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Review

Hygrothermal ageing of composite structures. Part 2: Mitigation techniques, detection and removal

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ABSTRACT

The first section of this work is dedicated to the exposure of possible methodologies and solutions that can be implemented to face hygrothermal ageing effects, avoiding reduction of service-life and increasing safety margins. It's underlined the importance of the material and manufacturing process selection, and its impacts on issues related to the hygrothermal ageing. Nanofiller, Surface and fiber treatments are investigated and the their influence on the moisture uptake is discussed. The second part is dedicated to the technique used to detect and remove moisture. It shows how an early detection and the moisture removal can be useful to elongate the service-life and to avoid irreversible loss of mechanical properties. Particular emphasis is given to innovative structural health monitoring (SHM) systems combined with fiber optic sensors, which allows to check on board, real-time, the status of composite component subjected to moisture aggression, thus keeping high performance standards, while preventing failures.

1. Introduction to moisture related issue in composite structural components

Composite materials has greater mechanical properties compared to traditional metallic materials: mainly strength, stiffness and resistance to fatigue, but also greater strength-to-weight ratio, corrosion, impact resistance, design and manufacturing process flexibility, low coefficient of thermal expansion and resistance to high temperatures as described by E. Kececi in [1], Y.F. Niu et al. in [2], N. Zimmermann and P.H. Wang in [3], B. C. Ray et al. in [4] and A. Krauklis in [5]. Thanks to their property, in the last decades, composite materials have been widely used in the aerospace sector for primary structures such as fuselage, wing, flaps, fairings, but also for other non-structural interior aircraft parts. Another common application of these advanced materials is for wind and tidal turbine blades. C. Kassapoglou in [6] stated that the main reason to prefer composites materials in aerospace field to traditional configurations lies in the better strength-to-weight ratio.

As shown in Part 1 [7], composite structures used for aircrafts, wind and tidal turbine blades are placed, most of the time, in direct contact with the external environment (humid air of the atmosphere or sea water), therefore they interact easily with atmospheric agents or water and they are more prone to suffer moisture damage during their operation. In fact, composite materials absorb more moisture from the environment, compared to traditional materials, with a detrimental effect on mechanical, physical, electrical, thermal properties and service life reduction as shown by K. Shetty et al. in [8] and B. C. Ray et al. in [4]. More in detail, E. Kececi and R. Asmatulo in [9] observed that sandwich panels and adhesive bonded joints tends to enhance the moisture ingress since they are not perfectly isolated from the external environment and they are realized by connecting more parts together (laminate layers, substrates, adhesives, honeycomb core), with an increased risk of water penetration at the interfaces. Since moisture-induced damage can seriously affect component performance, high reliability standards must be maintained to meet the requirements of regulatory authorities, which increases the cost of wind and tidal turbines as showed by D. A. Katsaprakakis et al. in [10].

Part 2 focuses on the detection, removal and mitigation techniques used to oppose hygrothermal ageing. Section 2 analyses the techniques used to reduce moisture absorption in composite materials. The importance of materials (matrix and fibres) and manufacturing

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Abbreviations: AE, Acustic Emission; APPT, Atmospheric Pressure Plasma Torch; CNT, Carbon Nanotube; FBG, Fibre Bragg Grating; NDT, Non-Distructive Techniques; PLA, Poly-lactic Acid; QD, Quantum Dots; SEC, Size Exclusion Chromatography; SEM, Scanning Electron Microscopy; SHM, Structural Health Monitoring; UAV, Unmanned Aerial Vehicle.

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techniques selection is presented. For several solutions are indicated advantages, drawbacks and mitigation mechanisms.

Section 3, on the other hand, investigates the maintenance techniques that are used to identify absorbed moisture or moisture-related damage in all critical components identified in part 1 [7]: sandwich structures, adhesive joints, wind turbines, tidal turbines and bio-composites. Removal techniques are also presented, emphasising the importance of minimising their invasiveness, and showing how non-compliant repairs can worsen the behaviour of components subjected to an hygrothermal load. It is highlighted how early identification and removal can prevent the onset of effects leading to irreversible loss of mechanical properties and consequent damage. According to this, structural monitoring systems capable of providing real-time monitoring and early diagnostic information are of paramount importance. Very often, such systems, use fibre optics and FBG sensors.

2. Techniques to reduce humidity uptake in composites

Several techniques and solutions can be applied to mitigate the problem of hygrothermal ageing in composites. This can be done already in the composite production phase, but also during maintenance and by using monitoring techniques to check the health status of the structure in response to moisture absorption. From the design point of view, the choice of composite materials (resin and fibers) less sensible to water aggression is fundamental but is generally not sufficient to face the problem of ageing, so other solutions must be found to counteract humidity damages.

Special treatments can be applied to the fiber surface to enhance their resistance to water absorption and voids formation at the fiber/matrix interface level. The use of surface treatments is required especially for the protection of bio-composites from hygrothermal ageing, since they tend to be more sensible to water absorption on respect to traditional composites. In addition, appropriate non-destructive maintenance techniques can be used to remove the water content accumulated inside the structure, but they require a certain downtime where the component cannot be operative.

Finally, more efficient solutions are presented in section 3, involving the development of real-time structural monitoring systems (SHM), which are an evolution of the standard and known maintenance procedures like the non-destructive techniques, since they are more effective in damage prevention. To maximize the efficiency of these monitoring systems to detect moisture absorption in advance and contrast its consequences on the performance of the structure, the target is to identify water uptake as soon as possible.

2.1. *Material choice and production methods*

From the design perspective, the choice of materials (resin and fibers) less sensible to water aggression is fundamental. Polymer resins with low content of polar hygrophilic groups are the best option, since they absorb less content of water. Not only the choice of the polymeric matrix, but also that of the fiber reinforcement must be taken in great consideration: they give high mechanical properties to the composite material but at the same time they are responsible of the interface bonding quality, which depends on the interaction between chemical groups on fiber surface and matrix. The fiber type choice must fall to the ones most resistant to water aggression.

Another important parameter is the fiber volume fraction. P. Davies et al. in [11] showed that the choice of an optimal fiber volume fraction is also fundamental to have a good behaviour of the material in contrasting the moisture aggression, since fibers are generally hydrophobic, so their amount influences the mechanical properties change during ageing. An optimal value is around 55–60 % of the total volume occupied by reinforcement fibers.

S.G. Prolongo et al. in [12] and Y. Hu et al. in [13] studied advanced carbon nanotubes (CNT) fibers. They considered instead of traditional fibers, CNT reinforcements dispersed mechanically into the epoxy matrix. They exhibit high mechanical properties and also play a fundamental role in hindering the penetration of water deeply into the material: in fact, CNT tend to fill the empty spaces inside the composite resin and reduce the free volume (thanks to their size they can occupy the voids between the polymer chains), thus contrasting the penetration of humidity and preventing direct diffusion paths, acting like barriers, reducing the diffusion coefficient, compared to the neat resin case. In addition, they are more hydrophobic than other reinforcement fibers like glass. However, Y. Hu et al. in [13] concluded that the use of carbon nanotubes is not a definitive solution for the humidity problem, since their strong resistance to chemical agents attack weakens during time, because they start acting as preferential paths along fiber–matrix interface, allowing a faster degradation of the interface (debonding, loss of adherence), thus causing a reduction of operative life and reliability of the component.

