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In situ analysis of delayed fibre failure within water-aged GFRP under static fatigue conditions

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Abstract

A stress corrosion model has been applied to the microscopic analysis of the delayed fibre failure processes occurring within a water-aged unidirectional glass/epoxy composite under static fatigue loading (i.e. relaxation). By means of in situ microscopic observations, the individual fibre failures within an elementary volume located on the tensile side of the flexural specimens have been quantified as a function of time under various applied strain levels. It was found that the time dependence of the in situ fibre failure processes obeyed a stress corrosion model. From the microscopic observations, it was possible to assess consistent values of the parameters characterising the in situ fibre strength distribution and the subcritical crack propagation law. A comparison with separate static fatigue experiments using unimpregnated fibre bundles demonstrated that the specific physico-chemical environment encountered by the glass fibres within the aged epoxy matrix can induce significant changes in the subcritical crack propagation rates, as compared to stress corrosion cracking data collected in humid air.

Keywords: Glass/epoxy; Water ageing; Three-point bending; Fatigue; Stress corrosion

1. Introduction

The assessment of the durability of polymer matrix composites is a key problem in many structural parts exposed to the combined effects of fatigue loading and environmental ageing in hot and/or wet conditions. One of the main difficulties regarding the development of reliable durability models arises from the interaction between several physico-chemical and mechanical processes, which occur over very different time and length scales. As each degradation process has its own activation energy, a predictive approach to the durability problem may indeed become rapidly untractable. In some particular circumstances, it is, however, possible to identify the predominant physico-chemical and mechanical degradation mechanisms involved in the lifetime of the structure. In such a context, it may be reasonably envisaged to develop of physically-based durability model and the associated accelerated characterisation procedures.

These requirements are at least partly fulfilled in the case of unidirectional glass/epoxy composites subjected to tensile or flexural fatigue, i.e. in a situation where the delayed tensile failure of the moisture-sensitive fibres can be regarded as the primary event which controls the process of damage development [1–3]. During moisture diffusion through the composite material, the accumulation of water at the interface can significantly enhance the stress corrosion processes involved in the failure of the glass fibres, thus resulting in a decrease in the fatigue lifetimes of the aged composite [4,5]. Although stress corrosion cracking (SCC) processes in bulk glasses have been investigated in detail, the relevance of the associated concepts within the context of the fatigue behaviour of glass fibre-reinforced composites has been the subject of a limited number of investigations [6–12]. The available research work essentially demonstrates that an SCC approach can account for some of the main features of the kinetics of macroscopic crack propagation within corrosive environments. Little insight was provided, however, into the SCC effects upon the initial microscopic damage accumulation stages which play a major role in the nucleation of cracks.

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The present study was directed towards establishing the relevance of SCC concepts to describe—at the microscopic scale—the accumulation of broken fibres during the initial stages of the fatigue life of aged unidirectional glass/epoxy composites. The main objective was to determine whether the time dependence of the delayed fibre failure processes exhibits some of the main characteristic features which are associated with subcritical crack growth within glass materials. In addition, the question was also to determine how the subcritical crack growth rates were affected by the physico-chemical environments encountered by the fibres within an aged epoxy matrix. The approach was essentially based upon an in situ microscopic analysis of the initial fibre failure processes which occur within a flexural specimen, before the nucleation of macroscopic matrix cracks. Under such loading conditions, the broken fibres are concentrated within the superficial composite layers located on the tensile side. This allowed us to restrict the optical observations to a limited material volume which was located on the specimen surface and beneath the loading span, in an area where a significant portion of the overall amount of broken fibres was concentrated. Moreover, stress and ageing conditions within this reduced elementary volume could be regarded as uniform, which simplified considerably the application of the SCC model.

2. Experimental details

2.1. Materials

A unidirectional composite was manufactured using a glass reinforcement and an epoxy matrix. Commercial E-glass fibres (Advantex®) provided by Owens Corning Fiberglass (OCF, France) were used in the form of a 1200 tex roving. The average fibre diameter was 13 μm. During manufacturing, the fibres were coated by a diglycidyl silane-based sizing. The epoxy matrix was obtained by mixing 100 parts of a DGEBA-based prepolymer (Epikote 828, Shell) with 90 parts of an anhydride hardener (NMA).

