A Review of the Effects of Fluid Absorption/Desorption on the Residual Strength of Carbon Fibre Reinforced Composite Materials

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I. Abstract

This paper provides an outline of research into the investigation of the effect that absorption and desorption cycle of saltwater has on the mechanical properties of Carbon (Graphite) Fibre Reinforced Plastics (CFRP) and current state of the art in fluid cycling effects on composite materials. The use of CFRP is ever increasing in the aerospace industry however; from a survey of the current literature the topic of salt water cycling effects has had little investigation. This topic of saltwater cycling effects on CFRP is now of current interest as more composite airframes are deployed at sea. The principle focus of this paper is a literature review on the mechanisms of saltwater absorption/desorption and its effects on CFRP. Future research by the authors will aim to determine whether absorption and desorption cycles of saltwater does leave trace elements, and whether the presence of these trace elements affect the mechanical properties of the CFRP.

Another objective of this research is to add to the body of knowledge held by the Australian Defence Force (ADF) for the ageing of platforms partly-constructed from CFRP, namely the MRH-90, the Tiger Attack Helicopter and eventually the F-35 Joint Strike Fighter. From experience working with today's ageing aircraft, any knowledge of how the materials will behave after a period of environmental degradation will be of great benefit to the platform structural managers of the future. This is especially relevant in the formulation and running of environmental degradation management systems such as an Environmental Degradation Management Plan (EDMP) as used in the ADF. The EDMP details the organisational roles, relationships, people, processes and tools that assess, manage and report the environmental degradation of an airframe. The assessment of environmental degradation is based primarily on condition data, design data and specialist advice. The intent of this study is to add to the specialist knowledge in the area of moisture absorption of CFRP materials. By doing so, this will ultimately have an impact on airworthiness, cost of ownership and availability for a number of platform types in the ADF.

II. Introduction

A. Aim

The aim of this paper is to present the findings of a literature survey on the effects of moisture (saltwater) absorption/desorption cycling of Fibre Reinforced Plastics with the aim of applying this knowledge to Carbon (Graphite) Fibre Reinforced Plastics.

B. Scope

The scope of this paper is the effect of environmental conditioning, namely moisture absorption, of the aerospace materials - Carbon (Graphite) and Glass Fibre Reinforced Plastics (CFRP and GFRP respectively). The conditioning investigated is the cycling of moisture absorption and desorption and the effect of this conditioning on the mechanical properties - compressive, tensile and shear strength.

C. Application

Fibre composite materials are attractive structural materials for high-performance applications that demand attributes such as high specific strength and stiffness, corrosion resistance and fatigue resistance. To-date, these applications have been in the aerospace, marine and petrochemical industries. Carbon (Graphite) composites are widely used within military and civil airframes - especially with the production of the Boeing 787, the Tiger Attack Helicopter and the MRH-90 helicopter. Knowledge and utilisation of composites is also growing in industries such as offshore platforms and infrastructure rehabilitation [2-6]. This research paper belongs in the environmental degradation of aerospace composite materials body-of-knowledge [2]. It is important that the behaviour of such materials following environmental degradation is known so that the health of the platform can be managed throughout its service life.

D. Relevance

The impact of this research is an understanding of the particular phenomena of fluid absorption/desorption cycling that may affect airworthiness, reliability and cost of ownership.

E. Fibre Composite Materials

In general, a fibre composite material is a combination of a filamentary phase embedded in a continuous matrix phase. These constituents retain their identities; that is, they do not dissolve or merge completely into one another although they act in concert. Normally, the components can be physically identified and exhibit an interface between one another [2]. The mechanical behaviour of a composite is not only dependent on the mechanical properties of the constituents; but the quality of the interface between the constituents as well as the volume fraction of the fibres [2, 7]. These mechanical properties are susceptible to environmental degradation phenomena such as moisture absorption, temperature extremes, thermal spiking, osmosis, ultraviolet radiation damage and erosion [8]. These effects on the mechanical properties of many common FRP materials are well researched and known (e.g. GFRP for shipbuilding applications) [3, 5, 9-15]. What research is not readily reported is the effect of saltwater absorption of CFRP. Furthermore, the effect of absorption/desorption cycling is not well researched in general. The reason for this may be preservation of intellectual property following extensive experimentation during development of a platform. Therefore, this paper aims to present the research in these areas and use that information to hypothesis what will be seen for CFRP.