B. Castanie ert al in [14] and A. Bayatpour in [15] showed how the improvement of curing process for the composite manufacturing (by reducing voids formation generated by the contraction of the resin) is equally useful to prevent or limit moisture ingress. Factory conditions must be controlled in order to avoid any possible absorption of humidity from the environment. To avoid pre-existing defects over the composite surface, an extreme care must be taken during the handling, in the production phase, and also during transportation and installation on site.

A. Krauklis in [5] found-out that slowing down the ageing process is made possible by the insertion in the epoxy resin, during the curing phase of the composite, of particular phenolic anti-oxidant additives capable of picking up and removing the radicals responsible for breaking the polymer chains, thus preventing or at least delaying the thermooxidative yellowing, one of the several irreversible hygrothermal ageing secondary effects which leads to a loss of mechanical properties.

Regarding adhesive joints, M. Davis and J. Tomblin in [16] noted that service bonding failures are often associated to improper bonding techniques due to improper surface preparation and various manufacturing errors. To minimize the deterioration of adhesive bonded joints, it is of paramount importance to choose the better resin for both adhesive and substrate, in terms of compatibility with the substrate. M.D. Banea et al. in [17] showed that the selection of the adhesive resin is linked to a different variation of the glass transition temperature, with different effects on mechanical properties during ageing. For the substrate, polyester resin should be preferred on respect to epoxy, since it enables to reach higher bonding strength, thanks to the higher concentration of chemical groups able to form covalent or hydrogen bonds with the adhesive, so an improved molecular bonding at the interface and stronger interface forces. However, N. Encinas et al. in [18] observed that in the choice of the resin type is not sufficient to obtain high bonding quality, so surface treatments are applied to guarantee better roughness, thus improving mechanical interlocking between adhesive and substrate, and also increase the quality of the chemical bonding at the interface, with consequent higher resistance to moisture absorption.

2.2. *Surface treatments, coatings and nanofillers*

A typical solution to prevent moisture ingress and hygrothermal ageing is the use of protective skin layers and hydrophobic barrier films applied to the surface of the composite laminates [9,4]. As well, surface treatments for fibers are considered one of the most advanced and effective strategies to reduce the moisture content absorbed by the composite structures, with a more visible effect at low temperatures. They enable to obtain better fiber/resin interface adhesion, leading to enhanced mechanical properties and improved resistance to hygrothermal ageing. Then, in a similar way to surface treatments, composite material are frequently treated with special additives, during the manufacturing process, in order to limit moisture absorption, especially at the fiber/matrix interface, where they act as barriers against water penetration, thus guaranteeing a better resistance to moisture absorption and increasing the operational life of the component, with reduced material deterioration, as showed by M. Li in [19].

Nanofillers are used to improve water resistance for both bulk epoxy and adhesive interface bonding, depending on the applications. Starkova et al. in [20] pointed-out that the nano-sized inclusions may influence the properties of environmentally-conditioned nanocomposites in two ways: they reduce the rate and the amount of water uptake acting as a barrier and improve the overall mechanical properties of the plasticized nanocomposites. In addition, they can be embedded in the composite and used as sensors which work in synergy to monitor the material manufacturing process. The incorporation of nano-structured carbon fillers into epoxy matrix may improve their effective properties and also introduce "sensing functionality" by increasing the electric conductive properties of the material. The combination of a nanotube-modified matrix together with conventional fiber-reinforcements provides a new generation of multifunctional materials with health status monitoring capabilities [20].

From the tests conducted by M. Campo et al. in [21], polymer epoxy matrix is known to be hygrophilic: a water contact angle around $63^{\circ} \pm 4^{\circ}$ was measured during tests. Then, thanks to nanofillers presence, the hygrophobicity of the composites reaches values of 70/80°. In fact, when an epoxy composite is manufactured, it can be treated by adding nanofiller substances such as graphene and carbon nanotubes (CNT), which fill the voids and porosities in the epoxy resin acting as a barrier for moisture ingress. They can be used also to create coating layers for composite substrates, especially useful for adhesive joints protection from humidity. By adding nanofillers, the nanomechanical anchoring between composite parts is improved, so a better adherence of coatings over fiber-polymer substrates is achievable [21].

Even though nanofillers treatments allow better hydrophobic behaviour, there is also a downside which manifests for prolonged ageing processes, which is the propensity of the fiber/matrix interface bonding to become weaker over time, because of the nanofiller addition, leading to a faster water accumulation with consequent interface degradation. So, on the one hand, the addition of nanofillers increase the toughness and the stiffness (thanks to the higher effective load transfer granted by a high quality nanoparticles/epoxy interface), while on the other hand, as water is absorbed more and more, the advantages of nanofillers are reduced, negatively impacting on the mechanical properties [21]. The same issues are present not only for the bulk epoxies but also in the case of coatings applied on a glass/epoxy substrate, which show a reduction of adherence after ageing, losing the original advantages. Results from immersion tests on nanodoped composites coatings bonded on a substrate show that before ageing a light increment of the interfacial adhesion is originated by the nanofiller addition, associated to an improved wettability of the treated material on respect to the neat resin. The nanoscaled mechanical adhesion over the substrate rough surface is also enhanced. However, the progression of the diffusion process weakens the interface between the coating and the substrate, directly proportional to water uptake, with a consequent reduction of adherence of composite coating [21].

The interface region between matrix and fiber is particularly subjected to irreversible damages caused by ageing, so it is of paramount importance to focus the research on new methods to avoid the degradation of the interface. In order to improve the bonding quality at the interface, treatments to coat fibers are considered, since they allow to enhance the adhesion between the fibers and the epoxy resin. Y. Zhang and C. Mi in [22] developed a technique to enhance the bonding strength. It consists in a very thin layer of silica nanoparticles at different concentrations disposed over glass fibers by evaporative deposition. After the treatment, the surface roughness of glass fibers appears improved, as also highlighted by scanning electron microscopy (SEM). Tensile and bending tests were also performed, comparing advanced composite with the untreated one; in all cases, the coating leads to an improvement of residual strength and stiffness [22]. Tests on different silica concentration levels were realized. Then, SEM images were also considered by B. Yang et al. in [23] for visible comparison of fractured specimens after mechanical testing, by examining the surface morphology before and after coating[23,22]. Water absorption tests were made for different temperature levels. Results assess, for every case, a significant reduction of water diffusivity and saturation water content thanks to silica coating, thus enabling to slow down the hygrothermal ageing process. However, by increasing temperature, the advantages in terms of diffusion are limited. At the beginning of the ageing, the diffusion seems to follow Fickian behaviour with a water content increasing proportionally to the square root of the immersion time. However, for longer immersion times, temperature is responsible of important variations, with a non-Fickian behaviour. This effect is associated to a coupling between silica coating and temperature, and is explained by Y. Zhang and C. Mi in [22] through the so-called water-channel diffusion (Fig. 1).