The composite specimens were manufactured using a filament winding technique which ensured an optimum impregnation of the bundles with a limited amount of trapped air bubbles (the void fraction was less than 0.5 vol%). The composite was cured at 80°C for 2 h followed by 1 h at 140°C. Plates of thickness 2 mm were obtained with a fibre fraction of 57 vol%. Specimens $2\times10\times100$ mm^3 have been cut from the plates for the three-point bending experiments. The glass transition temperature of the unaged material was 140°C (as measured by dynamic mechanical thermal analysis (DMTA), at 2°C/min and 2 Hz).

2.2. Microscopic observation of delayed fibre fracture under three-point bending conditions

A three-point bending testing machine was used that allows the in situ optical detection of the broken fibres on the tensile side of the composite specimens [4,13]. A span-to-depth ratio equal to 30 ensured that the shear stresses were minimised. The fibre failure processes were monitored using a microscope device which was mounted on a three-axis stage driven by stepper motors located in front of the composite specimen; Fig. 1. The X and Y axes of the stage allowed scanning on the tensile side of the specimen in the vicinity of the loading nose, while the Z axis was used to focus on the analysed surface. The superficial broken fibres appeared as dark spots (Fig. 1) which were quantified by means of an image analysis software. The experiments to be reported consisted in acquiring successive images within a restricted area 1.5×7 mm^2 which was centred at midspan. The observations were restricted to the initial stages of the damage development, i.e. before the appearance of macroscopic damage in terms of matrix cracks. Two different loading procedures were used:

1. *Step-by-step monotonic loading*. The specimens were loaded incrementally at 5 mm mn^{-1} up to the optical detection of a macroscopic damage in the analysed area. At each strain increment, the specimen surface was scanned to detect the broken fibres. This image acquisition stage took about 2 min. The experiments have been carried out using an average of six strain increments before the occurrence of macroscopic damage.
2. *Relaxation*. The specimens were loaded at 5 mm mn^{-1} up to the specified value of the imposed strain. Image acquisition was carried out periodically during the relaxation stage using the same scanning procedure as for the monotonic tests.

2.3. Ageing conditioning

The water conditioning procedure of the composite specimens was designed to achieve a homogeneous ageing state within the elementary volume under investigation. The appropriate ageing time and temperature were deduced from a preliminary analysis of the water sorption kinetics. The latter was based on classical weight gain experiments [14] carried out in distilled water. At moderate temperatures (25°C and 40°C), the change in the relative weight gain, M_t, as a function of time exhibited a classical Fickian shape with an equilibrium weight gain close to 0.5%. At a more elevated temperature (60°C), matrix hydrolysis was evident from the positive deviation of the sorption curve from the saturation level. Room temperature conditions (25°C) were

