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Influence of matrix ductility on the high-temperature fatigue behaviour of quasi-isotropic woven-ply thermoplastic and thermoset laminates

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ABSTRACT

The tension–tension fatigue behaviours of quasi-isotropic woven-ply thermoplastic (Polyphenylene Sulphide – PPS) and thermosetting (Epoxy)-based laminates are discussed in this paper. The service temperature of these materials is such that $T_g|_{C/PPS} < T < T_g|_{Epoxy}$, and matrix ductility is expected to reflect on the fatigue behaviour. The contribution to the knowledge base from this work rests on the experimental evidence supported by microscopic evaluation at different stages of the fatigue life and fracture surface analysis. These observations exhibit two distinct damage accumulation scenarios in both laminates dominated by fibre breakage in carbon/PPS and debonding, as well as delamination in carbon/epoxy. The potential benefit of matrix-rich regions in woven-fabric laminates is the development of plastic yield zones at the cracks tip as intra- and inter-ply cracks propagate. Depending on matrix ductility, the localized matrix plasticization is instrumental in ruling fatigue damage mechanism, as it may delay the cracks onset and subsequent propagation.

1. Introduction

It is well established that metals and composite materials accumulate damage in different ways under cyclic loading. In metals, failure usually occurs after the propagation of a single macroscopic crack; in contrast, in composite materials it takes places after accumulation of multiple damage modes, such as crazing and cracking of the matrix, fibre/matrix debonding, fibre fracture, transverse-ply cracking, and delamination [1]. These different modes can arise independently or interactively from each other and their onset can strongly depend on both material properties and loading conditions, making it complex to determine the damage appearance chronology during fatigue.

Indeed, the fatigue behaviour of polymer matrix composites (PMCs) is obviously based on the nature of the constitutive elements (matrix and fibres), as well as the orientation of the plies, thickness, and architecture (unidirectional- or woven-ply). It is also well known that time-dependent or ductile behaviours are instrumental in ruling the fatigue response of PMCs [2–7]. Even though there have been some studies on the fatigue behaviour of thermosetting-based (denoted TS) composites, the fatigue response is still an open question when it comes to thermoplastic (denoted TP) laminates displaying a ductile behaviour [8]. TP-based laminates are becoming more and more attractive in industry, because of the promising alternatives compared with TS matrix composites (e.g. PPS – or Polyether ether ketone – PEEK), especially with a reduced manufacturing time and their recycling properties. However, further growth of TP-based composites is directly linked to the knowledge of their long-term behaviour (fatigue and creep), for which there is a lack of experimental data when it comes to investigating their high-temperature fatigue behaviour.

1.1. Literature review

Over the last four decades, damage accumulation under cyclical loading has been widely studied in unidirectional (UD) laminates [9–11] with TS or TP (PEEK essentially) matrices. However, the fatigue behaviour of laminated composites made of woven layers is still a field that raises a lot of questions because of the complex damage behaviours exhibited [12–16]. Indeed, woven-ply laminates are characterized by a very specific geometry, arising from the non-planar inter-ply structure of woven plies and the undulation of the warp fibre bundles over the weft fibre bundles according to the given weave pattern [17]. Such reinforcement architecture is usually characterized by regions where a plain matrix will be predominant after consolidation. These regions are prone to damage initiation, and cracks usually appear in the transverse fibre bundles and can propagate across the matrix-rich areas, or at the interface between the warp and weft fibres in the overlapping areas in the

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case of on-axis loadings [18,19]. This phenomenon is also known as "meta-delamination" (see Section 3.2.3). The fatigue behaviours of UD and woven laminates have been studied, and proved to be very similar for quasi-isotropic (Q-I) lay-ups [20], even if damage seems to appear earlier in woven-ply laminates. The damage scenario is also closely related to the test conditions, the specimen's geometry and the matrix ductility. Indeed, the concept of introducing soft regions, sometimes referred to as softening strips, into a fibre composite to provide barriers to crack growth depending on the matrix ductility and so, raise the intrinsic toughness of the material [21]. As far the fatigue performance is concerned, it is therefore potentially interesting to associate woven fabrics with highly ductile TP matrices [2,22], the effect of which is even more noticeable when service temperature is higher than the material glass transition temperature [23], even in Q-I laminates whose behaviour is fibre-dominated. Because of the possible time-dependent behaviour of the polymer matrix, PMCs are highly sensitive to the loading conditions, especially the loading frequency, even in laminates with a stacking sequence whose behaviour is fibre-dominated. This aspect is closely related to dissipation and heat generation during cyclic loading [24]. In fibre-reinforced epoxy laminates with an orthotropic lay-up, longer fatigue lives have been reported as frequency increases [25]. Moore [26] studied the frequency's influence on UD C/PEEK laminates with Q-I sequence for 0.5 and 5 Hz under tensile–tensile fatigue solicitation, and found that an increase in frequency results in a shorter fatigue life. The same results were found on C/PEEK angle-ply laminates between 1 and 10 Hz [27–30]. Pandita et al. [18], and Lin et al. [27] observed significant temperature increases at the specimen surface as frequency increases in laminates subjected to off-axis loadings. This temperature can even exceed the material T_g, decreasing dramatically its mechanical behaviour and explaining a shorter fatigue life. It is even more noticeable in highly ductile TP-based laminates. It also appears that, at high loading levels, the increase in loading frequency will result in an increase and accumulation of cyclic deformation due to local heating and stress heterogeneity, as evidenced by the increase in damage rates [31]. The presence of matrix-rich regions in woven-ply laminates makes it essential to evaluate the contribution of time-dependent behaviours (viscoelasticity and viscoplasticity) to the fatigue behaviour of TP-based laminates at high frequencies. The specimen geometry is also of importance, especially for fatigue loadings. From the conclusion drawn in [32], it appears that specimens with a common rectangular geometry with end tabs, as recommended by standards ASTM D3039 and D3470, were not suitable for cyclic loadings on C/PPS laminates. A large majority of specimens failed underneath the end tabs, resulting in under-estimating fatigue life. Thus, the authors adopted dumbbell-shape specimens which proved to be much more relevant for this type of loading compared with rectangular specimens.

