorillonite clay platelets (MTM) were assembled in a highly structured $(PDDA/MTM)_n$ composite with platelets waviness, resulting in a tensile strength and Young's modulus similar to values presented by nacre [65] and lamellar bones [78]. *Podsiadlo et al.* [79], produced poly(vinyl alcohol)(PVA)/MTM and PVA/MTM-glutaraldehyde(GA) composites. The tensile strength and Young's modulus of PVA/MTM multilayered composites are approximately 275% and 665% over the values obtained for pure PVA, respectively. The PVA/MTM-GA displayed a tensile strength and Young's modulus approximately 167% and 715% over the uncross-linked PVA/MTM multilayer values, respectively, outperforming nacre strength [64,65]. *Bonderer et al.* [80] produced submicrometric Al_20_3/chitosan composites that showed an inelastic deformation of 17%, opposing to the catastrophic failure observed in others clay composites [77]. Regardless of having a low hard component percentage, the composite displayed a tensile strength 85% over that of nacre and a Young's modulus comparable to those of dentin [81,82] and bone [78] and almost 630% lower than that of nacre [65]. More recently, *Gao et. al* [83] produced brushite ($CaHPO_4 \cdot 2H_2O$) platelets – biocompatible sodium alginate (SA) nacre-mimetic films were produced by bottom-up assembly and hot pressing, which presented an impact strength and ultimate flexural strength, 407% and 55% higher, respectively, than the values for natural *C. plicata* nacre. Despite presenting an ultimate stiffness much lower and fracture toughness slightly lower than natural *C. plicata* nacre, the maximum fracture toughness was 48% higher.

In the last decade, ice templation method has been used to reproduce the inter-surface roughness observed in nacre. *Deville et al.* [84] produced alumina/Al-Si and alumina/Al-Si/Ti hybrid materials in which the addition of Ti interfacial bonder increased 50% and 82% the tensile strength and fracture toughness, respectively, compared to the hybrid standard material, presenting a fracture toughness of the same order of magnitude that of nacre and a much higher tensile strength. More recently, *Bouville et al.* [85] synthetized nacre-like alumina-glass phase based on ice template method, which showed an increase of tensile strength, Young's modulus and fracture toughness by 177%, 314% and 175%, respectively, compared to those of nacre.

3D printing is an emerging manufacturing technique for obtaining nacre-like composites at the microscale. *Espinosa et al.* [17] studied the

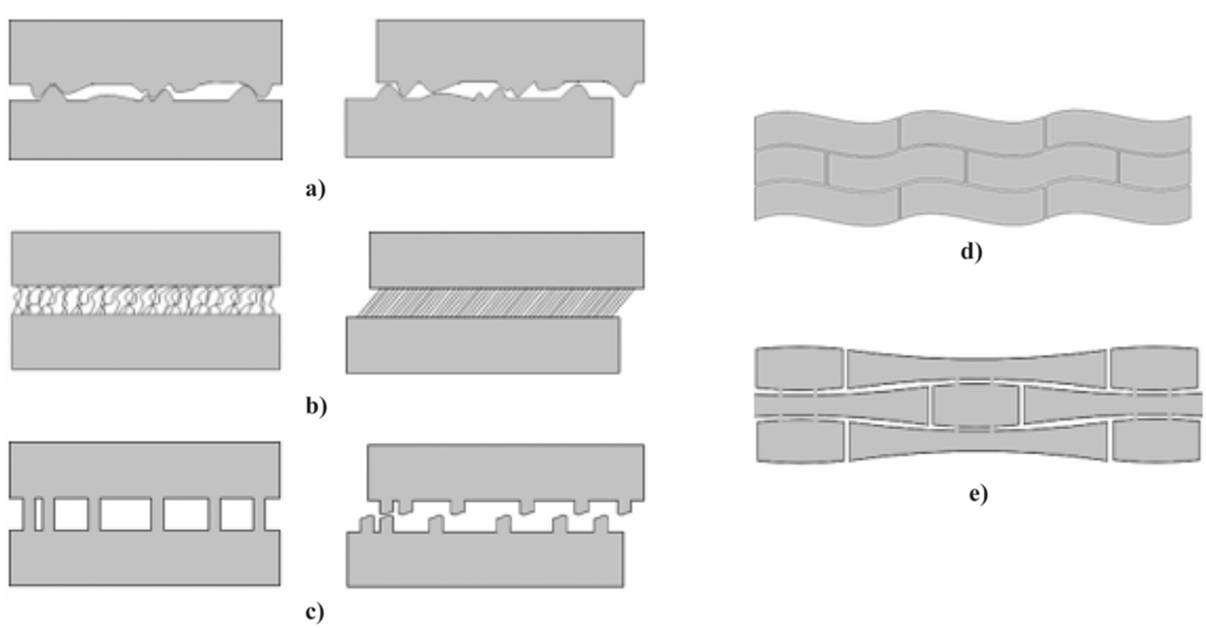


Fig. 4. Several interlaminar interlocking mechanisms. a) asperities roughness. b) viscoelastic organic 'glue'. c) Interlaminar mineral bridges. d) Platelets' waviness. e) dovetail-like platelets.

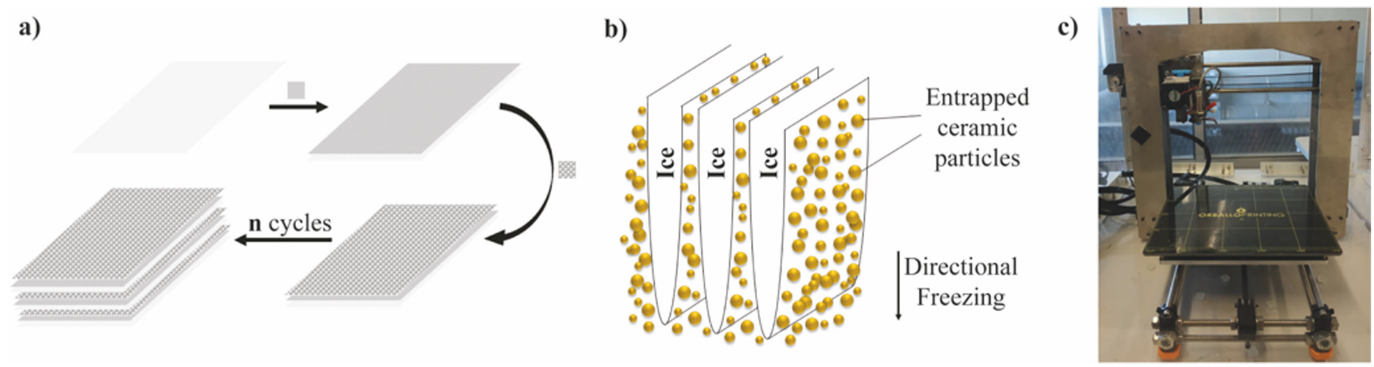


Fig. 5. Scheme of manufacturing techniques: a) LBL, b) Ice-templation and c) 3D-printing.

platelet geometry influence on the toughening mechanisms of ABS (acrylonitrile butadiene styrene)-epoxy dovetail tablet composite through energy dissipation measurement. The major energy dissipation was achieved with a dovetail angle of 1° and a relation of 3.1 between the dovetail length and platelets thickness, which surpass more than 130% the value obtained for the 0° dovetail angle sample. This platelet geometry is within the range of the one found in natural nacre, mimicking its hardening/fracture failure mechanism. *Rim et al.* [86] manufactured an ABS/Chitosan and Alumina/Chitosan composites by fused deposition modelling (FDM) to optimize platelet geometry for major energy dissipation during tension, reaching similar conclusions as previous studies [17]. More recently, *Zhang et al.* [87] printed a rigid photopolymer VeroWhitePlus (VW) and rubber-like material (mixture of VW/TangoBlackPlus photopolymer) composite that displayed a high specific loss modulus and damping characteristics. Additionally, they demonstrated that optimal interfacial strength of these 3D structures, which correspond to the highest impact resistance, decreases with the increase of impact velocity [88].

Other methods have also been used with the purpose of mimicking nacre structure. *Clegg et al.* [89] conducted the very first attempt of producing SiC (silicon carbide powder)–boron/graphite composite by simple pressing. The material presented a non-catastrophic crack growth, as well as a fourfold and hundredfold increase of the fracture toughness and work of facture, respectively, compared to the monolithic SiC. *Wang et al.* [90] combined rolling compaction, hot-pressing and slurry coating to produce diverse Si_3N_4/BN composites focusing on interlayer bonding improvement, reporting values over 50% increase on the bending strength and fracture toughness compared to the standard laminate, and matrix reinforcement, whose addition in the composite yield values of fracture toughness reaching a 86% increase. *Ekiz et al.* [91] developed alumina/epoxy laminar composites by Hot-press Assisted Slip Casting (HASC), which displayed values of flexural modulus and tensile strength 283% and 63% superiors to the ones of the simple mixing process, respectively. These values and the work of fracture value were inferior to the ones presented by nacre, despite of demonstrating high flake's alignment. *Walther et al.* [92] produced PVA/MTM composites via adsorption and separation, followed by layer orientation via paper-making, doctor-blading or simple painting, whose strength surpassed those of nacre.In recent years, *Barthelat et al.* [93] welded millimetre-size wavy poly-methylmethacrylate (PMMA) tablets with fasteners, which showed strain-hardening behaviour present in nacre despite of displaying very low strength. *Li et al.* [94] produced nacre-like R-PVA/GO composite films by solution casting method. These films showed an increase of 60% and 93.5% in tensile strength and failure strain, respectively, compared to the values obtained for PVA/GO composite. Also, the highest tensile strength and failure strain surpassed those of nacre. More recently, *Cheng et al.* [95] synthesized FDWCNT (flattened double-walled carbon nanotubes)-epoxy composites by a combination of stretching, functionalization, hot-pressing with or without crosslinking. The cross-linked aligned composites displayed an increase of tensile strength, Young's modulus and toughness by 256%, 234% and 155%, respectively, compared to those of the random unlinked composites, which clearly surpass those of nacre.

Recently, authors [96] developed thick nacre-mimetic GO-based bulks and concluded the flexural strength of the composites increased, the energy absorption increased and the failure mode was progressive.