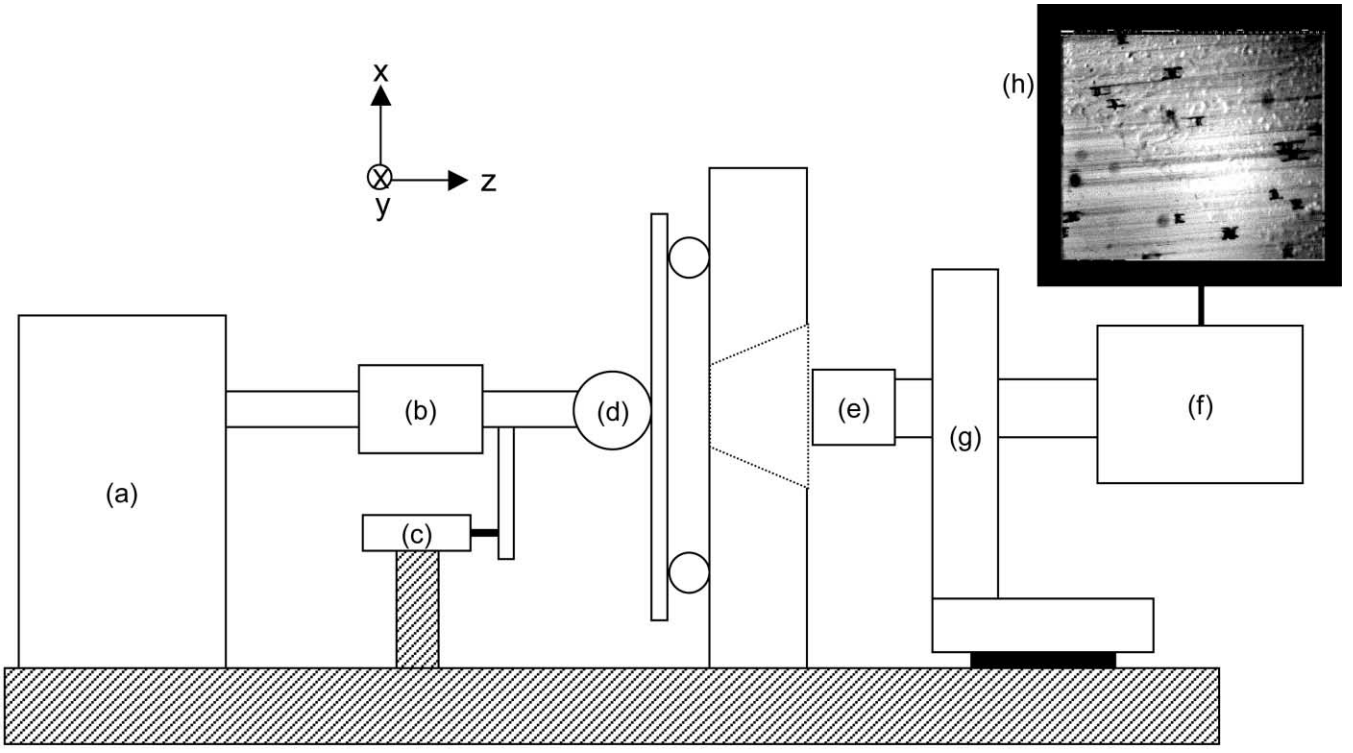

Fig. 1. Schematic description of the three-point bending device equipped with an optical microscope: (a) motor driving unit; (b) load cell; (c) LVDT transducer; (d) glass span; (e) microscope; (f) CCD camera; (g) three-axis stage; (h) frame grabber and image analysis software.

therefore selected for all the experiments to be analysed to avoid the heterogeneous degradation which can result from the combination of hydrolysis with transient water diffusion steps.

An ageing time of 10 days was found to be sufficient to ensure the saturation of the analysed superficial layers of the composite. A Fickian analysis of the 25°C sorption curves provided a value of about 1.2×10^{-13} m s^{-1} for the diffusion coefficient, which means that at the end of the conditioning procedure, about 90% of the water saturation level was reached within the maximum observable thickness (about 150 μm).

3. Results and discussion

3.1. In situ statistical distribution of fibre strength

The first stage in the analysis of the delayed fibre fracture processes consisted in determining the initial strength distribution of the reinforcement within the composite. This analysis was carried out using non-aged composite specimens under a monotonic flexural loading. In such conditions, the stress corrosion mechanisms were assumed to be strongly reduced compared to the water-aged specimens. As mentioned above, the observations were restricted to the initial microscopic damage accumulation stage, which means that the tests were stopped as soon as transverse matrix cracks propagated through the scanned area. In Fig. 2, the number of broken fibres within the analysed elementary volume is reported as a function of strain. These data points correspond to six separate experiments carried out using different specimens, illustrating the good reproducibility of the experiments.

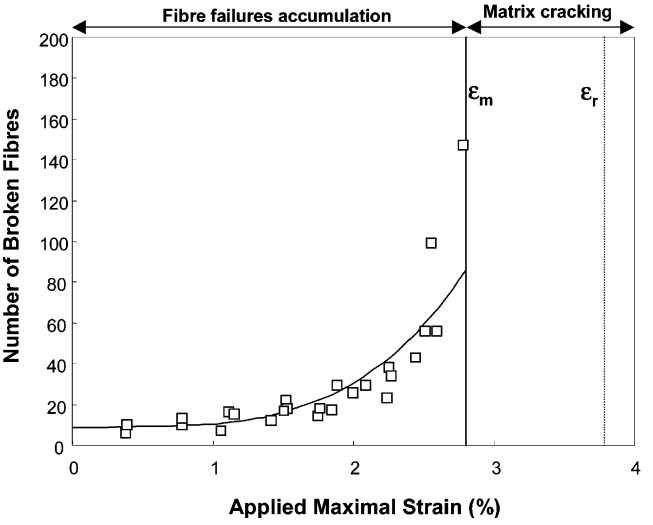

Fig. 2. Number of detected broken fibres as a function of the applied maximal strain under monotonic loading. Unaged composite; ε_m is the average strain level corresponding to the appearance of a macroscopic damage within the analysed area; ε_r is the mean failure strain corresponding to the maximum load.