Furthermore, the paper will present the experimental direction that will be taken by the authors to attempt to quantify the effect on mechanical properties of the composite material following absorption/desorption cycling.

III. Literature Review

A. Moisture Absorption by Composite Materials - Mechanisms

Unless a composite material is manufactured and operated in a completely moisture-free environment, it is inevitable that the composite material will absorb moisture throughout its life. Typically, moisture uptake of fibres is negligible in a composite (except for Aramid fibres) - that is, the matrix phase contributes to the bulk of the moisture uptake compared to the fibres. When a composite is exposed to moisture for a sufficient amount of time, moisture concentration through the composite will become uniform. For common epoxies, typical saturation moisture content equates to a 1.1 to 1.3 percent weight gain while typical equilibrium moisture content equates to a 0.6 to 0.9 percent weight gain of the specimen [1]. There are many mechanisms for such water absorption, which include diffusion, capillary action and material voids [7, 8]. All of these mechanisms depend on a number of macro and micro environmental, physical and chemical factors listed in Table 1 [9, 16-20]. These numerous factors interact in such a complex manner that attempting to model their mechanisms or predicting their effects is very difficult.

There is a wealth of research on the mechanisms of moisture absorption of composite materials - and this knowledge is summarised in various texts and handbooks describing the thermodynamics and kinetics of materials. Vidocka [8] has also presented a very comprehensive review of the moisture absorption theory and effects. It is the intent of this paper to build on this knowledge.

In essence, moisture absorption can be characterised in two ways - trapped moisture and free moisture. Trapped moisture relates to chemical bonding between the moisture ions and the structure of the composite (i.e. hydrolysis of polymers); whereas, free moisture relates to both moisture molecules not chemically bound to the composite and moving freely through the composite as well as moisture found on the macro scale within voids or along interface boundaries [1, 7]. Free moisture can be introduced into a composite material through a number of routes, which include diffusion or through preferential routes such as cracks, voids or interfacial disbonding by capillary action [7, 9, 21]. Diffusion is defined by Crank [22] as, “the process by which matter is transported from one part of a system to another as a result of random molecular motions”. That is, over a period of time, liquid molecules will travel in all directions within a composite material, which will eventually lead to a state of equilibrium within the material. Moisture will also be absorbed through the preferential routes that are present due to a material’s manufacturing and service imperfections. Composite materials will always contain imperfections such as voids, cracks and delamination - what varies is the amount of imperfection as a result of manufacturing quality control processes and service environments [1, 7]. Preferential routes that are shaped like small tubes may become routes for capillary action - caused by the adhesion between the wall of the tube and the moisture molecules. The adhesion force during capillary action is strong enough to overcome the mutual attraction of the molecules to pull the molecules up the wall [23]. In summary, free water relates not just to the diffusion process, but phenomena that include capillary action and transport of water through preferential routes or voids.

Table 1: Physical and Chemical Effects from Environmental Exposure

Environmental	Physical	Chemical
• Temperature • Relative humidity	• Specimen geometry • Void content • Matrix mass loss • Composite swelling • The degree of disbonding between matrix and fibres	• The polarity of the matrix molecular structure • The degree of matrix cross-linking • The degree of matrix crystalinity • The presence of residuals in the composite material • Absorbed solution composition • Chemical composition of matrix • Chemical composition of the fibre sizing

The other characteristic of moisture absorption is trapped water. This is the chemical bonding of hydroxide ions (from water) with the structure of the composite material - in particular, to the polymer chains of the matrix material. Most aerospace composite materials are a combination of an epoxy resin matrix and a high tensile strength fibre. Epoxy resins are the most-used polymer matrix for Carbon (Graphite) Fibre composites – being about 90% of the advanced composite materials in current service. Epoxy resins are cured through utilisation of a curing agent (or hardener). This creates a highly cross-linked, three dimensional infusible structure (aka cross-linked polymer chains). These polymer chains are susceptible to hydrolysis as they provide sites for hydrogen bonding of water molecules. This chemical bonding leads to the degradation of the matrix and is commonly known as plasticization [7, 24]. The effects of plasticisation will be discussed in more detail in the section on effects of moisture absorption.