2.1.2. Strombus shell (Conch shell)

The Strombus shell (Conch shell) possess an optimized structure for impact resistance and crack arrest conferred by three aragonite macro-layers: outer, middle and inner layer, assembled in a crossed-plywood structure [14,97–103]. Each layer is divided in crossed sub-lamellae, first-order, second-order and third-order lamella (Fig. 6), which have lagged orientation between them, conferring anisotropic mechanical behaviour to the tessellated structure [85,87,89,104]. Also, after crossing the material strength limit, it allows crack branching through the lamellas tortuous path. These lath-like crystal lamellas are connected to each other by an organic binder whose role is crucial in the toughening mechanisms, namely, through fibre pull out, microcracking in interlamellar boundaries, crack bridging, and microstructurally induced crack arrest [98–101,105–108]. It should be noted that despite of this layer having a lower organic fraction compared to nacre, it displays a higher fracture toughness [100,103,108,109]. Despite of being carried out few attempts to mimic this structure, it was stated that the maximum fracture toughness was obtained for a 60° rotation angle between the fibres

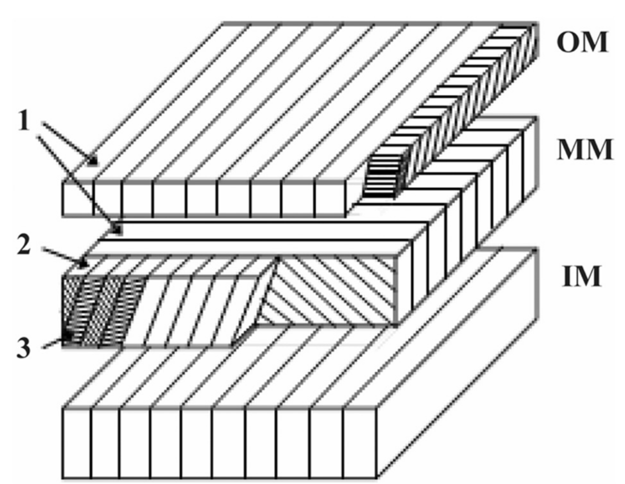


Fig. 6. A schematic of Strombus shell's structure: OM – Outer macrolayer; MM – Middle macrolayer; IM – Inner macrolayer; 1 – First-order lamella; 2 – Second-order lamella; 3 – Third-order lamela.

of the layers [98,102]. Recently, authors [96] developed a proof-of-concept single edge notched panel that mimics the composite structure of conch by 3D printing, showing improvements in strength and toughness compared to the bulk panel.

2.1.3. Brachiopod shell

The Brachiopod shell displays a tri-layer calcite structure: columnar inner layer, fibrous middle layer and hard outer layer. This outer layer is characterized by a jigsaw pattern of meso-crystals, which allows crack deflection, bridging and stress distribution, yielding high hardness and toughness to the interdigitated structure [29,110–116].

2.1.4. Deep-Sea mollusc

The deep-see mollusc (gastropod) shell is a tri-layered composite: mineralized outer layer (OL), organic middle layer (ML) and calcified inner layer (IL) (Fig. 7), with superior wear resistance and flexural stiffness. Under stress, the major energy dissipation happens through inelastic deformation of the ML, preventing cracks in the IL. Moreover, the interface waviness between OL and ML is crucial to stress distribution through local multiple delamination events, while the hard IL reduces radial displacement and yields flexural resistance [117,118].

2.1.5. Sponge spicules

The sponge spicules (Fig. 8) are well-known for its outstanding flexural toughness. These cylindrical amorphous silica rods are characterized by a onion-like structure, which in conjunction with the interlayer organic component induce high toughness and crack arrest capabilities [14,48,49,119–121].

2.1.6. Ammonite shells

The structure of the ammonite fossil shell is segmented into chambers (Fig. 9a) divided by internal mineralized steptas, which connect to the outer layer through suture joints (Fig. 9b). This multilevel hierarchy is crucial to yield high strength and stiffness to the aragonitic shell [29,122–126]. Moreover, some studies [122,123] evidenced the enhancement of crack growth resistance and flaw tolerance with higher hierarchical levels of the suture joints.

2.1.7. Bone

The bone is a composite of collagen fibrils reinforced with hydroxyapatite nanoparticles, known by its high plasticity and toughness. At the microscale level the structure is characterized by a cortical compact bone, whose microstructure displays oriented mineralized collagen fibres assembled in a lamellar structure forming a plywood-type stacking, and a cancellous porous bone consisting of trabecular interconnecting framework [16,127–130]; whereas at the nanoscale level, the mineralized fibres form a brick-mortar-like structure [78,131]. This structure allows several toughening mechanisms, namely, viscoplastic flow, microcracking, crack bridging and crack deflection [127,128,132–135],

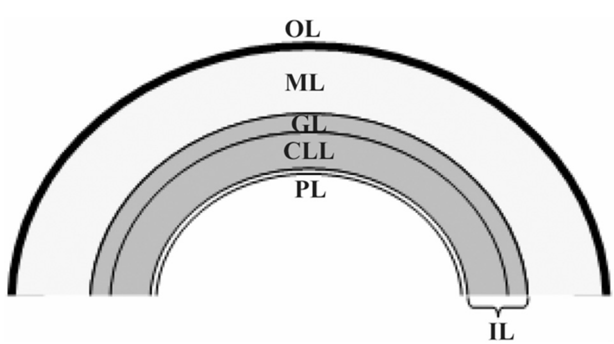


Fig. 7. Schematic of Deep-sea mollusk structure: OL – Outer layer; ML - Middle layer; IL - Inner layer; GL - Granular layer; CLL - Crossed lamellar layer; PL - Prismatic thin layer.

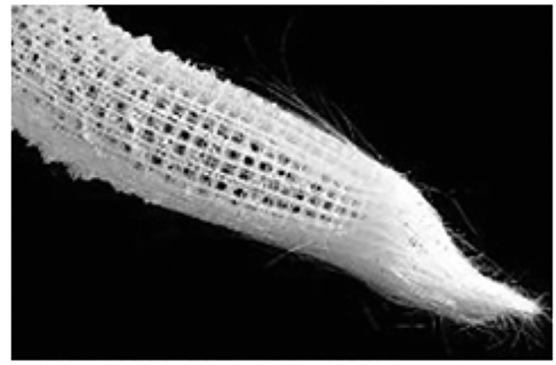


Fig. 8. Sponge spicule.

yielding high toughness and torsion resistance. In the last two decades, several authors tried to mimic this lightweight, strong and flexible structure [30,71,131,136–141].

2.2. Biological polymers and polymer composites

Biological polymers and polymer composites are mainly composed by soft tissues (e. g. fibres). In this work they are only represented by the arthropod exoskeletons of crustaceans, e. g. lobster and crab.

2.2.1. Arthropod exoskeletons (crustaceans)

The arthropod exoskeletons of crustaceans, e. g. lobster and crab, are characterized by a strong multilayer cuticle (exocuticle–outer and endocuticle–inner layers) of mineralized α-chitin fibres/protein planes in a 180° rotation stacking twisted plywood (Bouligand structure) (Fig. 10 and Fig. 11) [20,21,142–146], which in case of the lobster form a honeycomb-like chitin-protein plane (Fig. 10) [144].

Some authors described the crucial role of the plywood density and of the degree of mineralization of these layers in the anisotropic mechanical properties. Specifically, the exocuticle layer showed higher hardness and stiffness due to higher mineral density and lower distance between the planes in the Bouligand layer compared to the endocuticle layer that shows more resistance to compression applied in the normal direction of the surface than tension in the transverse direction [147–151].

Moreover, it has been reported some energy dissipation mechanisms in a Bouligand stacking structure, such as: fibrillar stretching, interfibrillar and interplanar rotation, delamination along heterophase interfaces and microcracking [20,21,154,155,22,143,145,147–149,152,153]. In addition, the porous network of ductile tubules present in the exoskeleton act as a glue between the fibres, yielding toughness to the structure [20]. Due to these suitable features several authors mimicked the Bouligand plywood for impact resistance applications. *Grunenfelder et al.* [23] stacked carbon fibre/epoxy prepregs standard (0°/±45°/90°) and bioinspired with rotation angles of 7.8°, 16.3° and 25.7°. This bioinspired with large rotation angle composites showed a reduction of damage through thickness after impact, due to a higher in-plane damage spread, and a residual strength 18% superior to the standard composite. Moreover, *Ginzburg et al.* [156] showed similar results using helical carbon fibre/epoxy prepregs, reaching 26.6% enhancement of energy absorbed during impact and lower damage area compared to quasi-isotropic layouts (standard). *Apichattrabrut et al.* [157] assembled a 40-layer carbon/epoxy prepregs helical composite (10° rotation), which showed higher energy absorption and penetration resistance and lower damage area. *Cheng et al.* [152] produced glass fibre/epoxy prepreg standard and helical composites that reached values 54.17% and 83% over the flexural stiffness and residual strength of the standard composite. *Yang et al.* [158] fabricated Bouligand-type MWCNT/resin composites with different rotation angles, using electrically assisted 3D-printing technology. The composites with

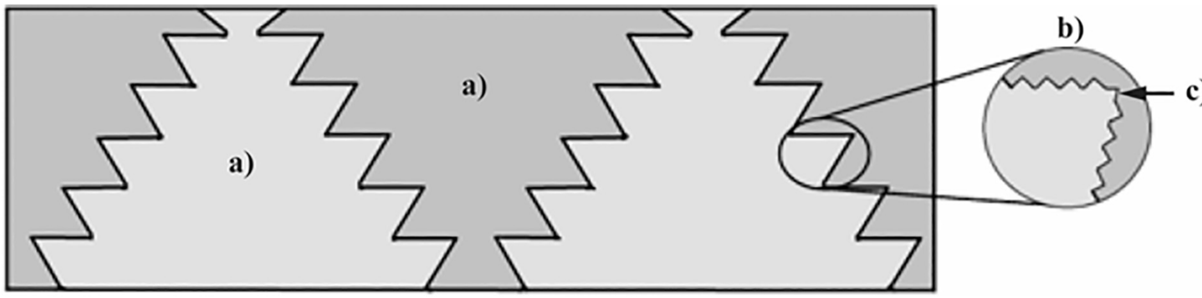


Fig. 9. Rigid saw tooth pattern of hierarchical multilevel suture joints (b) and thin soft interface layer (c).

the smallest rotation angle displayed the highest energy dissipation and impact resistance. Recently, *Mencattelli* and *Pinho* [159] used thin prepregs to mimic and manufacture Bouligand structures with different mismatch angles (2.5°, 5°, 10°, 20° and 45°). Their first study has shown that under low velocity impact the reduction of pitch angles leads to a smooth though-the-thickness helical damage, which reduces damaged area, higher load-bearing and damage tolerance. In other study conducted by the same authors, they have demonstrated that, under quasi-static indentation tests, Bouligand-like structures (especially those with 2.5° pitch angle) may delay catastrophic fibre failure, increase load-bearing and energy dissipation [160].

Another example of a highly impact resistance structure is the plywood observed in the stomatopod dactyl club (Fig. 12) [161]. The particularity of this structure is its division in a typical bouligand inner region and a denser herringbone outer region, in which, each plywood layer presents a characteristic waveform pattern, This out of plane configuration leads to an interlock system, which delays delamination by constant crack re-orientation, yielding higher energy absorption [162]. Recently, *Mencattelli* and *Pinho* [163] produced a high-performance Herringbone-Bouligand microstructure with Carbon Fibre Reinforced Plastic (CFRP) by micro-moulding. This composite showed reduced (71%) delamination damage and higher damage-tolerance compared with the classical Bouligand structure.

2.3. Biological cellular materials

Biological cellular materials are characterized by a compact outside surface, which support higher stresses, and a lightweight cellular interior in order to reduce the material density without compromising the strength and impact resistance. An example of these mechanical performance is the wood structure, which is divided into honeycomb-like macro-fibrils by an amorphous matrix and subdivided in multi-order cell walls with helical interlaminar transitions [15,164,165]. Another light-weight natural material is the toucan and hornbill beaks whose structure consist of an external hard keratin shell with a fibrous closed-cell interior system and a hollow core. Not only this foam-like interior allows energy dissipation under stress and ensuing mechanical stability, but also the shell and foam-like sandwich composite yield high stiffness to the beaks [15,28,166,167].