In a first stage, the potential of a simple Weibull analysis to describe the change in the density of broken fibres as a function of strain was investigated. As a first-order approximation, it was considered that the fibre portions enclosed within the elementary volume under investigation behave like isolated glass filaments in a bundle. This assumption implies that the contribution of the stress transfer processes to the first fibre failures can be neglected. The validity of this hypothesis was supported by two pieces of experimental evidence. First, very limited multi-fragmentation of single fibres was found to occur within the investigated strain range, which indicates that the reloading of broken fibres by interface stress transfer did not contribute appreciably to the overall fibre failure processes. Second, the formation of n-plets of broken fibres due to stress concentration in the vicinity of a broken fibre (Fig. 3) was found to be restricted to elevated applied strains (ε>2.5%). Below this threshold, the detected broken fibres may thus be reasonably considered as non-interactive defects.

Within the frame of the "bundle" approximation, the number of broken fibres at a given strain level is simply given by $(1-P_s(\varepsilon))N_t$, where P_s and N_t are respectively the probability of survival and the total number of fibres within the elementary volume. P_s was assumed to follow classical Weibull statistics [15]:

$$P_s(\varepsilon)=e^{[-(\varepsilon/\varepsilon_0)^m]} \quad (1)$$

where ε is the applied strain, ε_0 is a scaling factor including the effects of the fibre gauge length within the analysed elementary volume and m is the Weibull modulus.

P_s was calculated from the ratio of the number of broken fibres, N_f, to the total number of fibres, N_t, within the elementary volume under investigation. The determination of N_t relies on the estimate of the thickness of the elementary volume, which is not known with precision. Two limiting values may, however, be derived which correspond respectively to a single fibre layer (~15 μm) and the depth of focus (~150 μm) of the microscope objective used. The corresponding values of N_t were respectively 450 and 4500 fibres. It is worth noting that this uncertainty in the value of N_t affects the estimate of the scaling parameter, ε_0, but does not significantly affect the determination of the Weibull parameter, m. This can be verified in Fig. 4, where $\ln(1/P_s)$ has been reported as a function of strain in a log–log plot for the two limiting values of N_t. Two straight lines are obtained with nearly identical slopes, yielding an average value of m=3.65±0.05. This value is consistent with previously reported results for unimpregnated E-glass fibres [16–18].

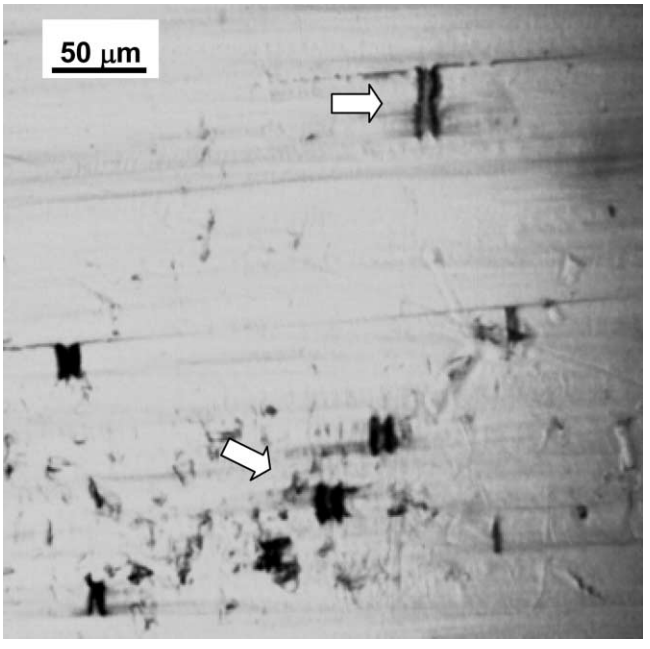

Fig. 3. Formation of n-plets of broken fibres (indicated by white arrows).