B. Modeling Moisture Absorption in Composites

Diffusion is the most commonly studied phenomenon of moisture absorption and can be represented through mathematical models such as Fick's Laws or the Langmuir model [7-9, 22]. In general, the rate of transfer of a diffusing substance in an isotropic medium, whose structure and diffusion properties are constant in all directions at any point can be characterised by Fick's Law [22]. Fick's Laws can be applied to composites to generate bulk diffusion properties (e.g. diffusivity or exposure/saturation time) as shown by the work conducted by Bonniau et al [9]. as well as Xiao et al [25]. There are limitations, however, to the use of Fick's Law - these include accounting for anisotropic medium and describing only free moisture traveling through a single medium. To account for this, Bonniau and Bunsell [9] suggests that a number of diffusivity constants could be derived for each condition, for example, diffusivity parallel to fibres, perpendicular to fibres and unreinforced resin matrix. That is, discretising the material into its components or interfaces. This is quite a complex approach for application to composite materials; and also assumes that the absorption behaviour is described solely by Fick's Laws (i.e. Fickian).

Non-Fickian behaviour is a term used to classify the phenomena that creates divergence from pure Fickian diffusion. As discussed, this includes free moisture entering the composite by capillary action through preferential routes such as cracks, voids and interfacial delamination; matrix mass loss during moisture absorption, and moisture trapped through hydrolysis. These phenomena are not related to diffusion and thus are not described by Fick's laws. One model that attempts to account for this is the Langmuir model. Langmuir takes a different approach by using probability of a molecule becoming trapped or liberated. If the probability of a molecule becoming trapped is zero, then the model resolves to Fick's second law [7]. Both Fick's Law and the Langmuir model were applied in the work by Bonniau et al. [9] to describe the diffusion of GFRP, where the epoxy was cured with different hardeners. Their studies showed that moisture absorption behaviour is altered by the type of hardener used (i.e. the chemical composition of the matrix) however; correlation between experimental results and the theoretical models can be achieved by refining the model to account for real-world factors [9].

The most detailed modeling method that has been found during this literary review was developed by Whitcomb et al. [26] and is analysed as an Finite Element Model of the composite. These researchers simulated fibre orientation and weave in three dimensions and included the matrix and fibre diffusion properties in their simulation. They were able to show differences in the moisture diffusion properties as a result of the weave microstructure. Their results were verified through correlation with experimental results [26].

C. Moisture Absorption by Composite Materials - Effects

Moisture absorption will affect the behaviour of the composite by altering the physical and thermomechanical properties of the matrix, fibres and interface of the composite material [7, 16, 27, 28]. There are a number of texts that describe these effects in detail [20, 29], thus, the aim of this section is to provide the context to the experiment literature surveyed.

1. Physical effects

Absorption of moisture can be identified by measuring the percentage weight gain of a component over the period of exposure (typically 0.6-0.9 percent weight gain to equilibrium for solid composite components) [1]. The uptake of this moisture leads to swelling of the composite which may cause localised areas of stress and cracking of the matrix. Stresses caused by moisture absorption work in the opposite sign to residual thermal stresses of the matrix following curing. Generally, moisture absorption due to service conditions will reduce the residual thermal stresses. A sudden swelling behaviour may indicate that the composite degradation factors have changed for example, disbonding between the fibres and matrix or delamination between composite plies. Swelling may also be a reversible change in the composite material through desorption [29]. However, swelling can lead to matrix cracking, composite warping or delamination between plies - non-reversible failures. Following from this, it is considered that the reversibility depends on the severity of the swelling, which is important when considering the cycling of moisture absorption and desorption of the composite. Furthermore, cycling is thought to expose the material to more swelling strains and hence accelerate degradation of the composite material properties [30, 31].