When a composite is manufactured, it is inevitable that air bubbles remain trapped into the material. In addition, the curing process release a large amount of heat which further induces various defects such as cracks, voids and pre-existing small damages, which become possible paths of diffusion during the hygrothermal ageing process. Then, due to the different mechanical properties of the fibers and the resin, the interface region promotes defects formation, also during manufacturing phase [22]. During hygrothermal ageing, water molecules penetrate along the fiber/matrix interface by capillarity and they tend to accumulate in those voids and micro-cracks present in that region. Water uptake continues until all voids are filled. Then, low temperature slows down the decomposition rate of the epoxy matrix, resulting in the preservation of the equilibrium water content. Even if water still diffuses, the net water content stays constant. High temperature is instead responsible of relevant resin decomposition rates that cannot be neglected. Then, during the removal of resin and its movement along the fiber/matrix interface, new voids are formed, where more water can accumulate, also considering the fact that the water density is much lower than that of resin. In addition, the resin decomposition at high temperatures is also enhanced by thermal stresses. For this reason, long immersions in hot water strongly impact on the weight of composites, which sensibly reduces [22].

The addition of the silica filler brings two favorable effects on the water diffusion along the fiber/resin interface. In the first place, micro-cracks and voids originated by the production process are filled by the addition of nanoparticles of such type. Without the coating, these act as preferential sites where water is stored. Then, water diffusion paths become longer because of the obstacle created by silica nanoparticles, which force water molecules to deviate and follow indirect paths around the nanoparticles. The net effect of silica coatings is to reduce both the maximum amount of water that the composite can absorb and the water diffusion rate, highlighting their positive contribution as a preventing solution from hygrothermal ageing [22].

Many other studies evidence the advantages given by coatings and treatments to improve ageing performance of composite material, like N. Li et al. in [24], who assessed the interfacial bonding quality during hygrothermal ageing, after a treatment based on the application of a silane layer coating. Another study by F. Yu [25] confirmed the relevant role that coatings have to protect the composite surface from hygrothermal ageing: a combination of polyacrylate polyurethane coating was tested with convincing results, showing good mechanical, adhesion, hydrophobic and antifouling properties. In addition, these coatings are further improved by the addition of nanoparticles like silica, which, if correctly treated, deposit on reinforcement fibers surface leading to a better rugosity. The contact angle of water molecules which touch the new surface is increased up to 116°.

On regard to **surface treatments and coatings**, a special mention has to be done for wind turbine blades, which need to respect strict operational requirements, in terms of efficiency, cost and reliability. Nonetheless, moisture absorption and its consequences can put a serious

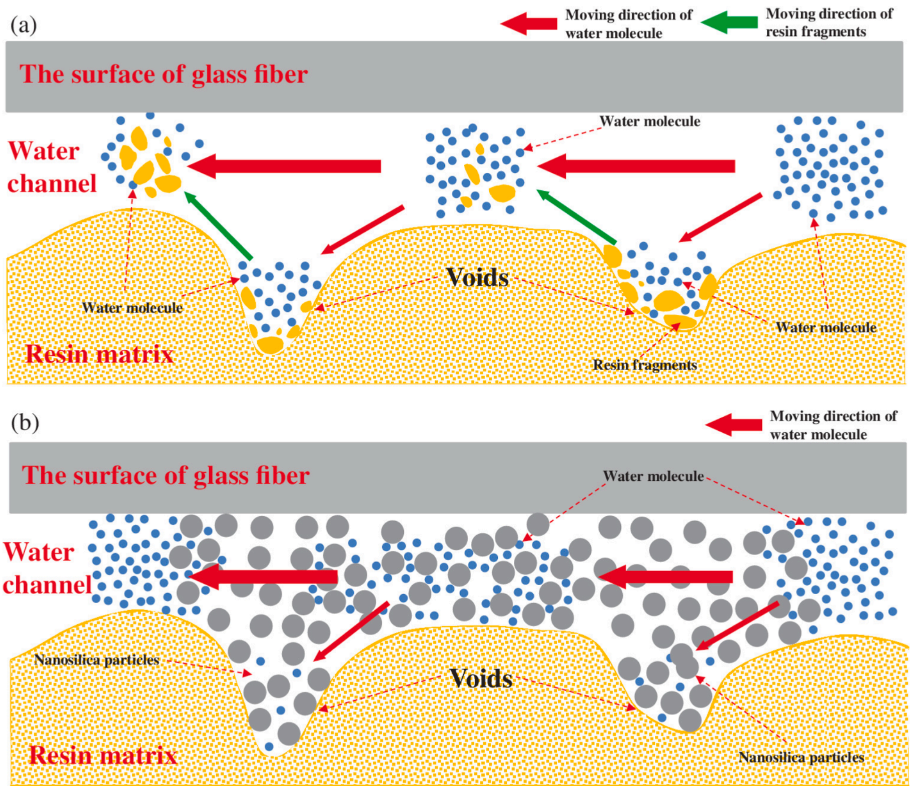

Fig. 1. Schematic illustration of the channel diffusion of water molecules along the fiber/matrix interface in polymer composites embedded with (a) not coated and (b) silica-coated glass fibers. .
Adapted from [22]

threat on the performance of the structure, so effective solutions in limiting the problem are essential, like for example the application of protective films and coatings on the blade surface. For turbine blades, application of anti-corrosive and protective sealing strips (e.g. polyurethane elastomers, resistant to abrasion) or multi-layer coatings like that in Ni-Co alloys with addition of reinforcing powders. Elastomers contain both rigid and soft portions of material, giving the material stiffness and ductility at the same time. Mechanical tests were done by H.M. Su and T.Y. Kam in [26] to assess the effectiveness of blade coatings: the results show that coatings limit the reduction of the strength caused by ageing, with strong improvements for high protective film thickness (Fig. 2). On the other hand, the solution is only partial, since it is not able to prevent water absorption but only limit the diffusion and its effectiveness strongly decades for long exposures to humid environments and high temperatures. Thus, alternative and more effective solutions need to be found to better counteract the humidity damages.

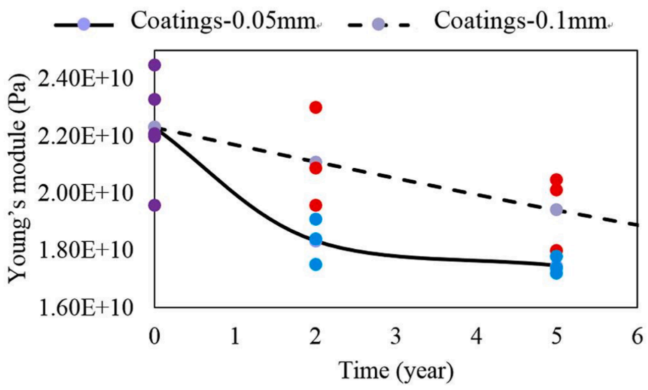

Fig. 2. Effect of coating thickness on Young modulus during ageing. .
Adapted from [26]