2.4. Biological elastomers

Bivalve molluscs' byssus threads are an example of highly strong, tough and self-healed biological elastomer, whose fibrous core is coated by a hard proteinaceous cuticle. This coating formed by submicron-sized dihydroxyphenylalanine (DOPA)– $Fe3+$ doped granules and a soft matrix (Fig. 13) is responsible for abrasion resistance under compression and high extensibility of the structure, respectively, preventing catastrophic failure of the thread by extensive microcracking of the matrix [168,169]. Based on granules composition, *Podsiadlo et al.* [170] assembled DOPA-Lys-PEG/Montmorillonite clay + $Fe3+$ films by LBL, which showed an improvement of strength and toughness by over 400% and 500%, respectively, compared to Lys-PEG/Montmorillonite clay films.

2.5. Functional biological materials

Engineering found in Nature a wide source of inspiration for advanced multifunctional artificial materials: from the example of the butterfly wing, which possess self-cleaning, structural colour, superhydrophobicity, adhesion and chemical sensing properties [42]; to spider vibratory sensory system; to sensitive [27,171] and carnivorous [172] plant pressure sensitive system; to self-sensing properties of human skin and other living beings. The mechanosensory system of living organisms roughly consists in a network of mechanoreceptors activated by pressure changes, triggering a series of responses. For example, in spiders the detection of local exterior vibration is made through a mechanical amplification mechanism consisted of deformable slits, which causes sensitive membrane deformation, triggering a reaction by the organism [24,173]. Differently, in the human skin tissue, skin

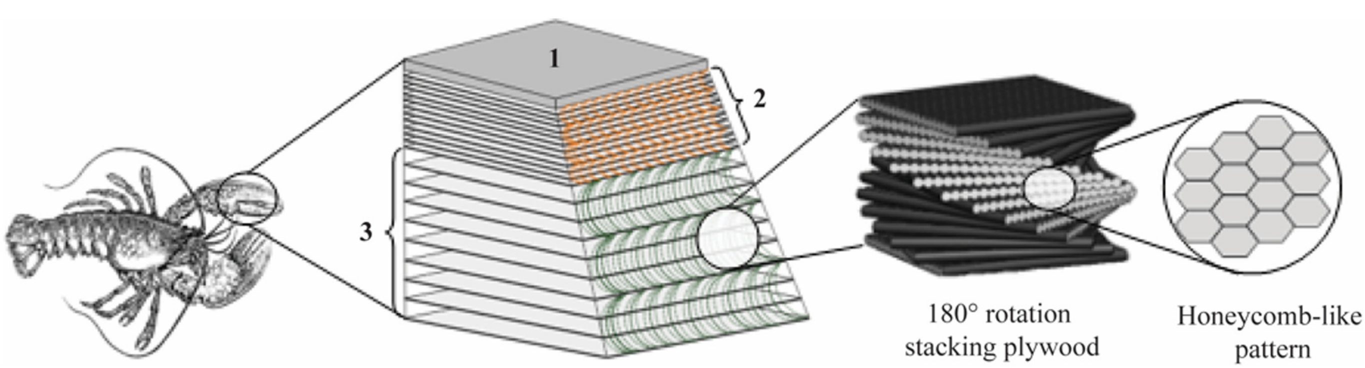


Fig. 10. Hierarchical structure of multilayered lobster's exoskeleton: 1–Epicuticle (surface); 2–Exocuticle; 3–Endocuticle.

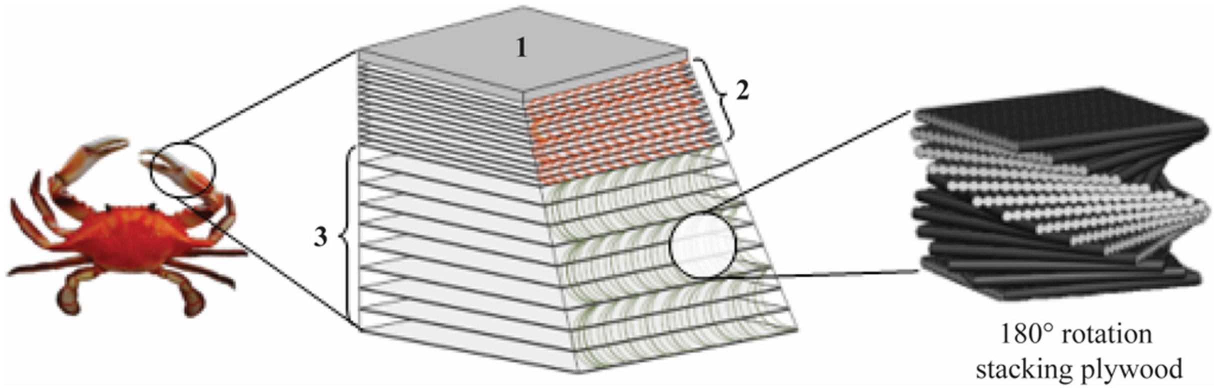


Fig. 11. Hierarchical structure of crab's exoskeleton: 1–Epicuticle (surface); 2–Exocuticle; 3–Endocuticle.

displacement activates a chain of ions pump-dependent reactions transmitting position and intensity of the pressure contact point from the superficial cellular mechanoreceptors network through nerves until reaching the brain [25,174–178]. An important feature of this highly sensitive system is the ability to detect fine spatial separation due to its small receptive field. Therefore, the most sensitive skin parts are densely clustered (Fig. 14) with sensors to thoroughly recognize the stimulus [26,179,180]. Besides, densely populated with pressure sensors, skin possesses multi-sensing capabilities (e.g., pressure, temperature, humidity, wet/dry, roughness).

In order to achieve high sensibility range, some authors [181–184] produced sensors mimicking the crack-shaped structure of the spider slit organs, while others [185,186] tried to reproduced human tactile sensing based on piezo concept: piezoelectric [187–191] and piezoresistive [192–197] sensors that generate electrical charges or change their electrical resistance, respectively, when subjected to deformation. More recently, some authors produced biomimetic skins with multifunctionalities [198–201]. These sensors have an anisotropic distribution being densely clustered in the highly sensitive skin areas. Moreover, due to its small receptive field it is possible to detect fine spatial separation.

Surprisingly, some authors even used the wood structure to produced high sensitive pressure sensors [202]. In addition, others have started to explore ionic conduction for human-like wearable devices [203].

In this review, several biological materials with high mechanical performances and also some with multifuncional properties were presented. Despite the different high impact resistance structures presented, they have in common the energy dissipation mechanisms, e.g., asperities interlocking or crack deflection along the soft adhesive bonds or along angle-oriented layers, that are responsible for the high mechanical performance of biological structures. Several efforts were made to mimic these structures using different techniques, however many of them were done in an unsuitable scale or with mechanical properties that did not reach the desirable performance. Regarding mimicking multifunctional materials, there are several interesting studies, however they present production, efficiency or applicability limitations. In an attempt to respond to the shortcomes in the production of advanced bioinspired composites with optimised mechanical performance and multifuntionality, several bioinspired solutions are presented latter, considering an engineering approach to mechanical and functional improvement described in the following. section.

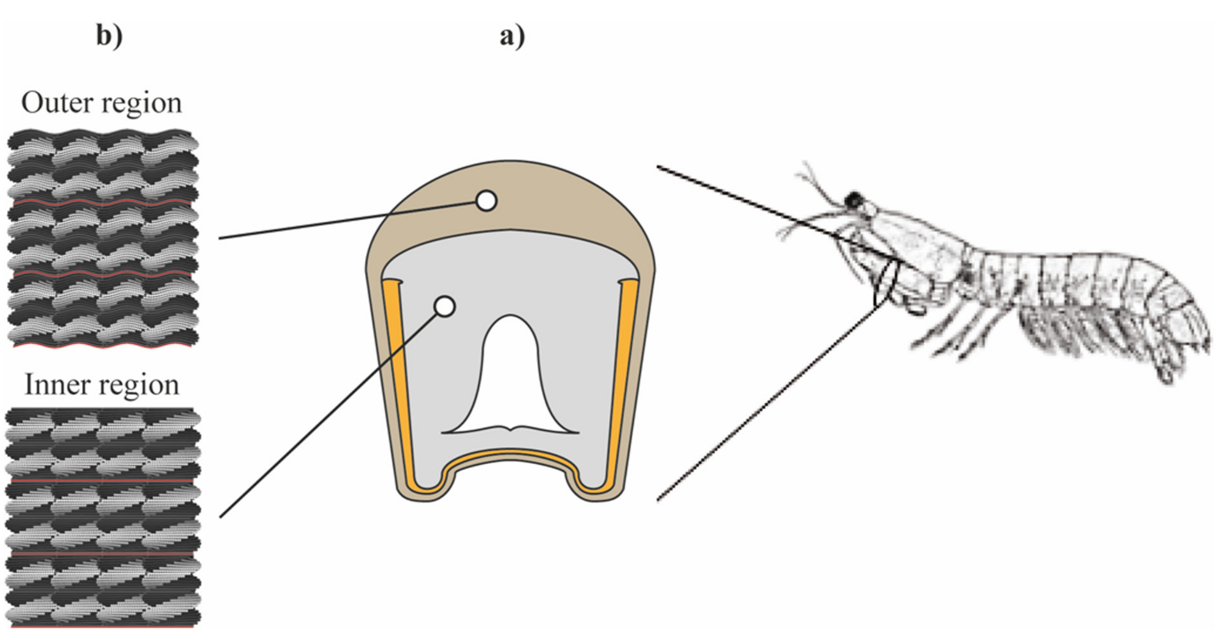


Fig. 12. Structure of the cross section of stomatopod dactyl club (a) which is divided in inner and outer region (b).

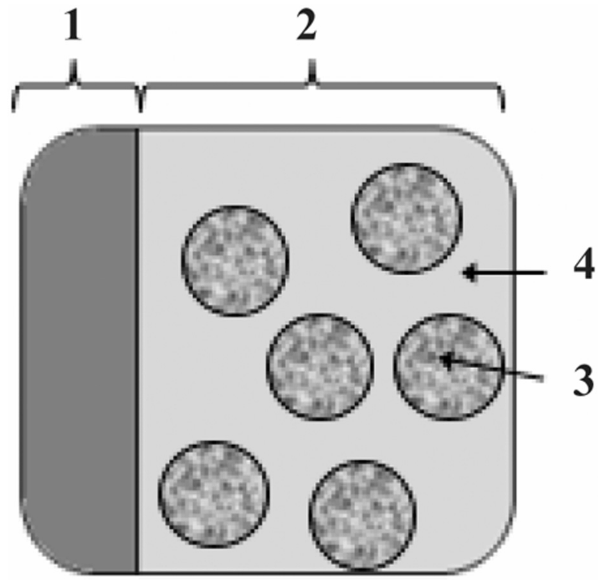


Fig. 13. Schematic of byssus threads structure: 1 – Fibrous Core; 2 – Cuticle; 3 – Granule (DOPA-Fe3+); 4 – Extensible Matrix.