3.2. Delayed fibre failure processes within the aged composite

3.2.1. Kinetics of damage accumulation

The occurrence of subcritical crack growth processes within the aged composite was investigated by monitoring the delayed fibre failures within specimens subjected to a constant strain condition. In these experiments, the SCC processes were activated by ageing the specimens in water prior to mechanical testing. As in the monotonic tests, the analysis was restricted to the early stages of damage accumulation, i.e. before the appearance of macroscopic cracks. At the macroscopic scale, the corresponding maximum stiffness loss was about 5%.

The effects of the ageing conditioning upon the delayed failure of the glass fibres are shown in Fig. 5. At 25°C, no significant difference was observed between 10 and 23 days immersion, which indicates that a stationary state is reached within the analysed volume

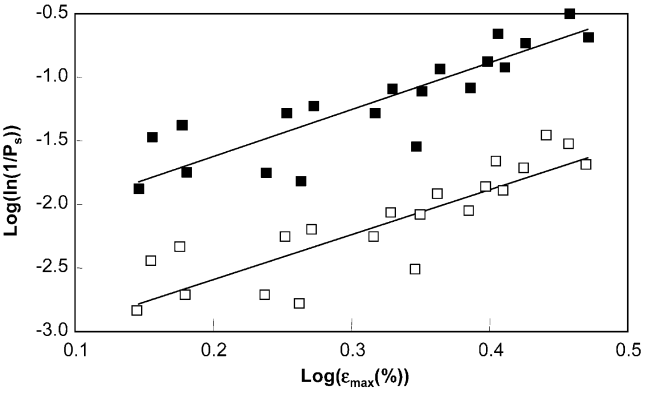

Fig. 4. Weibull plot of the fibre probability of survival, P_s, for two hypothesis regarding the maximum number of fibres, N_t, within the analysed elementary composite volume: (■) N_t=450; (□) N_t=4500.

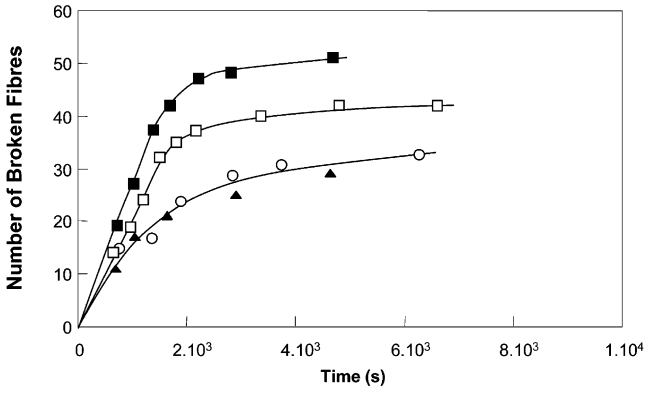

Fig. 5. Number of broken fibres against time under relaxation loading (ε_{max}=1.7%). Composite aged (▲) 10 days at 25°C; (○) 23 days at 25°C; (□) 5 days at 60°C; (■) 18 days at 60°C.

regarding SCC mechanisms. On the contrary, data at 60°C reveal that the delayed fracture processes are activated as the ageing time increased. This result is indicative of the hydrolytic degradation of the epoxy matrix, which affects the subcritical crack growth through a change in the physico-chemical environment of the glass fibres. In such a situation, the homogeneous ageing state of the composite volume under investigation can no longer be guaranteed, which complicates considerably the identification of the SCC model from the experiments. As a result, only the data obtained after 25°C water ageing will be considered in the subsequent part of this paper.

In Fig. 6, the number of broken fibres has been reported as a function of time for various applied strain levels. At the most elevated strains (ε_{max}=1.9% and 2%), the tests were interrupted due to the early appearance of macroscopic damage in the form of transverse cracks. On the other hand, saturation of the number of broken fibres is achieved for $\varepsilon_{max}\leq$1.8%. This latter observation could be interpreted by considering that, in the plateau