Primers are used as a barrier layer to improve interface resistance to water absorption and so the joint performance. Since the adhesive interface plays a crucial role when an adhesive joint is subjected to moisture absorption, L. Jian et al. in [27], J.P. Sargent in [28] and A.J. kinloch et al. in [29] evinced that an adequate surface preparation of adhesives with pre-treatments and primers at the interface is extremely important in order to contribute to the bonding durability, avoiding debonding due to corrosion and limiting ageing deterioration. In adhesive joints, the improvement of adhesion is detected by looking at the surface energy and wetting properties, which increase after the application of surface treatments and coatings. The surface roughness at the interface plays also a key role: a too low roughness (polished surface) enhances the decohesion process, since it is not capable of creating a mechanical interlocking at the interface between the adhesive and the substrate, while an excessive roughness may lead to an excessive water accumulation at the interface, which increase the diffusion at adhesive/substrate interface through preferential paths (Fig. 3). C.S.P. Borges et al. in [30] stated that if the surface is too polished, the transition from cohesive to adhesive failure is enhanced. The studies of M. Michaloudaki et al. in [31], M.P. Falaschetti et al. in [32], M.D. Banea and L.F.M. da Silva in [33] and H.R. Gualberto et al. in [34] verified that an accurate roughness level enables to increases the path followed by water molecules along the interface thus causing a reduction of diffusion, with low decrease of strength after ageing. The modification of the surface roughness can be achieved with treatments like sandblasting, through mechanical action [27].

Other frequently applied treatments are: peel-ply, chemical immersion, plasma exposure, introduction of nanoparticles embedded in the adhesive composite surface. These treatments play a central role to improve the adhesive joint durability, but have also drawbacks, especially the risk to damage the interface by creating micro-voids and microcracks, as reported by S. Budhe et al. in [35]. **Peel-ply** is the most

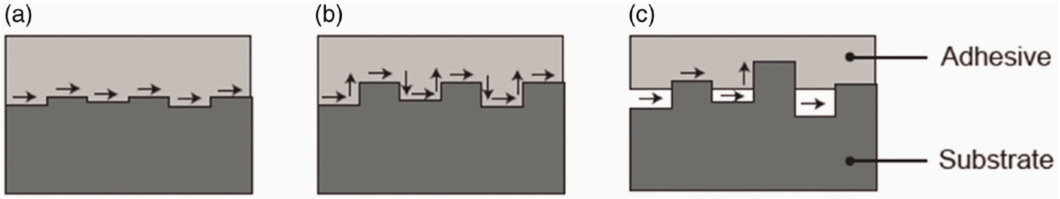

Fig. 3. Different roughness is achievable by surface treatments. . Adapted from [27]

common surface treatment used to protect the adhesive/substrate interface from moisture penetration, increasing the adhesive joint durability. It is a layer of woven fabric, glass or nylon applied on the surface of the composite and then peeled off before the bonding process. During the curing phase, this layer absorbs part of the resin and becomes an integral part of the laminate. Then, before the bonding phase, this layer is detached to obtain a clean and well roughened surface at the interface. Thanks to this, it's possible to obtain an high quality bonding, by increasing the bonding properties of the joint thanks to chemical agents which better react with the polymer molecular chain during bonding. It also prevents contamination from external environment in the storage time preceding the bonding, thus limiting the so-called pre-bond moisture contribute, by protecting the surface of the composite. The main disadvantage of the peel-ply method is the presence of residues on the composite surface, which removal prior the bonding is challenging and possibly leads to internal defects inside the joint [35].

Another possible treatment is based on an **atmospheric pressure plasma torch** device (APPT): the setup is equipped with a rotating torch and a nozzle which allows the expulsion of air plasma gas, at a controlled pressure of 2 bar. A platform carrying the specimen is positioned at a few millimeters of distance. When the component is impacted by the ion plasma flux, a surface more susceptible to generate adhesion is obtained. After the process mechanical pull-off tests are performed by N. Encinas et al. in [18]: results show that the wettability properties are improved, giving a stronger adhesion. The surface energy is in fact higher, given by the formation of chemical groups which boost the interface bonding. The treated surface manifests 34–48 % higher tensile stress, so more adhesive joint strength [18]. The combination and synergic contribution of mechanical interlocking, given by roughness level, and the chemical adhesion, given by chemical additives, enables to obtain a bonding with an adequate joint strength to counteract the moisture absorption and its consequences on the adhesive (tests show an increase of bonding strength up to 60 %) [18,35,19]. This surface treatment also improves the shear capacity of the adhesive joint and is cost-effective. H.R. Gualberto et al. in [34] observed that the application of combined surface treatments gives the adhesives also an high resistance to fatigue.

As far as **natural composites** are concerned, new developments are still required to find affordable and efficient solutions to ageing issue. S. Kalia et al. in [36] observed that interfacial adhesion and resistance to humidity can lead to serious threats for the safe use of these materials. To reduce the propensity of water absorption and improve composite durability, surface treatments can be applied, using chemical compounds like alkali and silane, or through graft co-polymerization or plasma effect, which chemically alters the surface of the natural fibers, acting as a barrier against moisture absorption and improve surface roughness, removing impurities that hinder the creation of good interfacial adhesion [37,36,38,39]. Hygrothermal treatments allow to modify the chemical structure of the cellulose, increasing its crystallinity. It consists of extracting a part of hemi-cellulose, the main responsible of moisture absorption in natural fibers, thus reducing the propensity to absorb water [36]. Mechanical tests performed after treatments indicate a reduced moisture gain and an improved tensile strength. The higher resistance to moisture absorption is achieved thanks to the treatment-induced reduction of the hydroxyl groups on the fiber surface and removal of some other substances present on the fiber surface which can promote water absorption. Treated natural fibers becomes more hydrophobic. G. Ma et al. in [37] and M. Curto et al. in [39] underlined that these treatments also induce a better chemical bonding between the fibers and the matrix, thus strengthening the interface region and reducing the voids and pores which could facilitate moisture penetration inside interface.

V. Prasad et al. in [40] studied the combined effect of nano TiO_2 and silane coupling agent as an innovative coating for flax fibers. The treatment performed on natural fibers enhanced the chemical compatibility with resin, creating several new bonds between additives and chemical groups of the cellulose fibers, thus hindering the attack of the fibers from water molecules, as shown in Fig. 4.

Without the coating, water would easily penetrate at the interface and interact with hydroxyl groups on the surface of hydrophilic natural fibers, disrupting the adhesion. Nano TiO_2 are considered extremely performing against hygrothermalageing, because of their excellent hydrophobic nature, insolubility in water and resistance to swelling. The authors obtained encouraging results: a reduction up to 42 % of water diffusivity in the bio-composite and an increase of mechanical properties around 20 %. The interface region of composite is critical, to which particular attention must be paid when studying the phenomena, in order to propose suitable solutions to effectively counteract the problem. Other main variables influencing the absorption process are internal voids amount and chemical composition of the fibers [38].

Other studies proposed solutions to improve hygrothermal ageing resistance of sisal/epoxy bio-composites: sodium bicarbonate treatment and poly-lactic acid (PLA) coating were considered by P. Sahu et al. [38]. The results, obtained after several mechanical tests, showed that treated and coated specimens perform better in terms of resistance in humid environment: moisture content reduced by 30 %, mechanical properties increased from 10 % to 30 %. Since some treatments (alkali, silane) are found to be dangerous and unsafe, new eco-friendly treatments (by $NaHCO_3$) and coating (with PLA) are being developed. SEM images confirm that fiber/matrix debonding and swelling are present before the operation, while the treatment allows to achieve an increase in interface adhesion. The authors conclude that treated sisal fibers bio-composites could be suggested also for high-performance applications, including aerospace [38].