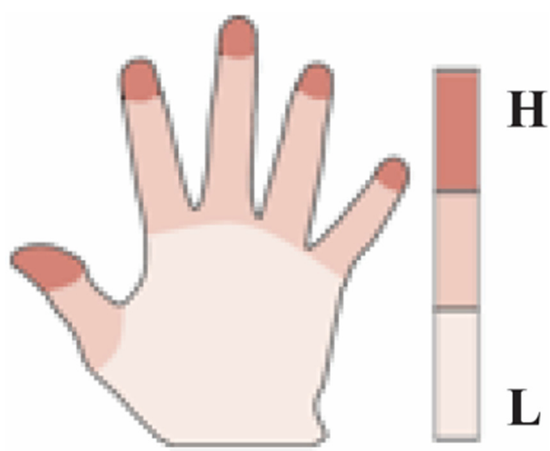


Fig. 14. Skin's mechanoreceptors density distribution: H – high density; L – low density.

3. Engineering approach to mechanical and functional improvement in composites

In the last century, the massive usage of plastics opened the door to the polymeric matrix composites that, combining different materials, took advantage of the low density and mouldability of polymers to perform in a large range of fields, from electrical, thermal, non-structural or structural applications [1,2].

With lightweight and outstanding mechanical performances when compared to traditional materials, FRP produced using long fibres of carbon, aramid or boron, so called advanced composites, have attracted attention of advanced applications, such as aeronautics, aerospace, marine, automotive, sports, etc. [1,2]. Usually produced from dry pre-impregnated materials or liquid thermosetting resin transfer processes, these materials may be used under extreme thermal and mechanical service conditions. However, when exposed to shear, impact and dynamic stresses, they tend to develop internal damages that may propagate during the lifetime of parts, compromising their performance in service. Issues related to the layer-by-layer nature, adhesion between different phases and intrinsic brittleness of these materials, were already identified as the most influent ones to the development of those damages [204–206].

The following sections summarise the most common manufacturing technologies of advanced composite materials and different approaches proposed to mitigate the development of internal damages.

3.1. Composite manufacturing technologies

Nowadays, FRP laminates are widely used in high performance and advanced applications, e.g., aeronautics, aerospace, marine, automotive sports etc., especially due to their exceptional balance between weight and mechanical properties [1]. To achieve the desired performances, it is important to select the appropriate reinforcements and ensure that their content and orientations remain according to those previewed in the design, after manufacturing. Thus, the composite manufacturing techniques being used must accomplish these objectives and, at the same time, ensure a homogenous impregnation over the entire part.

From the several processes used to manufacture advanced composite parts, some use liquid resin to impregnate fibres through the application of positive or negative pressures and other are dry consolidation techniques that use pre-impregnated fibres (prepregs) with a partially cured thermoset matrix (B-stage material) or thermoplastics (using films or other methods). Both present their own advantages, but cost remains has the main difference between them. Dry processes, using pre-impregnated fibres, tend to be more expensive when compared to liquid resin ones [207]. The scheme shown in Fig. 15 presents a general view of the major available composite processing methods.

Among liquid resin moulding processes, RTM is one of the most popular techniques used for producing laminate composite components with very good cost/volume of production ratio. The process uses a fully closed solid mould, where liquid pressurised resin is injected to impregnate reinforcement tissues previously placed inside it. RTM allows producing composite parts with smooth-finishing surfaces on both sides, good thickness control, high fibre contents and, consequentially, mechanical properties [2,208,209]. The process presents several variants, being the mostly known ones the HPRTM (High-Pressure Resin Transfer Moulding) [210][211], VARTM (Vacuum-Assisted Resin Transfer Moulding), SCRIMP (Seemann Composite Resin Infusion Moulding Process) [2,209,212] and SRIM (Structural Reaction Injection Moulding) [209].

The vacuum infusion (VI) process, very often also known as RTM light, presents many similitudes with RTM technology. Dry reinforcement tissues placed inside a sealed solid mould are impregnated by a liquid resin subjected to vacuum. The resin impregnates the reinforcements by being forced to replace air voids and flow throughout fibres due to the differential caused between the atmospheric and vacuum pressures. This technique allows produce large composite parts having low void content, good surface finishing and high mechanical properties [2,212]. The major differences between this technique and the traditional RTM rest in the use of vacuum instead of moderate pressure to force the resin to impregnate the dry fibres as well as in the employment of minimal and cheaper mould structures (namely, a semi-flexible material/structure is commonly used in the upper half part of the mould).

Based on the same principle and due to its lower tooling cost, Vacuum Bag Infusion (VBI) is one of the most successfully variations of VI. By using a flexible half part of the mould (usually, consisting in a thermoplastic film), VBI becomes much more cost-effective than VI. However, only one side of the final part presents good-surface finishing, because the surface contacting the plastic film always shows rougher surface than the one from the rigid mould side [2,213–218]. The VI variation, so-called Controlled Atmospheric Pressure Resin Infusion (CAPRI), allows full control of the pressure differential throughout the laminate, leading to minor thickness variations and much more accurately control of the fibre volume fraction on final parts [219,220].

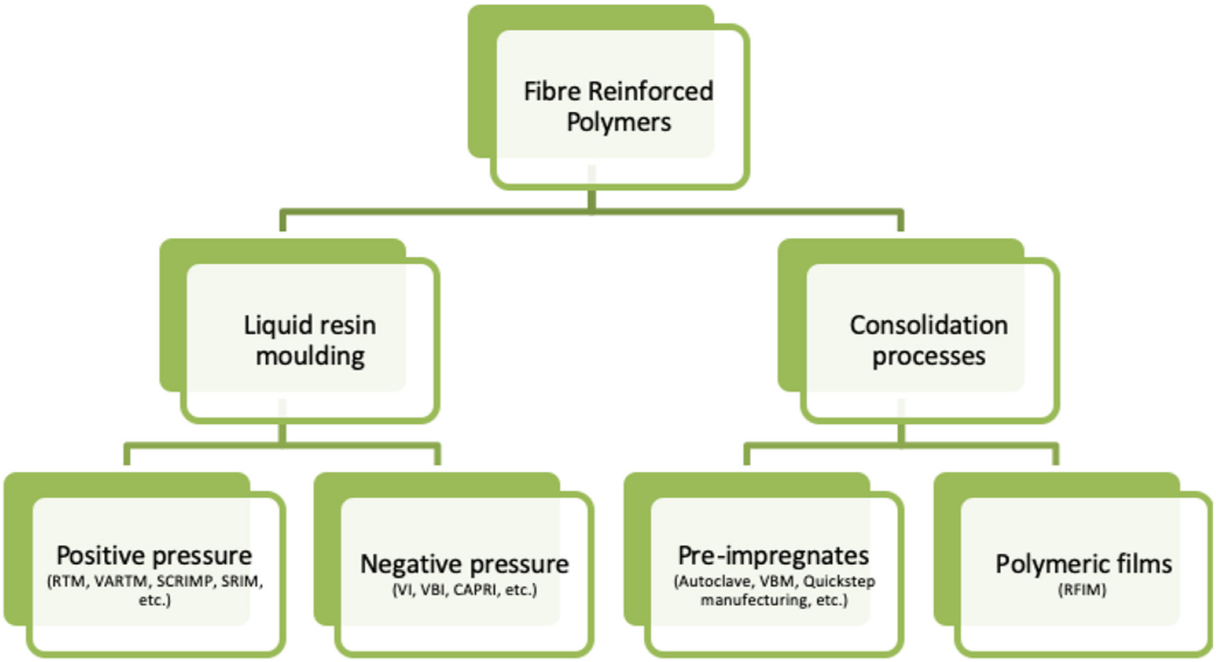


Fig. 15. Composite processing methods general view.

In all processes using fibre impregnation by liquid resins, the resin viscosity and tissues permeability are very important factors to be taken into account due to the risk of obtaining dry spots inside the composite part. Commonly, despite the additional costs that are usually associated to their use, dry manufacturing processes using pre-impregnated fibres (prepregs) are considered more reliable to avoid this problem and, in addition, they also allow obtaining higher fibre contents and control them with much better accuracy in the final composite parts.

Autoclave moulding is one of the processes mostly used to consolidate pre-impregnated fibre tapes and tissues, producing high performance composites for advanced markets. In this process an autoclave is used to consolidate the pre-impregnated reinforcements placed in a mould, simultaneously, maintained under pressure and/or vacuum and subjected to a carefully scheduled temperature cycle. Scheduled stages of temperature allow an accurately control of the curing reaction, while the pressure and/or vacuum ensures the inter-ply consolidation process and void removal. Despite the large initial investment required in equipment and the final part size limitations imposed by the autoclave internal dimensions, autoclave moulding enables good surface finishing on one side of the part and remarkable mechanical properties in final composites [2,207,209,221].

Vacuum Bag Moulding (VBM) is another consolidation process using pre-impregnated reinforcements or hand-lay-up preforms. Using similar setup to the one of VBI, after vacuum-bagged, the resin cures inside an oven or at room temperature. This technique is very common on the aeronautic industry due to the high fibre volume fraction and low void contents that may be achieved and, as in vacuum bag infusion, the lower cost of process apparatus [2,207]. Recently, Quickstep Technologies Pty Ltd. [221–223] developed a new out-of-autoclave (OOA) composite manufacturing process, where vacuum bag moulded pre-impregnates are warm up and pressurize inside a chamber containing a heat transfer fluid (HTF). The technique claims to enable obtaining composites presenting good mechanical properties with significant cost savings.

Another example of consolidation process is Resin Film Infusion Moulding (RFIM). In the process, dry reinforcement tissues are placed between or interleaved by thermoplastic films, and then the whole set is vacuum bagged and heated at high temperatures inside a woven. Due to the temperature, the films melt and impregnate the fibres. Beside outstanding mechanical properties and fibre content accuracy, the production costs can be minimized due to the unnecessity to use pre-impregnated reinforcements. However, the process is time-consuming and requires the use of moulds and accessories able to resist to the high temperatures reached [207,209,212,224].

3.2. Through-thickness mechanical property improvement techniques

Delamination is one of the main causes of failure of advanced polymeric composite laminates. This phenomenon occurs more often in laminates submitted to impact and flexural loadings, in which shear and dynamic stresses are developed. To overcome or mitigate those weaknesses, some attempts were already been made or are being pursued, such as, the use of three-dimensional (3D) woven fibre reinforcing structures or of interlaminar reinforcements with particular properties modification of matrices, optimisation of the adhesion and compatibility of fibre/matrix interface, etc. [5–10,225]. Next paragraphs will describe the mostly common methods to improve through-thickness properties of laminated FRP composites.

The structure of this chapter is represented in the following scheme (Fig. 16).

3.2.1. Through-thickness reinforcements

According to their manufacturing methods, three-dimensional fibre reinforcements are usually divided in: 3D fabrics, Z-pinning and stitching tissues (Fig. 17). Those methods consist basically in creating macro-mechanic bonds between layers improving thereby laminates through-thickness toughness.

