Composites in extreme environments: Effects of high strain rate, humidity and temperature

G Quino1, A Pellegrino1, N Petrinic1

1 Dep. of Engineering Science, University of Oxford, Parks Road, Oxford, OX1 3PJ, United Kingdom

Email:gustavo.quinoquispe@eng.ox.ac.uk

Abstract. Multifunctionality, lightweight and resistance to corrosion are some of the several advantages that fibre composites display with respect to other materials across a wide range of applications. In aerospace and marine industry, however, we can encounter challenges arising from certain working conditions that can significantly affect the mechanical response and integrity of composite structures. First, humidity or water exposure can induce reduction of the material strength and stiffness. Second, hot or cold temperatures that change the level of ductility in the composite. Finally, dynamics loads that may appear during service require strain rate sensitive properties to be considered. In this work, we show experimental capabilities and methodologies developed to account for all those three effects: rapidly applied loads (impact), humidity and temperatures from -55°C to 93°C. This understanding sets the basis for the development of advanced models to design better structures.

1. Introduction
There is no doubt that composite materials have taken an important role in the development of novel structures in a wide variety of industrial fields, ranging from marine, civil, wind energy, and aerospace. Their lightweight, multifunctionality, and resistance to corrosion make them very suitable for such applications. However, there are some challenges that arise from their working environment. In this study, we consider three main challenges: (i) The dynamic loads that the structure must bear, (ii) humid and wet environments, and (iii) the effects of extreme temperatures.

Dynamic loads trigger deformations at high strain rates. For example, a shock in the range of 1-10 m/s can produce strain rates from 100-1000 s⁻¹ [1]. Composite materials for industrial applications are strongly rate dependent; both glass fibre reinforced polymers (GFRPs) [2-5] and carbon fibre reinforced composites (CFRPs) [6-9] have shown, in general, an increase in the strength in many of their failure modes at higher strain rates. Since the mechanical properties of composites depend on the strain rate, there is a need for numerical models that can capture the trend. Such models need to be calibrated and validated against a reliable and comprehensive set of experiments.

Exposure to humid or wet environments tend to reduce some properties of the epoxy resin matrix such as modulus, tensile strength and fracture toughness [10,11], due to chemical interactions of polymer chains and water molecules [12,13], as well as the occupancy of free volumes [14]. Similar effects have been observed over a wide range of strain rates [15]. GFRPs and CFRPs have also been found to reduce their tensile strength, flexural strength, etc., when exposed to pure liquid water [16-19]
and humid air [20-22]. The changes in the material caused by the effects of liquid water and moisture are known as hydrothermal and hygrothermal ageing respectively.

Exposure to wet or humid working conditions takes place over many years. This timescale is almost prohibited in lab conditions, hence accelerated ageing techniques need to be applied. Acceleration of ageing, can be obtained by the increase of temperature [16,23]. Other methods to accelerate the absorption of moisture involve the exposure to varying relative humidities [24].

While ageing provides a slow change in mechanical properties, in-service temperatures can change very rapidly; which translates into a rapid change of properties of polymeric matrices that are, at low temperatures, hard glassy solids, while rubbers at elevated temperatures. Therefore, it is relevant to study the temperature-dependent behaviour of polymer-based composites.

Another pertinent environmental factor to consider, but out of the scope of this study, is UV radiation as it has been shown that decreases the tensile and flexural strength of CFRPs [25,26]. For this, UV chambers or lamps can be used to pre-condition specimens.

It is evident, that methods to replicate in-service conditions within the laboratory are needed. In this work, we discuss the techniques and methods we have developed and implemented to consider the effects of dynamic loads and harsh working conditions: water immersion, humid air, and extreme temperatures.

In the next section the experimental framework is described, including the characterisation methods as well as the techniques to perform hydrothermal, hygrothermal ageing and in-situ temperature conditioning. In section 3, selected results are provided to illustrate the importance of all these considerations and to appreciate how different results can be under different scenarios. Finally, conclusions are given, and future work is suggested.

2. Experimental framework

2.1. Material description

The GFRP system consisted of a 2.6 mm thick [45]s laminate of Formax FGE238 fibres and Prime 20 ULV resin (0.56 volume fraction). The CFRP system consisted of a unidirectional laminate of carbon fibre and toughened resin of 3 mm thickness.