3. Moisture detection and removal: Maintenance techniques

3.1. Non-distructive tecniques: NDT

While surface treatments are applied during production phase, other solutions to hygrothermal ageing are put in place during the operative life of the composite components. In fact, during regular maintenance checks, the structures can be tested and analyzed with the help of non-destructive techniques (NDT) in order to detect the presence of moisture trapped inside the composite and possible damages occurred. Then, the following step consist in taking corrective actions to remove the humidity absorbed during the operations. C. Li et al. in [41] and M.J. Morris in [42] emphasise the importance of using non-invasive

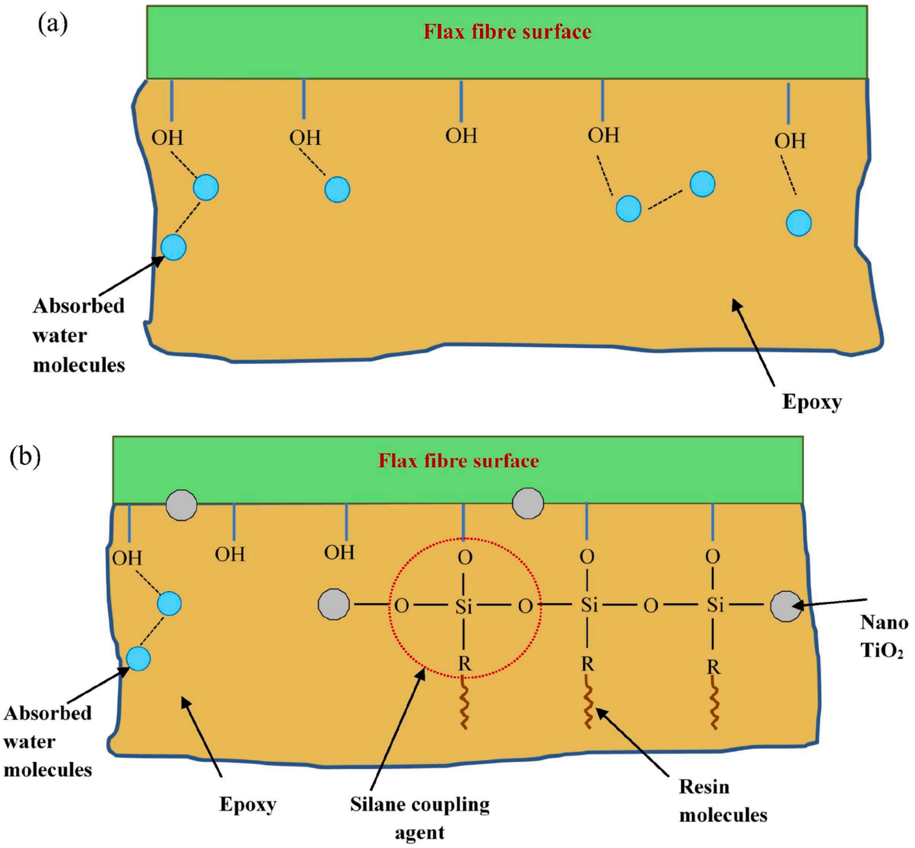

Fig. 4. Chemical interactions at the flax fiber/matrix interface before and after nano Tio_2 and silica coating. . Adapted from [40]

techniques during removal operations.

In detail, the maintenance of an aircraft must be organized and defined in accordance with the tight schedule of the flights. A rigorous plan is required in order to avoid economic drawbacks and each non-scheduled stop in case of the need of repair force the airline to re-program the timetables, generating both immediate and ripple effects. Then, in that situation, as explained by M.J. Morris in [42], a not expected repair leads also to limited corrective actions: if the hangar is not ready for the accommodation, an on-field repair is the only possible solution, which means that advanced techniques (like those for the removal of moisture, requiring high temperatures) cannot be used. For this reason, high standard maintenance planning is nowadays crucial to face unforeseen events. Predictive maintenance is the most optimized system to prevent these kind of issues, allowing to know when a component requires a check depending on its health status, continuously monitored. After water is detected by maintenance workers, it should be removed by using special techniques. However, some drawbacks have to be considered: the operation must be done by highly trained and skilled technicians (so it is also a matter of costs) and there is a strong risk, by directly taking away the water present inside the material, of creating new damages (also from the structural integrity point of view) and new voids [42].

S. Whitehead et al. in [43] investigated the removal of moisture in **sandwich structures**, where humidity accumulate inside the honeycomb core. The operation consists of creating a high void content in the bondline, which could cause a degradation of adhesive bonds or even blow-off (detachment) of the skins due to sudden increase of vapor pressure in the honeycomb cells. These critical issues can be partially overcome by minimizing the invasivity of the procedure. The timeof exposure tohumid environment isa discriminatingfactorforthe successof themoistureremoval: only ifthe water accumulated is fully and quickly removed, a recovery in the mechanical properties and of the original weight of the material can be achieved, while if the ageing has gone too far, the moisture trapped has reached deeper areas into the composite, up to the fiber/matrix interface, so a full recovery becomes quite difficult, compromising the performance of the component indefinitely. A partial or late removal means that some water is still present inside the structure. If this water remains trapped for long times, the hygrothermal ageing goes on, leading to the breakage of chemical bonds, with consequential delaminations and matrix-fibers interface debonding, which cannot be recovered anymore. So fast detection and precise techniques are needed to avoid any irreversible performance loss of the component [41].

An alternative solution is to drill holes on the panel surface (or honeycomb cell walls in the case of sandwich structures) to extract water accumulated, but with the risk of creating new internal voids and promoting new patterns for humidity penetration, which can eventually be closed with the usage of fillers. Another solution exploied by B. Geyer in [44] is to remove water through heat drying or use of a bonded repair patch over the drilled area. However, as showed by S. Budhe et al. in [45] and S. Sfarra et al. in [46] there are many drawbacks associated to these procedures. First, the complexity and risk of new damage formation. Then, structural weight may increase if fillers are used to fill the drain holes, which may affect aerodynamic performance of the aircraft. In addition, moisture ingress is not prevented and there is the potential of new water ingress paths originating from the drilled holes. Further bond repairs and replacements may be needed to ensure structural integrity [41]. Finally, some repair techniques work at high temperatures, which can lead, as showed by J.E. Shafizadeh et al. in [47], to the trapped water evaporation with a consequent increase in pressure, which can cause severe damages to the sandwich structure. Since water removal results quite difficult to be achieved, the efforts must be oriented in the identification of humidity inside composite structures, which, as previously said, can be done during maintenance using various non-destructive techniques, such as: gravimetric analysis, spectroscopy, ultrasonic techniques, guided waves, acoustic microscopy, thermography [48].

A typical non-destructive method to evaluate moisture uptake in **adhesive bonded joints** has been studied by M.D. Weir et al in [49], it consists of the ultraviolet (UV) reflection spectroscopy, through which it is possible to follow the spectral changes during water absorption and so estimate the fraction of hydrogen bonds formed between water and adhesive molecules. This method shows a good sensitivity to detect the presence of water in the adhesive [49]. The possibility to monitor in-situ the water absorption in a polymer by coupling this method with a fiber Bragg grating sensor was also considered by N. Sung et al. in [50].