3.2.1.1. 3D Wovens. 3D woven fibre structures, usually manufactured by weaving [226], are mainly used as preforms that are placed inside the mould in order to be impregnated by the resin. The use of preforms make their processing much cost effective by avoiding all manual cutting and stacking procedures that could be needed to place them properly in the mould. These reinforcements ensure an effective enhancement of though-thickness mechanical properties

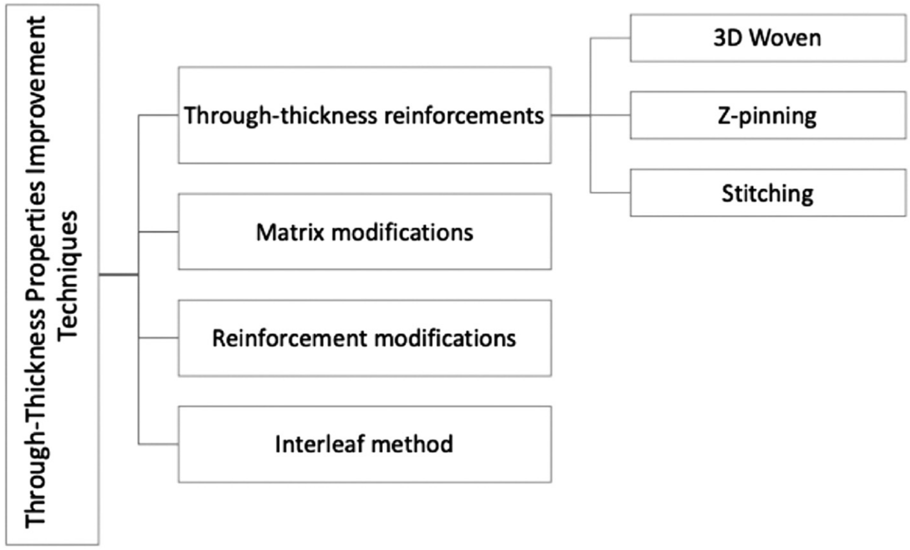


Fig. 16. Schematic representation of Chapter 3.2 - Through-Thickness Mechanical Property Improvement Techniques.

when compared to the traditional layer-by-layer manufactured ones [226,227].

It is well-known that 3D woven reinforcing architectures ensure higher damage tolerance, delamination resistance and impact energy absorption of composite [228–230]. However, there is some reluctance of composites industry using them due to the unpredictability of their in-plane properties [231,232] and behaviour in service [227]. Accordingly to some authors [233], during the manufacturing process of those fabrics, several damages are infringed to a small part of fibre filaments such as, breakage, misalignment and bending. In fact, considering these factors, and also the different laminates and types of fibres used, becomes extremely hard to predict and know what can be expected from the behaviour of composites in working conditions. Misalignment and fibre breakage may compromise the in-plane mechanical properties of composites, whereas gaps close to fibres-crossing areas may create resin-rich zones where cracks may start [226,227]. Regarding these problems and the lack of assessment to the mechanical response of 3D woven composites, it's easy to understand why they are not yet being widely used [227].

3.2.1.2. Z-pinning. The Z-pinning is another technique used to improve delamination resistance of composites, which basically consists on bonding (needling) groups of uncured prepreg laminae by inserting rods (reinforcing fibres) throughout their thickness. These inserting rods, so-called Z-pins or Z-fibres, can be made from fibrous cured composites or metals (titanium, for instance) and present diameters around 0,2-1,0 mm. Owing to their small dimensions, Z-pins generally represents a modest volume content (0,5 - 5 %) in the overall composite [225,234,235].

The Z-pinning method was, for the first time, used in the 1970's to improve though-thickness mechanical properties of composites and have proven to allow enhancing dramatically their damage and delamination resistances [236,237], and the bonding [235] and adhesion properties between the prepreg layers [225,238,239].

To apply Z-pins on prepregs, the laminate stack sequence is first sandwiched between two foam brackets and the Z-pins, previously disposed and aligned perpendicularly over the top bracket, are then mechanically forced to overpass throughout the laminate. After z-pined, the foam brackets are removed and the fasteners that overrun the laminate are cut and, finally, the part is ready to be cured [234,237].

Being easy to implement on existing composite processes, Z-pinning can be considered a promising technique to minimize through-thickness composites problems without relevant associated costs. However, some relevant variables should be taken into account to ensure the good final properties of composites. Some of those variables are: the laminate thickness, volume fraction of the z-pins, their areal density, their perpendicularity and also the material that they are made of [240].

Mouritz [225] evaluate the balance between through-thickness properties (mode I) and in-plane missed performances (tension, compression, bending, inter-laminar shear, and fatigue properties) on Z-pinned carbon/epoxy laminates. Despite the decrease of in-plane mechanical properties, the Z-pinned laminates exhibit an increase of the interlaminar toughness of about 500% relatively to the unpinned ones. The reduction of in-plane properties observed seems to be related to the damage infringed by the needling process to the prepregs fibres and to the resin pockets formed around the fasteners. Other studies conducted by the same author [241,242] have also shown the usage of Z-pins may reduce the delamination area caused by low velocity impacts

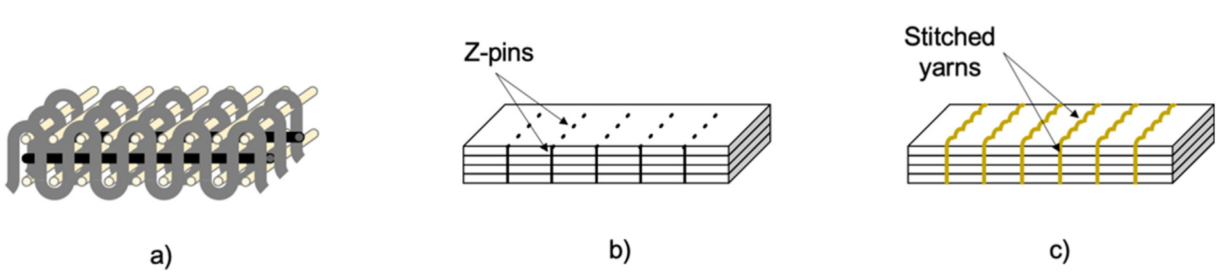


Fig. 17. Scheme of through-thickness property improvement approaches: a) 3D woven, b) Z-pining, c) stitching.

when the impact energy is higher than the threshold energy (in this case around 17 J for 4.3 mm thick samples). It was also demonstrated that the usage of Z-pins increases the damage resistance. However, this improvement was only observed for volume contents up to 2 %, after this amount they did not any additional improvement. Recently, *Francesconi and Aymerich* [243] studied performance of Z-pins on different laminates stack sequences under low velocity impact. In this work, they concluded that Z-reinforcements cannot delay interlaminar damage propagation, but they may reduce delamination size. The main reason for this behaviour was attributed to the activation of Z-pin bridging that reduces damage propagation. However, the same mechanism did not occur when delaminations were distributed across the laminate thickness in small sizes.

Despite the through-thickness property improvement, this technique may lead to a reduction of in-plane mechanical performance of the final composite parts. Such reduction is mainly result of microstructural fibre damages (i.e., fibre breakage) formed during Z-pinning process, misalignments and formation of resin pockets into composite part [37].

3.2.1.3. Stitching. Stitching is a process that uses yarns for bonding dry woven fibres and/or prepregs in order to give them extra though-thickness reinforcement. Some studies demonstrated that the use a small volume fraction of stitching yarns could improve the delamination resistance (in mode I and II) [244–246], impact damage tolerance [247] and dynamic behaviour of structural joins between composite parts [226,231,248].

The technique consists in using needles to stitch yarns throughout the thickness of laminates in order to bond and tie by knots their constitutive layers before impregnation or curing. Aramid yarns are usually employed in the process due to the flexibility they present but, in some cases, carbon or glass fibres are used as well [249]. There are three main types of stitches generally used in the industry: lock stitches, modified lock stitches and chain stitches. Modified lock stitches are the most common, because they use knots made on the surface, which minimize damages caused by needle and avoid larges resin rich pockets inside the laminate. Besides these two aspects, other relevant factors that may also affect the in-plane performance of the laminate, are the yarn denier or stitch density [37,231,248].

Some studies were already carried out in order to evaluate the through-thickness properties of stitched composites. *Solaimurugan et al.* [245] studied the mode I interlaminar fracture toughness of stitched composites with carbon and aramid fibres. They found that strain energy release, G_I, increased 5-23 times when aramid fibre stitches were used. On other hand, *Jain et al.* [246], studying mode II interlaminar toughness in CFRP laminates by using aramid fibre stitches, demonstrated that this property could increase up to 4 times in comparison to unstitched ones. *Francesconi and Aymerich* [250] studied the response to low velocity impact of carbon/epoxied laminates stitched by polyethylene threads. Their work revealed that stitched laminates could reduce damage propagation for impact energies above the threshold value (6 J in this case).

Despite the benefits of stitching on the through-thickness properties of laminate composites, studies made are not also fully conclusive about its effects on in-plane properties, although some of them reported a negative influence and in other cases no alterations or even some improvements were observed. The main reason for this divergence may be attributed to a lack of attention paid to some variables of the stitching process (e.g., stitches tightness) that may affect significantly the mechanical properties of final composites [231,248].

In fact, three-dimensional physical reinforcements do improve the through-thickness properties of composites, giving them better interlaminar resistance, impact resistance and tolerance and performance. However, their influence on the in-plane properties are unpredictable and not completely well-known, in some cases, studies revealed that they may even reduce them, compromising the performance of composites used in advanced applications.

All of those three through-thickness engineering approaches are themselves structurally comparable to mineral bridges structures that can be found in nacre interlaminar region, as described in section 2.1.1..

3.2.2. Matrix modification

Matrix brittleness is another key factor that normally contributes to increase the risk of damage in advanced composite laminates, namely, delamination and facture propagation. Thus, improving matrix toughness and, therefore, its energy absorption capabilities, without compromising in-plane mechanical properties, seems to be a valid approach to increase damage resistance and tolerance of advanced composites. However, due to the extreme aggressive environments that usually advanced composited are exposed, they tend to be manufactured using thermosetting resins (e.g., epoxy resins) as matrices, which, typically are much more brittle than other polymers, namely thermoplastics and elastomers. The high brittleness of thermosetting polymers strongly depends on cross-links density present in their structures [2,209].

Adding plasticisers to thermosetting matrices and reduce the density of their cross-links are methods that were already used to improve their toughness. However, these modifications may cause an undesired increment of viscosity and/or reduce the resin mechanical, thermo-mechanical and chemical performances. Another way of increasing the matrix toughness is mixing it with particles (Fig. 18), however it usually also imply an increment of viscosity and a disadvantageous poor dispersion and resin/particle interaction [36,251–255].

The inclusion of particles or liquid solutions of rubber in thermosetting resins has been studied in order to evaluate their effectiveness to improve toughness and through-thickness properties of advanced composite laminates. *Scott et al.* [256] employed epoxy resins modified with butadiene-acrylonitrile co-polymers (CTBN) as matrices of CFRP composites and verified that they contributed to increase considerably the toughness of laminates, showing an improvement on mode I fracture release energy without compromising in-plane properties. *Kim et al.* [257] also confirmed a significant improvement in interlaminar fracture toughness (mode I) behaviour of CFRP composites using rubber-modified matrix resins, when compared to those produced with unmodified ones. However, under transversal impact test (Charpy impact test) at ambient temperature or above, CFRF laminates using rubber-modified resin present worse results, compared to unmodified ones. Subsequently, in a posterior study concerning the post-impact mechanical properties of CFRP laminates [258], the same research team concluded that the rubber-modified matrix composites presented improvement up to 80% on delamination fracture energy (mode I) and an increase of 25% of flexural strength and modulus, in comparison to the unmodified matrix ones.