2. Thermomechanical effects

Moisture absorption will lead to a reduction in the glass transition temperature (T_g) of the matrix - also known as plasticisation of the matrix. Such a condition must be taken into account when selecting the resin systems for a composite that reduction in the T_g is outside the operation temperature range. Environmental stress corrosion cracking can be accelerated through chemical degradation of the interfacial bonds between the matrix and the fibres [20].

3. Osmosis

A major factor in the saltwater environment as the composite is thought to act as a semi-permeable barrier for saltwater solutions. When a concentration gradient exists, the water molecules will tend toward the region of higher concentration in order to equalise the concentrations [8].

4. Static Strength

The effect of moisture ingress into composite materials static strengths is basically an issue of whether the properties are fibre or matrix dominated. Specifically if the mechanical properties of the composite are fibre dominated then moisture ingress has little effect. If the composite properties are matrix dominated the the moisture ingress will be more significantly effected [2, 32]. In particular, with the combined effect of temperature conditioning and the lowering of Tg there is a more noticeable effect to static strength; such that matrix dominated behaviour should be formally addressed in the design phase under hot/wet conditions. Additionally, compression and bearing properties, whilst dominated by the fibres, is controlled by matrix performance. The matrix provides lateral support of the fibres under micro-buckling. Softening of the matrix due to hygrothermal effects will reduce the compression and bearing strength up to 30% in extreme cases [2].

5. Fatigue Performance

The static properties of composite materials the fatigue properties are also a fibre or a matrix dominated issue [2, 20]. The matrix tends to soften with moisture ingress, which actually improves the resistance of the matrix to crack and restricts crack propagation. Generally, the fatigue performance is slightly improved with moisture uptake in composite laminates.

D. Experiment Methods

1. Current Standards that pertain to moisture cycling in composite material are non-existent. But there are a number of test methods for moisture absorption such as ASTM D 5229 - Testing of moisture absorption of composite materials [27], and Experimental Characterization of High Performance Composites text [33].
2. Research experiments conducted to date includes a variety of experiments that attempt to quantify the effect one phenomenon or a combination of phenomena have on one or many of the mechanical properties of the composite material. For example, the effect of temperature, humidity and UV exposure on the strength and stiffness of graphite epoxy composites was explored by Shin et al [21]. The research conducted by Shin et al. was aimed at identifying the relationship between accelerated ageing tests and natural environmental ageing of a specific graphite epoxy. The outcome was that a correlation was found between the strength and stiffness losses following accelerated and natural environmental ageing. The application of this research is that specimens could be aged in the laboratory for approximately five percent of the natural ageing time but still achieve the same effect - as if they were exposed to the natural environment [21]. These researchers have shown that experimentation can accurately reflect the natural environment if a set of principles are followed - principles that align with the recommendations made by Vodicka [8] when undertaking accelerated environmental testing of composite materials. The recommendations made by Vodicka [8] follow an extensive literary review of the environmental degradation of composite materials and were derived after common elements were identified. They are [8]; a. the environment to be simulated by accelerated testing must be carefully defined, b. an understanding of the impact of environment on composite performance must be assessed; c. temperature and moisture produces a combined effect which significantly reduces matrix dominated composite properties.

The research that was surveyed was able to distinguish Fickian behaviour from non-Fickian behaviour, and was able to refine the method used to analyse or model the moisture uptake behaviour. These models were highly dependent on the accuracy of the experiments and required an in-depth understanding and analysis of the mechanisms of moisture absorption through specialised techniques and equipment. Most identify that moisture is absorbed by a composite material and in short time-frames (depending on specimen through-thickness), the behaviour can be characterised by Fick's Law [3, 9, 17].