Alternatively, M.K. Antoon et al. in [51] showed that by using the Fourier transform infrared spectroscopy (FTIR) it is possible to characterize the interaction of an epoxy matrix with water molecules and observe chemical changes in the adhesive polymer after water absorption, and also compare the response to moisture before and after fiber surface treatments. The absorption bands of the epoxy resin shift with absorbed moisture, since water molecules modify the electronic environment. Each variation of peaks and band shifts in the spectrum is associated with a certain alteration of the chemical bonds following the treatment and water aggression. The spectrum can be also affected by polymer swelling, which is responsible of morphological changes or bond distortion, thus influencing the vibrational spectrum [51,38].

Another non-destructive method to detect the presence of irreversible effects of humidity absorption is the size exclusion chromatography (SEC), a technique able to determine the molecular weight and distribution in the polymer, before and after the process. If the molecular weight is kept on an approximately constant value, no breakage of polymeric chains (hydrolysis) has occurred, therefore there are no irreversibilities. Short-term polymer test do not show any irreversible effect on the composite material.

For **wind turbine** blades, regular inspections of blades with non-destructive techniques (NDT) should be made, such as ([10]):

- visual tests: with the support of drones and remotely controlled aircrafts (UAV) capable of reaching blade height and inspecting them, identifying cracks up to 2 cm wide from a distance of 200 m and collecting images for analysis;
- ultrasound techniques: able to find delaminations, adhesion defects, areas with reduced resin content, even with information on the depth of the damage;
- infrared thermography: the damage is visualized through temperature patterns on the surface (hot spots, indicate damage or lack of adhesion between parts);
- radiography: evaluates different level of proton absorption through the material, with X-rays (gives information on density variations and therefore on the alteration of material properties as a result of damage);
- acoustic emission (AE): technique based on the propagation of elastic waves through the material. Acoustic events are passively emitted by the material, without the need of an external source; frequency and amplitude signals indicate the presence of various types of damage such as cracks, discontinuities, delaminations. The advantage of this technique is that it enables to separately identify different effects, depending on the waveform and its properties (amplitude and frequency). The waves are detected through piezoelectric sensors installed on the blade surface. Acoustic emission can be implemented also as a structural health monitoring system, a topic which will be treated later.

The major drawback of NDT techniques, according to R. Sarafaraz in [48] and M.J. Davis and A. McGregor in [52], is the difficulty of locating the origin of a damage and the late detection of defects caused by hygrothermal ageing, when the structural integrity is already compromised and the failure is already occurred. In addition to that, the conventional NDT methods cannot easily detect the moisture presence [48]. So, better solutions to monitor in an active way the hygrothermal ageing effects on the structure are needed, such as moisture detection methodologies based on the structural health monitoring system.

3.2. Acoustic emission and Lamb wave

Acoustic emission (AE) is a non-destructive technique implemented as a monitoring system, based on the detection of elastic waves in structural components generated by damages, such as the initiation and propagation of cracks. It does not require invasive actions to verify any possible damage and is a passive NDT method since no external energy should be supplied to testing structure, so it can work autonomously. M. Assarar et al. in [53] demonstrated that different damage mechanisms caused by hygrothermal ageing like delaminations, debonding, crack propagation can be identified thanks to this methodology, depending on the type of signal emitted [53]. In fact, the material is induced to emit elastic waves after a damage (discontinuity) occurs, associated to the release of an amount of deformation energy. Then these waves measured are collected by a piezoelectric sensor. The signal contains information about the damage and is compared with that obtained during ground calibration phase. More in detail, parameters like waveform and amplitude range are monitored, from which some considerations can be derived: medium–low amplitudes are typical for micro-cracks and resin deformation, while high amplitudes are identified when fiber breakage occurs. For short immersion times the absorption of moisture involves only polymeric matrix, so only the signal corresponding to neat resin is affected, while for longer exposures an increase in frequency of other signals is observed, indicating that the deterioration of the fibers and the interface becomes significant after the composite has reached saturation [53]. This system can be implemented through a possible an on-board computer unit to provide real-time data relevant to eventual critical issues, allowing fast corrective actions. The following step should be the development of monitoring techniques able not only to find damages when they occur but also predict them, by looking at the first effects of ageing process, which do not manifest macroscopically and for this reason are more challenging to detect. However, like has been stated by C.R. Ferrar and K. Worden in [54] in practice, the application of AE system may be laborious, time consuming and expensive, so more efficient solutions are required.

R. Gorgin et al. in [55] exploited a detection techniques through lamb waves. It is another methodology which enables to monitor the effects of humidity on composite structure and the mechanisms of damage during hygrothermal ageing, by means of a transducer that emits and receives a signal and the following analysis of data coming from ultrasound waves detection. Sensors can be integrated into structural parts or applied on their surface and used to gather information about changes within structures. This technique is useful to detect possible cracks developing in the structure due to ageing and to monitor how mechanical properties change during the ageing process. Parameters such as the wave numbers, the attenuation and the wave propagation velocity are a good damage indicator, because they change depending on the humidity content. In detail, has been shown, by R. Gorgin et al. in [55], M. Castaings and B. Hosten in [56] and J. Lee and Y. Cho in [57], how the phase velocity of the waves, which is directly related to the strength modulus, is monitored to evaluate how mechanical properties are impacted by ageing. Lamb waves propagate through the structure in different modes. To extract information about the damage mechanism it is necessary to separate these modes during data acquisition and/or following data analysis. M. Cinquin et al. in [58] set experimental apparatus up based on Lamb waves detection system: during ageing/drying tests, signals were monitored and an increase of the attenuation mode was detected (the only mode sensible to moisture variation), followed by a corresponding reduction during the drying phase, showing, in that case, the reversibility of the ageing process, with full weight recovery [56]. Still, mode separation becomes a major problem in real applications, due to multiple wave propagation effects such as reflections and other physical effects like dispersion attenuation, mode conversions and constructive/destructive interferences, which affect the interpretation of signals data. For this reason, it is difficult to identify the cause of every change in the signal during a given time trace and to detect and interpret small scatters due to changes in the complex Lamb wave signals. False damage identification and characterization is one of the main drawbacks of this technique. The sensitivity of these systems to environmental and operational conditions (temperature, vibration, load, moisture, bond defects, adhesive layer shear modulus and thickness) is still a big challenge and their real-world application is nowadays quite limited [55]. Further developments and studies are needed to optimize Lamb waves as a completely autonomous online monitoring technique of moisture level during flights.

3.3. *QD: Quantum dots*

Y Fang et al. [59] developed an improved, quick, simple and innovative system able to detect degradation in glass FRP composites used in marine applications, by using quantum dots (QD), which are special particles chemically bonded to glass fibers, at the fiber/matrix interface, acting as damage-sensitive links between glass fibers and polyester matrix (Fig. 5). When a separation of the resin from the glass fibers occurs, the loss of adhesion at the interface impacts directly on quantum dots, leading to a loss of fluorescence. Moreover, their positioning at the interface enables to evaluate the behaviour of the most critical region of the composite affected by hygrothermal ageing.