Despite the small decrease of pristine resin modulus, the incorporation of reactive thermoplastic modifiers into epoxy resins also has shown to lead to an increase on their fracture toughness. Typically, the thermoplastic modifiers previously incorporated into the epoxy resin generate a second phase separation during the curing process. *He at el.* [259] studied the micro-cracking behaviour on epoxy resins modified with poly(ether imide) (PEI), polycarbonate (PC), and poly(butylene terephthalate) (PBT) to produce CFRP composites for cryogenic applications. Using dynamic mechanical analysis (DMA), they found that all modified resins had proved to present an increasing in their storage module and a decreasing on the coefficient of thermal expansion, when compared to the unmodified ones. Optical microscopy also revealed that PEI and PC are more effective to improve micro-crack behaviour owing to their low coefficient of thermal expansion and high impact strength.

A better thermoplastic/thermosetting interfacial bonding conjugated with the decrease of the crosslinked of the thermosetting resin leads to a higher toughness [260]. However, despite an increment of

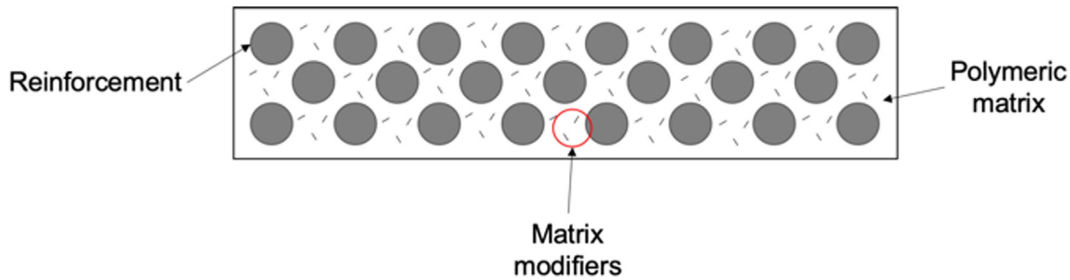


Fig. 18. Scheme of matrix modification approach.

thermosetting resin viscosity promoted by the modifiers, during the impregnation process, reinforcement fibres may act as a filter, inducing a pure toughing distribution across the composite part.

The inclusion of polymers with spherical molecular structures was already considered as an alternative to increase the thermosetting resin toughness due to their much moderate viscosity increment. The spherical structure of dendritic hyperbranched polymers (HBP) have shown to cause much less viscosity increase, for almost the same molecular weight increment, when compared to linear polymers. Moreover, being their structural morphology being core-shell type, a large variety of chemical arrangements may be promoted on the shell, enhancing a better bonding to thermosetting resins [251,252]. Several works [251,252,261,262] revealed significant improvements on toughness of thermosetting resins modified with HBP, with only a slight loss of stiffness. The lower increment of viscosity, in comparison to other matrix modifiers, have shown that HBP are also better suitable to produce advanced composites parts using liquid resin impregnation processing techniques.

The use of thermoplastic homopolymers to modify epoxy resins, depending on the percentage of incorporation, usually leads to undesired increments upon the viscosity due to higher molecular weight of the added polymer [254]. To overcome the problem, some research works have been carried out on the incorporation of copolymers in epoxy resins. *Denneulin et al.* [263] studied the behaviour under low velocity impact tests of aramid reinforced composite laminates produced from epoxy resins incorporating acrylate based block copolymer additives. It was found that the produced laminates from the modified epoxy resins presented better performance under impact loads due to nanostructures created by the block copolymers.

Recently, the interest from the use of nanoparticles to modify the polymeric matrices, such as exfoliated graphite, metals, silica, carbon black, carbon nanofibers (CNF) and carbon nanotubes (CNT), have also grown due to the improvements (with only a small content) that they could bring to the mechanical, thermal and electrical properties of polymers. However, those improvements are strongly dependent on the interfacial relationship between matrix and filler and degree of dispersion of it [5,6]. Another benefit that may result from the use of nanoparticles is the low increment on viscosity that they cause on the matrix in comparison to other microparticles and this advantage makes them more suitable for production of advanced materials by liquid processing techniques [6,264]. However, it is not already clear if the fibre reinforcements bed acts as a filter for nanoparticles during resin flow [264,265].

Since their discovery in the 1990s, CNT have been exhaustively studied due to their remarkable mechanical, thermal and electric properties. *Godara et al.* [266] studied the effect of incorporation of different types of functionalized and non-functionalized CNT in epoxy resins used in the production of prepregs for CFRP composites. They reported a reduction in coefficient of thermal expansion for resins modified by using thin-multi-wall (TMWCNT) and double-wall CNT (DWCNT), and a substantial improvement in ILSS and mode I crack initiation energy of MWCNT–epoxy system with a compatibilizer. *Ashrafi et al.* [267] also produced prepregs made from an epoxy resin modified by functionalised single-wall CNT (SWCNT). Composites manufactured from those prepregs revealed that the use of only 0,1 wt% of SWCNT enabled the reduction of impact damage area (5 %), higher compression-after impact (CAI) strength (3.5 %) and the increase in mode I and II interlaminar fracture toughness (13 % and 28 %, respectively).

Similar to the energy dissipation mechanism of bivalve molluscs' byssus threads (described in section 2.4.), doped synthetic matrices have proved to enhance their toughness and delay catastrophic failure by matrix diffused microcracking.

3.2.3. Reinforcements modification

The majority of failures observed in advanced composite materials occur in interface between fibres and matrix. One strategy to improve bonding between these two phases is to apply surface treatments to the fibre surfaces, in order to promote physical or chemical adhesion between them and the matrix. Some examples of surface treatments applied on carbon fibres to improve adhesion to matrix are oxidation treatment, coating and plasma processing methods [39,268].

Plasma treatment of carbon fibres is an effective method used to improve the bonding characteristics of the surface of fibres to the matrix. It usually brings roughness to the fibre surface and generates polar groups, both contributing to improve the interfacial adhesion and fibre/matrix loading transfer. Studying the effect of oxygen plasma treatment and isobutylene plasma polymerization on carbon fibres, *Pittman et al.* [269] concluded that the oxygen plasma treatment had increased the interfacial adhesion and interlaminar shear strength of composites without any significant effects on the tensile strength of fibres.

The application of electrolyte solutions on the carbon fibres was investigated by *Ma et al.* [268] as electrochemical surface treatment. Using sulphuric and phosphoric acids, sodium sulphate, sodium phosphate, ammonium bicarbonate as electrolyte solutions, they found that all of them promoted the physical bonding of the carbon fibres to the matrix.

Recently, the use of the so-called multi-scale composites has been seen as a promising method to improve toughness of composite parts. Incorporation of CNT on reinforcements surface (Fig. 19) appeared as an effective technique to overcome problems caused by stress concentration, voids and in-plane misalignments of fibres, that may result from other techniques such 3D woven fabrics, stitching and Z-pinning manufacturing [7]. Using electrophoretic deposition, *An et al.* coated successfully glass [270] and carbon [271] fibres with CNT. The coated fibres, used to produce composites plates by vacuum assisted resin transfer moulding, have shown an effectively improvement on shear strength and fracture toughness of final laminates. *Xu et al.* [272] proposed a new method to deposit directly CNT on carbon fibres surface. By using a floating device and the catalyst chemical vapour deposition (FCCVD) method, CNT aerogel is blown out directly from the FCCVD oven to the carbon fibres. Composite plates using those carbon fibres were then produced by hot-pressure moulding, and have shown higher flexural strength (16,04 %) and interlaminate shear strength (21,51 %) than composites produced by uncoated fibres.

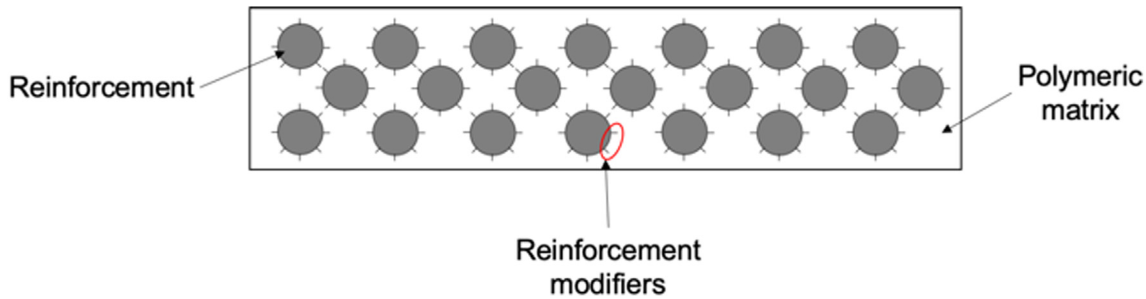


Fig. 19. Scheme of reinforcement modification approach.

The coating of carbon nanotubes forests grown direct on carbon fibres surface, using the chemical vapour deposition (CVD), shows up as an effective method to improve the loading transfer mechanisms and avoid problems, such as, agglomerations or uneven dispersion of CNT on the composites [273]. Single fibre testing showed that the presence of vertical aligned carbon nanotubes (VA-CNT) on the carbon fibres surface enhances the interfacial shear strength of composites and that such improvement results, essentially, from the higher interphase adhesion and strength between matrix and fibres promoted by the presence of VA-CNT [274,275].

In a work carried out by *Veedu et al.* [273], CNT have been grown by CVD on SiC fibre fabrics that were, then, impregnated with epoxy and cured in autoclave to produce composite laminates. The final 3D hybrid composites produced presented 348 % and 54 % higher values of interlaminar fracture toughness in mode I and II, respectively, than those without CNT. Moreover, in-plane mechanical tests revealed improvements in strength, modulus and toughness, which means that these properties were not affected by the increasing of the through-thickness properties. *Kepple* and his co-workers [276] reported as well an increase of 50 % in fracture toughness (mode I) in composites made from carbon fibres grafted with VA-CNT impregnated by an epoxy resin. *Wardle* [277] carried out a work to evaluate the performance of hybrid composite architectures using VA-CNT grown by CVD on the surface of microfibers. These hybrid architectures had shown to provide to composites more multifunctionalities and higher interlaminar shear strength. In another work, *Wardle* [278] reported that the same architectures presented improvements in both initiation and steady-state toughness when submitted to mode I interlaminar fracture tests and, an increase in bearing stiffness, critical and ultimate strengths when submitted to tension-bearing test. Interestingly, *He* [279] evaluated how the length of VA-CNT grown on carbon fibres surface, embed in two different epoxy resins as matrices, could influence in mode I fracture toughness. This property showed an increase in composites grafted by longer VA-CNT, due to the formation of a tortuous fracture growing paths and consequent larger crack propagation surface area. Contrarily, the resin type did not show a significant impact in toughness properties.

These reinforcement modifications, that yield surface roughness, may be compared to nacre interlocking mechanisms, i.e., asperities, mentioned in section 2.1.1..

3.2.4. Interleaf method

It is well-known that interlaminar regions of composite laminates play an important role on their mechanical performance, often showing up as common failure place due to their intrinsic brittleness resin-rich zone [280]. The interleaf (or interlayer) technique was introduced and studied as an attempt to improve the response to damage and increase the energy dissipation in these regions of laminates.