1.2. Fatigue behaviour of five-harness satin weave carbon-fibre reinforced PPS

The fatigue behaviour of five-harness satin weave carbon-fibre reinforced PPS was investigated at room temperature [16,32–36], in the case of on-axis and off-axis tensile fatigue loadings on $[(0,90)]_{4s}$ lay-up, with a stress ratio $R = 0$. With regard to the material itself, C/PPS has a very brittle behaviour under on-axis tensile fatigue loadings as there is no prior sign of failure (little stiffness degradation or permanent strain) [16]. When woven-ply C/PPS laminates are subjected to fatigue loadings, the dominant damage mode is not a gradual increase in the number of broken fibres, but rather seems to be related to the matrix [33]. Fibre breakage is primarily observed at the beginning (when the weakest fibres break), and at the end of the test when a catastrophic failure happens. From microscopic observations, it appears that damage mechanisms primarily consist of matrix cracking and meta-delamination instead of fibre breakage [16]. The frequency effect (2 and 5 Hz) on the fatigue lifetime has also been studied, as well as heating by measuring temperature at the surface of specimens. It turned out that higher frequencies yield shorter fatigue life at low stress levels, and this effect seems to decrease as the applied stress increases [36]. Regarding the off-axis sequence $[(+45,-45)]_{4s}$, the authors undertook fatigue tests at 1 and 2 Hz. Surprisingly, a small frequency increase tends to yield an increase in the specimen's life [33]. The fatigue behaviour can be described by three primary steps: (i) the run-in of the fatigue test where a certain amount of permanent deformation occurs without an increase in temperature; (ii) a steady-state phase where there is a gradual increase in permanent elongation without an increase in temperature; (iii) the end-of-life where there is a sudden growth in both temperature (higher than T_g) and permanent elongation. This type of behaviour is confirmed by [7] in woven carbon/bismaleimide laminates at different temperatures. The authors also showed that the hysteresis loops become bigger during the last phase of fatigue life, suggesting significant energy dissipation [33]. A creep test was also performed to verify if the fatigue permanent elongation results from fatigue damage, time-dependent effects or a combination of both. Thus, the strain increase during the fatigue test was shown to be primarily caused by fatigue damage. From this brief literature review, it appears that very few authors have investigated the high-temperature fatigue behaviour of carbon woven-ply PPS laminates. Franco et al. performed fatigue tensile tests (8 Hz – $R = 0.1$) at 80 °C to understand the relationship between fatigue resistance and environmental conditioning by a fractography analysis of the fracture surfaces [22]. They concluded that temperature is the most detrimental factor to fatigue life. In addition, considering the ductile nature of the PPS matrix at 80 °C (close to its T_g), the observation of failure surfaces shows that the matrix is characterized by a plastic deformation aspect, suggesting that the PPS matrix yields until fatigue failure. Finally, there are very few references available in the literature on the fatigue behaviour of PMCs when temperature is near to or higher than their glass transition temperature T_g [4–5,37]. Besides, they all examine the fatigue behaviour of UD-ply laminates.

1.3. Objectives of the study

The underlying idea of this work was to determine whether C/Epoxy can be replaced by C/PPS materials for applications in secondary structures of aircraft engine's nacelles, or not. Advanced aeronautics structures, and particularly nacelles, require high-performance fibre-reinforced PMCs, which can be used at maximum service temperature of about 120 °C. Both materials are therefore not tested in the same physical conditions (one being tested above its T_g and the other being tested below T_g). Thus, C/PPS laminates have a much more ductile behaviour than C/Epoxy laminates, and ductility is expected to reflect on the fatigue behaviour of Q-I laminates. In such an industrial context, the purpose of this study was multifold:

First, the tensile fatigue behaviours of TP- and TS-based laminates were studied at test temperatures T such that $T_g|_{C/PPS} < T < T_g|_{Epoxy}$. Through a comparison of two composite systems, the basic idea was to investigate the extent to which loading frequency influences the fatigue behaviour, and the extent to which the plain matrix regions may contribute to the fatigue damage mechanisms of Q-I woven-ply laminates. Second, the contribution to the knowledge of this work is also based on experimental evidence supported by microscopic observation and a fractography analysis of specimens at different stages of fatigue life in order to determine precisely the damage chronology. Finally, the concept