3. Saltwater solution used in experimentation varies significantly between the literature studied: from natural source to commercial seawater substitute products. In oceans, the chemical composition of the salt water is very similar and varies negligibly with time. There are a number of parameters that can be measures during experimentation to ensure that the composition remains consistent or whether it changes over time. They include salinity, chlorinity, pH and conductivity. There is no established method for the practical determination of the absolute salinity. There are three main salinity scales introduced in oceanography over the last century. However, there are standard techniques for the measurement of chlorinity, pH and conductivity [34, 35]. A number of methods of simulating seawater have been researched. Chiou et al. [28] used a commercial product to replicate seawater, *Instant Ocean*. This product is used in aquariums and is stated to be the standard in scientific research. The researchers added the commercial product to distilled water and used this solution to condition their samples. They found that the time to saturation was approximately 8 months and that saturation was at ~0.44% of the specimen weight. Gellert and Turley [5] used seawater from a natural source, which was maintained at a measured conductivity (which corresponds to a salinity of about 29%). The specimens were conditioned in 30 degC seawater, either unloaded or loaded at 20% of the maximum strain at flexural failure. The time-scale used for conditioning was in the order of 1,000 days (some samples still did not reach a saturation plateau even at this point). Regge and Lakkad [18] used a concentration of 0.5128 M and conditioned their specimens for durations in the order of hours (120 hours maximum). The standard practice for the preparation of substitute sea water (ATSM D 1141) [36] has also been researched. This standard, which is used in corrosion testing, uses a method of mixing various inorganic salts in proportions and concentrations to produce a solution representative of ocean water. Finally, Bonniau et al. [9] have carried out humidity experiments utilising six different saturated salt solutions within a humidity chamber. The choice of solution gave differing relative humidity values within the chamber, which proved to be relatively stable over a range of temperatures.

E. Moisture Absorption in Fibre Reinforced Composite Design

Throughout the life of a composite material, it will be exposed to various substances that will degrade mechanical properties; one of which is moisture. In aerospace applications, it is inevitable that composite materials will absorb moisture - unless manufactured and operated within a completely humidity-free environment [8]. As discussed, it is the matrix that absorbs the bulk of the moisture that is why it is generally the matrix-dominated mechanical properties that are decreased by moisture uptake [1]. To account for this known and unavoidable factor, designers use 85% relative humidity as a worst-case scenario, as well as a parameter known as the Material Operating Limit (MOL) when designing component strengths. The MOL is the maximum temperature at which a material can operate before there is a dramatic (and sometimes irreversible) reduction in mechanical properties. As shown in Figure 1 moisture uptake and temperature are closely related [1].

Thus, moisture absorption is already factored when designing an aircraft; however, what is not well known is the effect of cyclic absorption/desorption of moisture. The current data stated in MIL-HDBK-17F [1] states that the process is reversible. The process and effects are reversible because the water molecule does not permanently attach itself to the polymer chain and will be readily released under drying conditions. However, there are many non-reversible effects caused by moisture absorption, such as potential residual molecules and elements, impact on resin-to-fibre interfacial bonding, and changes to the glass transition temperature. Also, the moisture swelling may have resulted in permanent damage to the matrix and matrix-fibre interface (i.e. cracking). Swelling strains due to moisture absorption can be mathematically modelled [29]. Thus the effects of cracking can be determined through classical laminated plate theory based on induced hygrothermal strains. This can be evaluated against a suitable failure criterion.

IV. Discussion

The current state-of-the-technology in environmental effects on composite material structures is as following:

1. Moisture absorption/desorption is a reversible process in composite materials.
2. The process is driven by the matrix (resin) absorption characteristic.
3. The fibres play a limiting role in the absorption uptake, but will limit the level of saturation by their increased volume fraction.
4. Lowering of resin rigidity due to moisture ingress will result in a reduction in compressive and bearing strength.
5. For typical composite structures of relative high fibre fraction the moisture saturation level is less than 1% weight gain.
6. The Glass Transition Temperature is reduced due to absorbed moisture and this will lower the allowable operational temperature of the composite structure.