Quantum dots allow to evaluate microscale damages by looking at fluorescence microscopy images: on aged composite the fluorescence pattern is modified as a sign of damage presence at the interface. Fractures position is identified where fluorescence disappears. By increasing the immersion time, the area characterized by loss of fluorescence becomes larger and larger, indicating a widespread and severe deterioration of the bonding [59]. The main drawback of QDs depends on the difficult way to measure the changes of luminescence emitted by the particles: to achieve this goal, it is necessary to peel off the paint from composite surface and performing a laboratory analysis. The invasivity of this approach results too high to be acceptable. For this reason, limitations to the usage of this technique are still a major concern. This method may be implemented as a SHM system by embedding sensors able to detect luminosity changes and connected to a computer for data acquisition, storage, and analysis, in order to monitor the ageing process and the health of the structure in real-time: the moisture uptake can be followed step by step by analyzing the variation of these luminosity parameters.

3.4. *Optical fiber*

Nowadays, the more advanced methodologies for hygrothermal ageing damage detection and monitoring are based on optical fiber sensors, such as Fiber Bragg grating (FBG) sensors, as showed by C.S. Shin and T.C. Lin in [60] and M. Mieloszyk and W. Ostachowicz in [61], C.M. Bellot et al. in [62] and M. Girard et al. in [63]. This technology shows a clear improvement with respect to other non-destructive techniques, which cannot detect the material degradation in advance. Monitoring systems based on fiber optics have a predictive capability, since they can foresee how the structure will behave in response to moisture absorption, and not only identify a damage already present. In FBG system, the measure of the displacement is done by looking at peak strain shifts of the frequency spectrum, which are sensible to external environment; then, the wavelength measure is converted to a strain measure [60]. Fiber Bragg grating (FBG) sensors have several advantages like resistance to corrosion, have small dimensions, low weight and ability to register small changes of water content inside the material [61]. Furthermore, this method has the advantage to use sensors which have been largely studied, designed for various applications and reliable. In addition, this solution is cost-effective since it does not require to stop the operations of the component where the sensors are embedded; during operations, such as a cruise flight, the structure is constantly and actively monitored to know the health status of the system.

Fiber optic sensors, due to their minimal invasivity, are suited for embedment especially in adhesive bonded joints, which are realized by connecting an adhesive with a substrate; at their interface, ahead the bonding process, these sensors can be positioned to real-time monitor the health status specifically at the interface level, which is the most critical region subjected to hygrothermal ageing deterioration. Placing the sensors at the adhesive interface, precise data about strain and bonding resistance can be obtained, depending on moisture content [60,61]. However, deformation occurs when ageing process is already started and possibly also leading to visible damages of the material. In some cases, especially for applications like aerospace and marine sector, where safety is crucial, the prediction of the structure response to ageing is even more important. So the last developments suggest the possibility to use fiber optic sensors to monitor properties and physical quantities of the composite material which are directly dependent on moisture absorption, such as glass transition temperature and water uptake.

A. E. Krauklis et al. in [64] show a technique exploiting optical fibers to monitor the glass transition temperature. The ageing progression can be assessed in this way: if glass transition temperature is completely recovered by a drying procedure, the material mechanical properties are re-established and the material turns back to its original state after plasticization, without chemical bond breakage (no changes are detected in the polymer structure), while if it remains lower than the initial value, some kind of irreversibility has occurred, with alterations of the

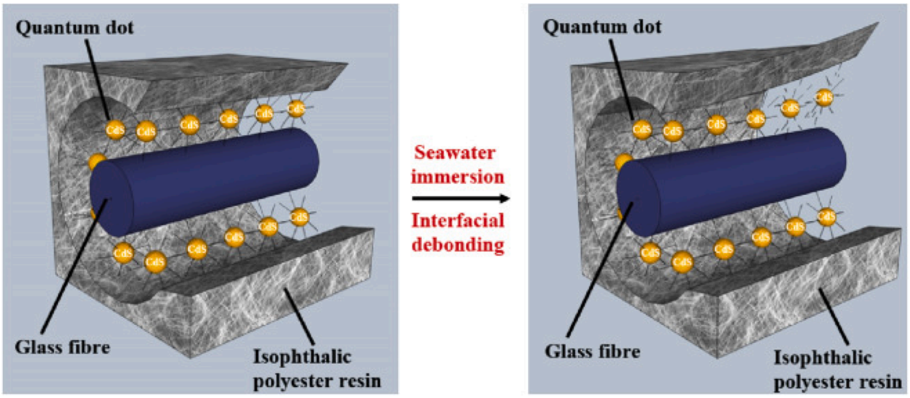

Fig. 5. Schematic detection procedure of fiber/matrix debonding in GFRP composites under seawater immersion environment by quantum dots (QD). .
Adapted from [59]

polymer structure and chemical degradation, leading to chains scission (hydrolysis). Tracking the changes of glass transition temperature becomes one of the most powerful monitoring strategies, which allows to detect humidity absorption in composite materials, from the very beginning, so preventing its consequences on the structure. This approach could be implemented in a on board system for real-time surveillance of component health status, in order to keep the performance of the structure and avoiding any possible issue deriving from the hygrothermal ageing advancement.

Recent developments in the field of structural health monitoring (SHM) systems are oriented toward the set-up of advanced methodologies for hygrothermal ageing detection at the early phases of the process, i.e. when moisture starts to be absorbed by the composite material. Several studies assessed the possibility to embed fiber optic sensors into composites for moisture level content monitoring through optical properties variation. These are direct methods for SHM monitoring of ageing since they look directly to physical quantities associated to moisture absorption, so they do not rely anymore on variables which define the effects of ageing, thus enabling a faster detection of humidity presence inside the structures and an optimization of the monitoring capability, with high standards of safety for the structure itself. Fiber optic sensors for SHM show high versatility and low costs besides signal data can be easily monitored even on board without damaging the structure, with the possibility of doing self-diagnosis [62].

M. Girard et al. [63] focused on the water diffusion characterization in an adhesive bonded joint interphase by a fiber optic sensor based on Fresnel reflection. They proposed a methodology that can be implemented for real-time monitoring of the ageing process for a specific marine application (tidal turbine blades). The problem considered by the authors is definitively the most critical in terms of ageing degradation of composite structures: in fact, it combines the use of adhesive joints, which are, as previously explained, one of the most susceptible composite structures degraded by moisture absorption, together with the most hazardous operating condition, which is the marine environment, where water strongly influences the structural behaviour and durability over time, more than in any other external environments. Consequently, a reliable solution to the problem must be found to guarantee the safe operation and high performances during the whole life-time. To reach this goal, they exploited a relation between the water content uptake in an adhesive joint and the change of an optical property, the refractive index n, given by equation (1), the so called Maxwell-Garnett equation:

$$c(z,t) = V_w \left(\frac{n_{a/w}^2(t) - n_a^2}{n_w^2 - n_a^2} \right) \left(\frac{n_w^2 + 2n_a^2}{n_{a/w}^2(t) + 2n_a^2} \right) \quad (1)$$

where $c(z,t)$ is the local water content in the adhesive, V_w is the water volume fraction, $n_{a/w}$(t) is the adhesive refractive index during immersion, n_a adhesive refractive index before immersion and n_w is the water refractive index.