The interleaf technique consists in inserting, for instance, films, nonwoven tissues, self-same resin films or other kind of materials or structures, between composite laminate layers (Fig. 20) to increase the plastic behaviour and provide higher damage resistance to those regions [8,36,281–283].

The use of more ductile resins in interlaminar layers to increase the composite laminates toughness has been used for a long time. In the 1980's a "high-strain resin" employed between the laminate layers had proven to improve the composite compression strength after impact. After this, many types of materials and structures have been tested as a possible solution to improve damage resistance and tolerance of advanced composite materials [284].

Singh et al. [284] studied carbon-fibres modified-thermosetting matrix prepregs interleaved with self-same resin films in order to study crack mechanisms and toughness in unidirectional composite laminates. Films made of prepregs self-same resin (cyanate ester/epoxy) were placed into the central resin-rich region where the crack would start on mode I and II interlaminar fracture tests. An impressive enhancement on composites delamination resistance of 50% and 200% in mode I and mode II, respectively, was observed when compared to non-interleaved ones. Toughness enhancement observed in both loading configurations was considered related to the constant reorientation of the crack front though the isotropic resin-rich layer.

Interleaved carboxil-terminated butadiene acrylonitrile (CTBN) and polyurethane (PU) modified epoxy resins were used by *Jang* and *Chen* [285] for studying the fracture behaviour of carbon/epoxy laminates. By spraying a thin layer of the modified resin on the surface of prepregs, they observed an increase of 40-50 % and 200-300 % in mode I and II fracture toughness, respectively, and an improvement in damage resistance of laminates. They also concluded that as higher was the thickness of the interleaved layer, as higher was the laminate fracture toughness obtained. *Cheng et al.* [286] studied the application of a polyetherketone with a phenolphthalein side group (amorphous thermoplastic) (PEK-C)

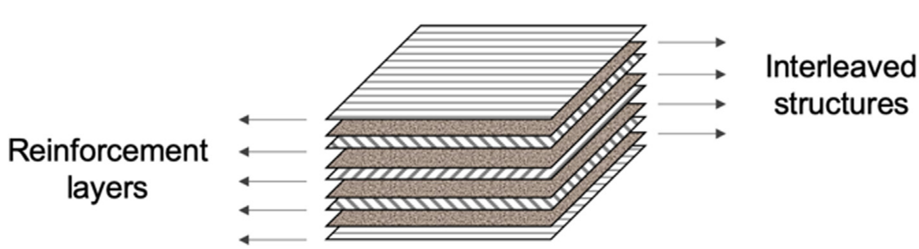


Fig. 20. Scheme of interleaf approach.

to tough carbon fibre laminates. They compared neat bismaleimide resin (BMI) matrix laminates with the following ones: BMI modified by PEK-C matrix (in-situ toughening), BMI interleaved by PEK-C films (ex-situ toughening) and BMI interleaved by BMI/PEK-C blend films with different concentrations (ex-situ toughening). The work revealed that interleaved laminates, when compared to that produced with neat BMI and BMI modified by PEK-C (in-situ) as matrix, had better performance regarding damage resistance and higher compression after impact (CAI) strength, particularly those ones that were interleaved by films composed by PEK-C and BMI.

Yasaee et al. [287] inserted thermoplastic film rings between layers to confine the damage area in low velocity impact and compression after impact (CAI) tests. Using different stack dispositions of polyimide film rings in glass-fibre/epoxy laminates, they restricted and reduced the impact damage area up to 38 % and increased the CAI strength up to 18 %. However, they also observed that some laminates presented worse behaviour due to the important role that fibre orientation plays in impact damage.

CNT were also used as interleaved structures for providing interlaminar toughness to composite laminates. *Chen et al.* [288] inserted between layers of a carbon/bismaleimide (BMI) composite, polyetherketone with a phenolphthalein side group (PEK-C) neat films and modified with MWCNT, to study how they work as interleaf tougher structures. Results obtained shown that the interleaved laminates, specially MWCNT modified films, reduced damage area in low velocity impact tests and improve CAI strength up to 33 % when compared to non-interleaved laminates. In other study, *Liu et al.* [289] incorporated CNT Buckypapers (BP – thin layer of CNT with random orientation) directly into interlaminar region of unidirectional prepreg composites. To investigate interlaminar fracture toughness in mode I and II, they used two different types of BP to interleaf carbon/epoxy laminates: as-prepared CNT BP and cross-linked CNT BP. Cross-linked CNT BP interleaved laminates provided an enhancement in interlaminar fracture toughness of 74 % and 82 %, in mode I and mode II tests, respectively. These improvements in toughening were attributed to the better interfacial bonding between CNT, to bridging mechanisms through a higher fracture surface area and to the extra force needed to pull out CNT from the resin.

Carbon nanofibers (CNF) BP were also studied by *Khan et al.* [290] to be used as interleaved tougher system in composite laminates. After impregnating CNF BP by epoxy resin, they were pre-cured and interleaved between unidirectional prepreg layers to produce CFRP laminates. Interleaved CNF BP shown to be an efficient tougher structure in interlaminar shear strength (ILSS) tests, improving 31 % shear strength, and 104 % in mode II interlaminar fracture toughness. ILSS improvements were attributed to the increasing of matrix shear properties resulting from the inclusion of CNF and better interfacial CNF/epoxy adhesion. On the other hand, the mode II improvement was associated to the better bridging mechanism, the higher CNF pull out force and the crack deflection.

The incorporation of VA-CNT in the interlaminar region of CFRP laminates was also investigate by *Wardle et al.* in [7] and [291,292]. By transplanting VA-CNT grown by CVD to prepregs surface, they create a kind of “nanostitching”. Interlaminar fracture toughness in mode I and II [291], bolt pull out critical strength, open-hole compression ultimate strengths and L-shape laminate energy [7] properties were investigate. VA-CNT provided to the composite specimens an improvement of 150-250 % and 300 % in mode I and mode II fracture toughness, respectively, being also reported improvements of 30 % in bolt pull out critical strength, 14 % in open-hole compression ultimate strength and about 25 % in L-shape laminate energy. The main tougher mechanisms responsible for those improvements were attributed to interleaved layer toughness, plastic deformation and crack bridging.

Recently, *Stahl et al.* [293] improved 2-3 times the interlaminar fracture toughness in mode I of carbon fibre composites, by interleaving arrays of horizontal aligned CNTs. Forests of multi wall carbon nanotubes grown by CVD were knocked down by a shear mechanical process. The horizontally aligned CNT obtained were then pre-infused by a low viscosity epoxy resin and interleaved between unidirectional carbon fibres prepregs. The results obtained have shown an increase of interlaminar fracture toughness is, probably, caused by the complex delamination tortuous path created through interlaminar films, crack deflexion to adjacent composite plies and a combination of both effects.

Thin nonwoven tissues, so-called veils, were also used as an interleaf structure to tough composite laminates. Their high permeability allow manufacturing hybrid interleaved composites using liquid resin processing techniques, such as, RTM, vacuum bag infusion, among others [281]. Incorporation of nonwoven carbon fibres between carbon prepregs to produce composite laminates, was first studied by *Noguchi et al.* [294]. Their initial studies demonstrated that these structures could improve in-plane shear behaviour, although a reduction on static tensile strength was reported. In-plane shear property improvement was found to be related to the delay of matrix cracking and delamination promoted by nonwoven carbon tissues. Improvements in interlaminar fracture toughness were also reported by the usage of the same interlaminar tougher. In mode I, interlaminar fracture toughness, an improvement of 28 % was reported in interleaved laminates when compared to non-interleaved ones. The mechanism responsible for these improvement was attributed to the breakage out-of-plane of the short fibres present into interleaved layer [295]. Subsequently, *Kuwata et al.* [9,10] studied the effect of using different types of veils in interlaminar fracture toughness mode I and mode II. In mode I tests, thermoplastic veils (polyester and polyamide) shown to be more efficient as a tougher system. Debonding between nonwoven fibres and the resin, in addition to the ability of plastic deformation of nonwoven fibres in themselves, shown up as the main tougher mechanisms. However, the adhesion between fibres and resin and the areal density of the fibre reinforcement seem to be important aspects to be considered. Results from interlaminar fracture toughness tests made in mode II were not so conclusive due to complex mechanisms associated to the application of loads, while seems to be more likely that nonwoven tissues architecture and shear resin properties played a much more important role in this case than in mode I tests. Recently, *Sampson et al.* [296] studied the effect of nonwoven thermoplastic tissue architectures, namely, polyphenylenesulfide (PPS) and polyetheretherketone (PEEK), in the interlaminar fracture toughness behaviour. No significant differences were observed between PPS and PEEK veils for the same areal density. The study revealed that in mode I the nonwoven tissues with higher areal density increased interlaminar fracture toughness, however a better performing was observed when the veils formed by low linear density (density per length unit) fibres were used. In mode II, the same dependence of veils areal density and improvement of interlaminar fracture toughness was observed, while no strong relation with linear density of fibres was observed. They reported also, that mode I and II interlaminar fracture toughness do not depend on fibres linear density in comparison to veil coverage, so they concluded that both mechanisms depend upon the fraction of crack propagation in the veil uncovered surface. Recently, *García-Rodríguez et al* [297] introduced a low melt temperature copolyamide (coPA) fibre veils between each single interlaminar region of carbon/epoxy laminates and found that, in some cases, this allowed to increase the CAI strength and reduce low velocity impact damage area in more than 100 %.

Electrospinning technology allows to produce nonwoven thermoplastic nanofibers tissues. Nonwoven nanofibers tissues produced by this process have higher surface bonding area to the matrix and, besides, they allow higher coverage without increase significantly weight and thickness [298]. Only a few studies of electrospun nonwoven tissues applied in structural composites can be found in literature, however the effective enhancement of composite interlaminar toughness has been already reported. *Beckermann* and *Pickering* [299] studied the effect of a range of different polymeric electrospun nonwoven tissues with various areal weight and fibre diameters in mode I and II interlaminar fracture toughness. The study showed that 4,5 g/m^2 polyamide 6'6 (PA66) nonwoven tissue seemed to be the best option when compared to the others polymeric nonwoven tissues tested, presenting an improvement of 156 % and 69 % of interlaminar fracture toughness in mode I and II, respectively. According to the authors, areal weight has influence in mode I interlaminar fracture toughness, increasing this property until a maximum of 4,5 g/m^2 and G_{IC} tends to stabilise above this value of areal weight. In mode II, the influence of areal weight seems to be similar as in mode I at the beginning, but G_{IIC} presented a slight decrease after the 4,5 g/m^2 areal weight. However, they didn't find any influence of fibre diameter in studied properties. *Van der Heijden et al.* [300] interleaved electrospun polycaprolactone (PCL) nanofibers on glass fibre/epoxy composites produced by VARTM. The study showed that the amount of nanofibers and the way they are placed between layers can influence mode I interlaminar fracture toughness. Composites containing 20 g/m^2 of PCL nanofibers directly electrospun over the both sides of glass fibre tissue surfaces before impregnation, improve almost 100 % mode I fracture toughness. Moreover, PCL nanofibers seemed to not influence the impregnation process, and neither tensile nor dynamic composite mechanical properties.