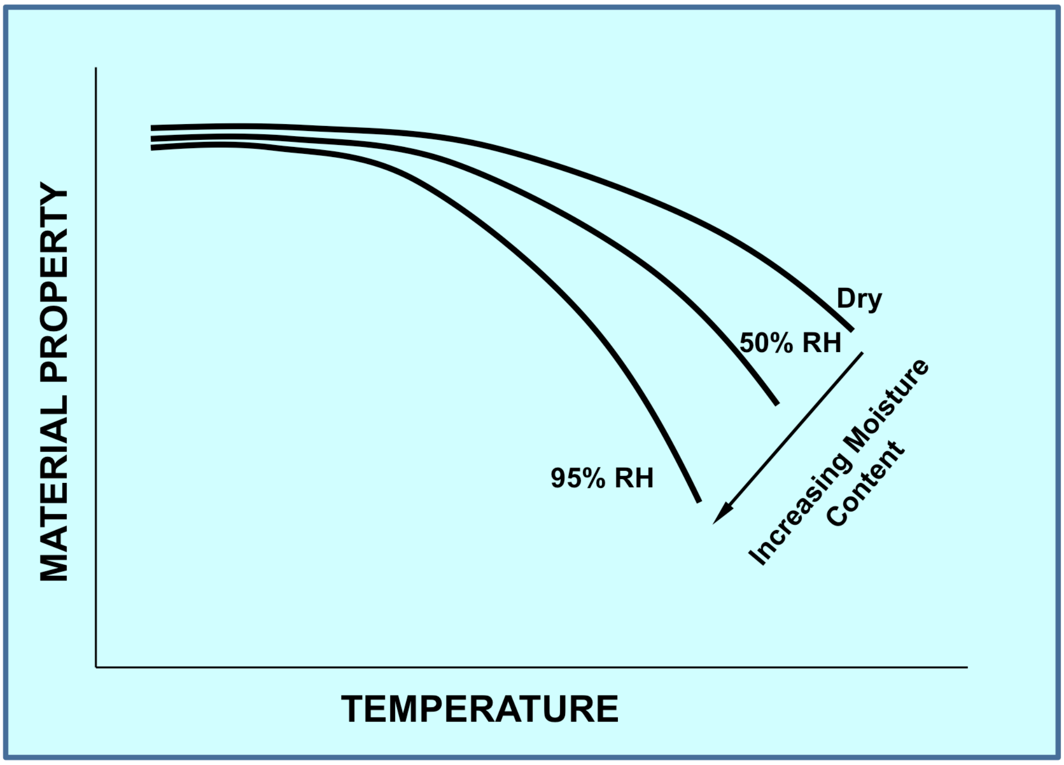

Figure 1 - Influence of temperature and moisture on matrix-dependent failure strain

(redrawing from MIL-HDBK-17)

What is not well understood in composite materials and structures is the effects of environmental cycling and the long-term impact on matrix and fibre/matrix interface behaviour. This is of particular interest in aqueous environmental that contain other elements, such as sea water. The issue is thus: will trace elements be retained in the composite material following desorption of the water molecule, or rather, are the trace elements adsorbed and desorbed in the composite material. This will only be determined by experimental testing with the following goals:

1. High humidity conditioning vs. Saturation Level in a salt laden environment, and
2. Matrix-dominated mechanical properties testing.

V. Closing Comments

Current understanding of moisture absorption/desorption is appropriate for the application to determining the stress-state effects and knockdown factors in composite structure design. The effects on matrix dominated properties are currently taken into account to determine material behaviour. However, there is little to no specific understanding on the cyclic effects of fluid uptake that may level behind residuals such as NaCl molecules from sea water absorption. Thus a study into seawater cycling effects is needed to determine if residuals are having an effect on the composite properties. This is of particular interest for matrix properties and fibre-matrix interface degradation.

VI. References

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