2.2. Mechanical characterisation

Specimen geometry is displayed in figure 1. Dogbone geometry was chosen as it provided with uniform stress field in the gauge length and trigger consistent failure within it. Specimens had threaded metallic endcaps to be mounted on the Hopkinson bar.

This in-house built Hopkinson bar, shown in figure 2, consists of an input and output bar, and was used to execute unidirectional tension experiments at strain rates between 100-200 s\(^{-1}\). The striker was shot by compressed gas, hitting the input bar and generating a tensile stress wave that travels along the input bar, the specimen and output bar. Using the measurements of the strain gauges, one dimensional wave propagation theory, and transmissions and reflections of stress waves, the history of the force acting upon the specimen could be calculated. The history of deformations was calculated using DIC on the images acquired during the test at 500000fps with the high speed camera Kirana from Specialised Imaging.
2.3. Pre-conditioning

In order to replicate the wet conditions during service, lab pre-conditioning should be similar to what is found in the real application, but in an accelerated manner.

For instance, exposure to liquid water of a submarine part can be replicated in an accelerated manner by simply immersing specimens in liquid water at an elevated temperature. This temperature should not be such that unwanted chemical or physical changes take place, because that would cause exaggerated results [15, 27]. A temperature reasonably below the glass transition temperature would be a safe temperature to work with. In this case, GFRP coupons were immersed in a distilled water bath at 50°C until saturation is reached.

For humid air pre-conditioning, we used the climatic chamber CSZ ZP8. This chamber can provide with controlled temperature and relative humidity and allows to program thermal cycles. CFRP specimens were placed in the environmental chamber at 70°C and 85% RH up to full saturation.

In both cases, specimens must be first dry at an elevated temperature to get rid of any humidity that may have been absorbed from the interaction with the environment of the lab, workshop or storage place. In our experience, 3 mm thick samples took about 3 weeks at 50°C to reach the dry state. To ensure that the fully dry and fully wet states are obtained, the weight was monitored periodically with a scale of 0.01 mg precision. The measured weight was used to determine the percent water content \(\%\Delta w_t\):

\[
\%\Delta w_t = \left(\frac{w_t - w_{FD}}{w_{FD}}\right) \times 100\%
\]  

(1)
where \( w_t \) is the weight at the monitoring time \( t \), and \( w_{FD} \) is the weight of the fully dry specimen (\( \%\Delta w_t = 0 \)). Complete drying and complete saturation were identified by the stabilisation of the percent water content. A more detailed procedure to monitor the weight can be found in the standard ASTM D5229 [28].

To investigate the recoverability of properties after saturation, some GFRP specimens were re-dry at 50°C.

2.4. In-situ conditioning
The hottest and coldest scenarios considered for CFRP specimens were 93°C and -55°C; GFRPs were tested at room temperature only. We designed and built an environmental chamber that provides with controlled temperature and can be mounted on the split Hopkinson bar (figure 3). Temperatures higher than the ambient were obtained using a Peltier junction while cold temperature were obtained with dry ice. An Arduino control system kept the temperature at the given set point.

![In-house thermal chamber for in-situ temperature conditioning mounted on the split Hopkinson bar.](image)

3. Results
Figure 4 shows the effects of water immersion on the strength of GFRC. There is a clear drop in strength followed by a small recovery after re-drying.

Results in CFRP, in figure 5, show the effects of exposure to humid air and testing temperature upon the strengths (normalised with respect to the Dry-Cold value). As expected from the cold embrittlement of polymers, cold tests display higher strengths. However, while those tests at 93°C (Hot) display a decrease in strength after full moisture saturation, the results in experiments at -55°C (Cold) show the opposite effect. Further micrographic analysis is required to understand the reasons behind this effect.
4. Conclusions
Composite materials are dependent on strain rate and temperature; hence their mechanical characterisation require the development and implementation of equipment that can replicate such conditions in the lab. In this paper we discussed the techniques used to emulate 3 extreme in-service conditions: dynamic loads, humidity, and cold/hot temperatures. The techniques involved the use of both commercial and in-house designed/built equipment. It was shown how different composites behave depending on the liquid water/moisture pre-conditioning and testing temperature.

5. Future work
SEM images will be taken to try to understand the reasons for the observed effects. In addition, we aim to study hydro/hygrothermal ageing of composites from a microstructural perspective, making use of homogenisation and other numerical techniques.

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