Those progressive cracks reorientation and bridging throughout the interlaminar region avoid catastrophic failure and reduce interfacial debonding, quite similar to nacre and bone energy dissipation mechanisms described in section 2.1.1. and 2.1.7., respectively.

Regarding the overview on advanced composite materials above presented and, despite all those approaches to enhance advanced composites mechanical properties have already demonstrated effectiveness to produce high performance engineering structures, several issues, such as fibre damages, increment of resin viscosity, thickness and weight overage, among others, still need to be overcome. On the other hand, composite manufacturing processes can be helpful to develop novel bioinspired materials/structures, which, at the same time, may provide additional ideas and different points of view to develop new successful engineering approaches and produce higher performance advanced composites.

4. Bioinspired engineering approaches

After the overview of the natural structures and the engineering approaches, it is proposed a set of bio-inspired solutions for mechanical improvement and self-sensing of composite structures. These crosses both Nature solutions (Chapter 2) and composite technologies (-Chapter 3) developments in order to improve mechanical performance and self-sensing of the composite structures.

4.1. Bioinspired mechanisms of composite toughening and sensing

Base on previous descriptions, some structural concepts for composite toughening mechanisms were selected, whose bioinspired and technology approaches are summarized in Table 1. Main objective of these approaches is to improve the energy absorption capabilities of the composite structure, namely, their fracture, impact or fatigue resistances. Several structural concepts are proposed based on different bioinspired approaches already abovementioned:

1. Brick-mortar structure, as in nacreous shells, taking advantages of their rigid-soft materials combinations and several interlocking mechanisms;
2. Bouligand structure, as in arthropod exoskeletons, showing a helical twisted stacking laminate with several energy dissipation mechanisms;
3. Granular inclusions, as in the doped matrix of mollusc byssus threads, with matrices and inclusions materials with distinct stiffness.

The main processes selected to produce the bio-inspired composite structures are based on conventional composites manufacturing technologies, e.g., vacuum bag infusion, complemented by 3D printing. Based on 3D architectures, it is suggested to use stacks of pre-cut reinforcing ultrathin veils of different geometries. These thin veils can be surface patterned by 3D printing with micro-roughness structures, mimicking the asperities observed in nacre. Also, VA-CNT pins can be applied on uncured thin films in order to improve interface bounding (e.g., local z-pining). The bio-inspired helical structures could be incorporated in composites by vacuum bag infusion through interleaf reinforcement, whose helical arrangement could be asymmetric with 13.33° rotation angle or symmetric to overcome infusion problems. Based on the composition of the mollusc byssus threads, it is suggested to dope the matrix resin with CNT, in order to mechanically reinforce the matrix. These bioinspired technological approaches can be combined in the same composite structure.

4.2. Bioinspired mechanisms of composite sensing

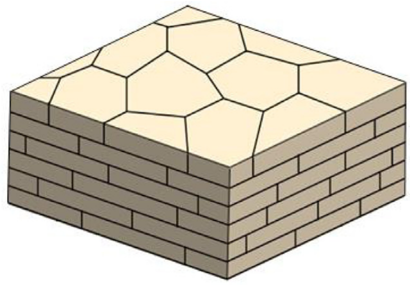
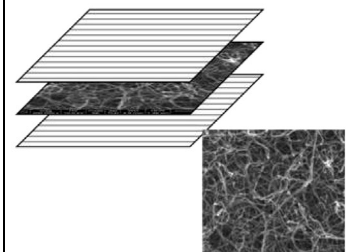
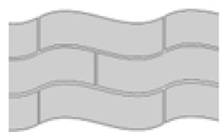
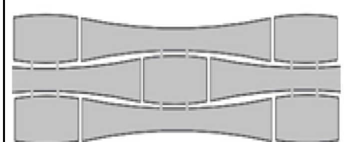
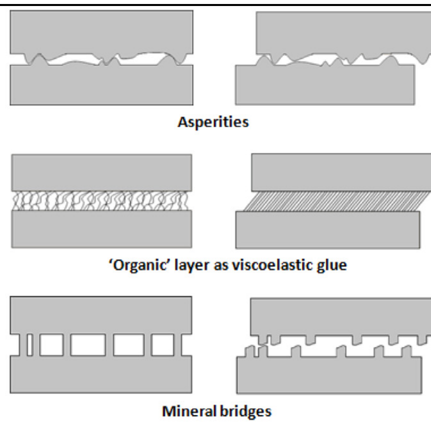
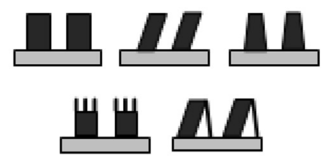
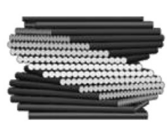
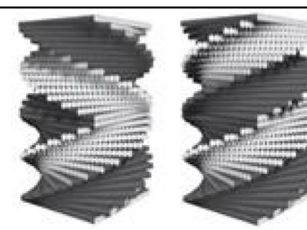
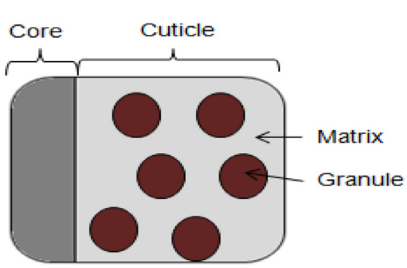
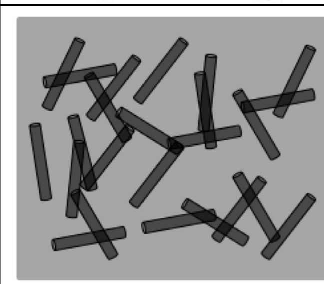
Based on the above described sensing mechanisms, some technological concepts for composite sensing were also selected, their bioinspired and technology approaches being summarized in Table 2. The main objective of these selected approaches is to add sensing functionalities (e.g., impacts, loadings, damages) to the composite structures. Several sensing concepts are proposed based on different bioinspired approaches already above mentioned, with focus on the large area and multisensing of human skin and sensory system.

From aeronautics to biomedical applications, it is still a challenge the development of highly efficient and sensitive system for critical structures or components. Specifically, in aeronautics, the urge of lower maintenance costs and faster damage diagnostic trigger the development of CNT based sensors [11,301,302], due to their outstanding conductive and mechanical properties. The proposed sensing mechanisms can be based on the use of CNT doped polymer matrix to create highly electric conductive materials. Carbon nanofibers coated fibres can introduce new functionalities in glass fibre reinforced polymer composites, including higher electrical conductivity [303]. Also, CNT can be grown directly on the surface of advanced fibres and used to produce Fuzzy fibre reinforced composites with improved mechanical and electrical properties [277]. Based on the highly sensitive mechanosensory system of human skin, it is proposed to assemble a network of oriented CNT based sensors to locate and quantify an impact on a surface. The impact detection can be based on piezoresistive effect using VA-CNT embed in a matrix or on piezoelectric effect using horizontal aligned CNT [280–283]. VA-CNT embedded in a polydimethylsiloxane substrate have been used to produce a flexible capacitive sensor [304]. VA-CNT can also be used between composite laminate plies to form a piezoresistive sensor and add a sensing function to the structure. Recently, VA-CNT were knockdown on a polymeric substrate to produce a piezo-resistive sensor [305]. Sensing capabilities can also be introduced in composite laminates using conductive ultrathin plies (e.g., in GFR composites), by thin film substrates printed with conductive inks or by knockdown VA-CNT, or by 3D printing of conductive materials on the laminae(a) surface(s).

5. Concluding remarks

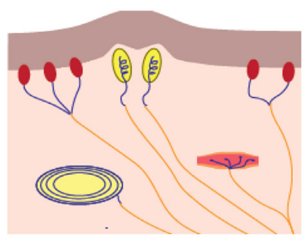
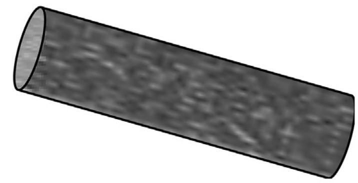
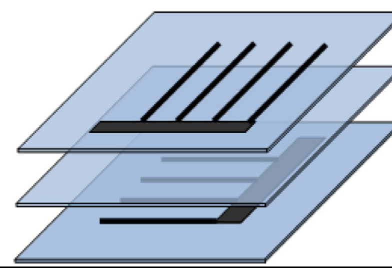
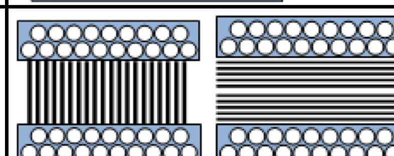
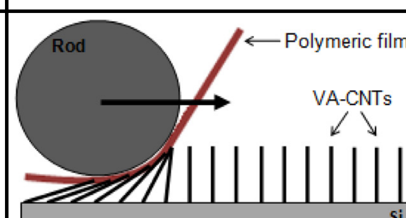
Nature has been prodigious in adapting itself and in developing solutions to optimize the performance and functionality of biological materials. Despite the ability of engineering to produce high mechanical performance and even multifunctional composite materials, the growing need for better and outstanding materials made us turn to the natural approach. Crossing both Nature solutions and engineering composites developments is a route for the manufacturing of high mechanical performance and self-sensing composite structures. Therefore, several biological structures and their mimicry, as well as, the engineering composite production and damage mitigation processes are reviewed.

Table 1
Summary of bioinspired and technology approaches for composite structures toughening.

Functionality	Structural concepts for toughening	Bioinspired approach	Process/ Solution	Technology approach
Improved energy absorption: Fracture resistance/ Impact resistance/ Fatigue resistance	Brick-mortar		Ultrathin veils	
	Waviness/ dovetail		3D printing	
	Asperity/ anchoring		Surface patterned ultrathin veils (3D printing)	
	Planar twisted plywood/ Bouligand structure		Helical stacked layup	
	Granular inclusions		CNT doped matrix	

Despite the efforts dedicated to mimic these natural structures and their multifunctionalities, their production and integration in advanced composites are still at an early stage, being an interesting technological-scientific challenge for the development of innovative solutions. Thus, several bioinspired approaches regarding mechanical improvement and multifunctional properties are also proposed, namely: brick-mortar structure, as in nacreous shells; Bouligand structure, as in arthropod exoskeletons; granular inclusions, as in the doped matrix of mollusc byssus threads. The main processes selected to produce the bio-inspired composite structures are based on conventional

Table 2
Summary of bioinspired and technology approaches for composite sensing.

Functionality	Sensing mechanism	Bioinspired approach	Process/Solution	Technology approach
Sensing (Impacts/Loadings/ Damages)	Skin sensing large area Skin multisensor Immune system sensors	  	CNT doped matrix	
			CNT coated fibers	
			Conductive ultrathin ply	
			Electrical grid - 3D printing/ Inkjet printing/ CNT knockdown	
			A-CNT – piezoresistive	
			VA-CNT – Knockdown	

composites manufacturing technologies, e.g., vacuum bag infusion, complemented by 3D printing.

This bioinspired engineering approach will allow the development of the next generation of composite structures, with better mechanical, thermal and electrical properties, and with self-sensing and self-healing capabilities.

Uncited references

[67,69,70,72–75]

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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