The refractive index is a measure of the amount of light reflected by a certain material and it depends on the fiber optic sensor positioning inside the adhesive and moisture concentration: the minimum refractive index is obtained at the interface, where the water content is higher due to stronger diffusion, while going far from the interface it decreases. Then, the refractive index slowly decreases during water diffusion; the chemical network (molecular structure) and its interaction with water molecules absorbed is responsible of the refractive index variation [63]. Thus, following the trend of this optical property, the moisture uptake can be traced back, to actively and continuously know how the hygrothermal ageing is progressing in the structure and so evaluate if a possible damage is imminent or not, predicting the behaviour of the component. In fact, if the moisture level is low, in the first phase of moisture absorption, plasticization occurs and the loss of mechanical properties is in general reversible, while for high moisture levels and long exposures to humid environments, irreversible damages cannot be avoided. Every time a variation, the refractive index is detected by the system, the operator knows that the humidity has entered in the material and the plasticization process has started or is going to start, giving a reference on the progression of the ageing itself and allowing the earlystage detection of ageing effects. An illustrative output of the local water content obtained by applying the MaxwellGarnett equation is shown in Fig. 6, in a good agreement with results from advanced numerical simulations, such as the Dual-Fick local model.

The results obtained from the simulation through the Maxwell-Garnett model are then compared to those coming from a gravimetric tests and a good correlation can be detected (Fig. 7), meaning that the model is able to describe quite precisely the behavior of the adhesive during ageing.

In addition, the study shows how the interface accelerates water diffusion in an adhesive joint, justifying the importance to find counter acting measures to limit the degradation of the adhesive bonding. The gradient model enables to characterize in detail the diffusive properties of the interface, understanding how moisture diffusion proceeds in time. This model should be implemented on-board to monitor the structure during operations, by embedding, during the production phase, fiber optic sensors inside the composite structure. Moreover, building a tracking instrumentation able to monitor in real-time the optical properties changes and a computational unit which exploits the Maxwell-Garnett relation, is possible to implement an algorithm able to directly transform the signal information (refractive index). The output value shows the moisture content inside the adhesive interface, as function of the thickness and the ageing time.

4. Conclusions

Different possible solutions to the humidity problem in composites, highlighting advantages and drawbacks. The necessity to find proper and efficient methodologies to solve the problem of humidity in composite structures is made clear by implications on economy, safety and sustainability. High attention must be paid to develop preventive and protective solutions, with a particular focus on marine applications, where the effects of hygrothermal ageing are extreme due to the presence of liquid water instead of humid air and cyclic loads.

Surface treatments, coatings, nanofiller additives, detection and removal of moisture through non-destructive techniques during maintenance and structural health monitoring (SHM) systems were then examined in detail, showing how recent developments in the field of nanotechnology hint at possible further improvements in finding new and efficient solutions based on nanofillers to face the humidity problem in composites.

The diffusion of sea water and moisture through the composite thickness is still considered a challenging technological issue that should be tackled at its early stage, in order to prevent long-term damage and expensive replacements and repairs. Since non-destructive techniques show several functional, practical and economical limits, the best way to achieve early stage detection of water diffusion is through real-time health monitoring systems able to operate remotely, with high safety standards, minimal invasivity and low costs. Above all, fiber optic sensors technology for SHM systems should be considered for possible innovative solutions, thanks to an already extensive technical knowledge, high performances, low invasivity, low weight and widespread in sectors like telecommunications and on-board wiring applications for aeronautics. Predictive maintenance plan can be drawn up on the basis of data got by the monitoring system in an adaptive way, depending on the real health state of the component. This also means that a component is replaced only when is really degraded, not needing of further maintenance investigation, which requires a downtime leading to economic losses. The advantages of this solution may be maximized through the installation of a control units able to predict the structure behaviour in real-time, to anticipate its response to the ageing process. Further developments are still required to assess the feasibility of structural health

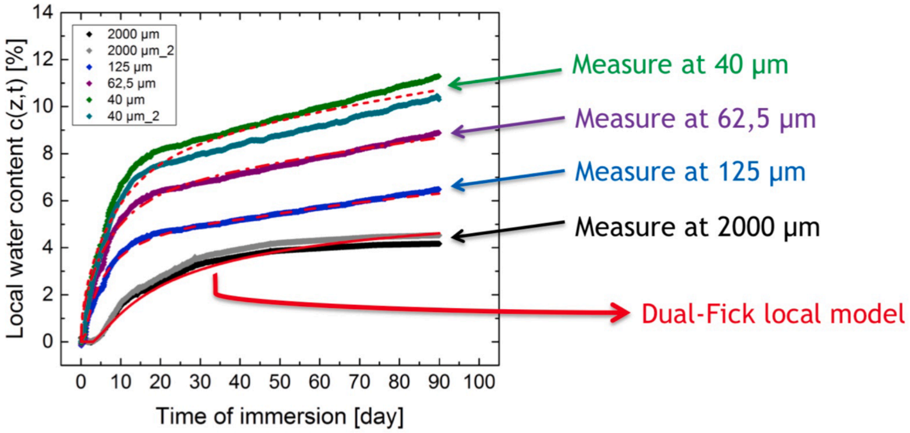

Fig. 6. Comparing results of water uptake: Maxwell-Garnett equation and numerical simulations. .
Adapted from [63]

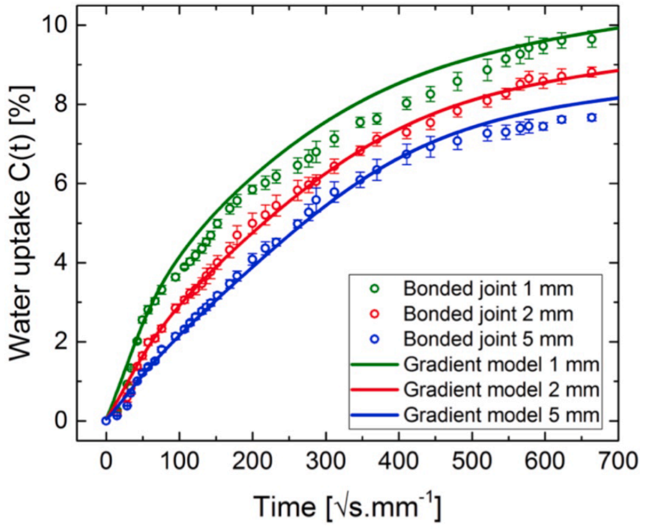

Fig. 7. Comparison between Maxwell-Garnett model and gravimetric test results. .
Adapted from [63]

monitoring implementation as on-board systems for aircrafts, wind and tidal turbines.

Lastly, the information extracted from real-time health monitoring could also help designers to find out more durable and improved design solutions of components and structures, since the manufacturing processes could be better understood thanks to the data obtained from in-situ devices. Smart manufacturing and smart structures are the future of composite materials in industry, and a lot is still to be discovered on the subject.

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.

Data availability

No data was used for the research described in the article.

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