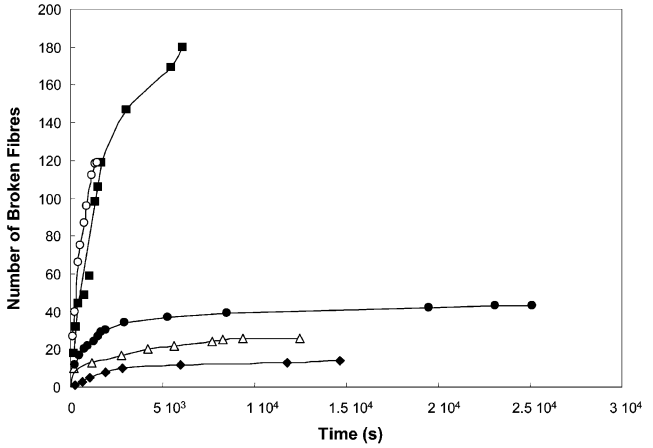

Fig. 6. Effects of the strain level upon delayed fibre fracture under relaxation loading. Aged composite: (◆) ε_{max}=1.5%; (△) ε_{max}=1.7%; (●) ε_{max}=1.8%; (■) ε_{max}=1.9%; (○) ε_{max}=2.0%.

region, the remaining fibre defects are loaded below their crack propagation threshold.

In order to investigate more quantitatively the features of the fibre SCC processes within the composite, the kinetics of microscopic damage accumulation was analysed within the framework of the "bundle" approximation. It was thus considered that the composite fibre failures obeyed the same SCC laws as within a fibre bundle under tensile loading. In a previous investigation using unimpregnated E-glass fibre bundles [18], we have shown that the changes in the subcritical crack growth rate, v, as a function of the stress intensity factor, K_I, exhibits the typical features of SCC mechanisms, namely the occurrence of a crack propagation threshold followed by a stage I and a stage II. Accordingly, the subcritical crack velocity, v, within stage I can satisfactorily be expressed as a function of K_I using a classical power law expression:

$$v=\frac{da}{dt}=AK_I^n \tag{2}$$

with $K_I=\sigma a^{0.5}Y$, where a is the crack length, σ is the applied stress, Y is a geometrical factor and A and n are two constants.

On this basis, an attempt was made to describe the fibre lifetime distribution within the composite elementary volume from a combination of Eq. (2) with the above-determined Weibull distribution of fibre strength. The fibres within the composite were loaded under constant strain conditions which are equivalent to constant stress conditions within each fibre. Under such a loading, a fibre fails at the time t_f due to the growth of a crack from an initial length a_i to a critical length a_c. The lifetime, t_f, can thus be obtained from the integration of Eq. (2):

$$t_f=\int_{a_i}^{a_c}\frac{da}{AK_I^n}=\frac{2K_{IC}^{2-n}}{AY^2(n-2)}\sigma_i^{n-2}\sigma^{-n} \tag{3}$$

where σ_i is the strength associated with the brittle propagation of the initial defect and σ is the constant applied stress. Eq. (3) can be simplified as follows:

$$\sigma_i=Ct_f^{1/(n-2)}\sigma^{n/(n-2)} \tag{4}$$

with

$$C=\left[\frac{AY^2(n-2)}{2K_{IC}^{2-n}}\right]^{1/(n-2)}.$$

In the context of a Weibull statistical distribution of defects, the probability, P_s, that a fibre will survive at time t is thus given by:

$$P_s(t)=e^{\left[\left(\frac{Ct^{1/(n-2)}\sigma^{n/(n-2)}}{\sigma_0}\right)^m\right]}=e^{[kt^{m/(n-2)}\varepsilon^{mn/(n-2)}]} \tag{5}$$

where σ_0 and k are two scaling constants. Accordingly,

a log–log plot of $\ln(1/P_s(t))$ against the time, t, will be linear, if the theory is appropriate, and the slope will yield a value for $m/(n-2)$. Note that the validity of this approach is restricted to stage I failure and that some deviation from the theory can be expected for the fibres which are loaded close to the propagation threshold. Similarly, the theory does not hold for stage II and stage III fibre failures, but the latter are not relevant to the composite fatigue behaviour, as the associated lifetimes are much lower than the composite lifetimes.

The time dependence of the fibre survival probability, $P_s(t)$, was assessed from the measured broken fibre density, assuming that the total number of fibres was N_t=4500. When $\ln(1/P_s(t))$ is plotted against $\log t$, linear relationships are obtained (Fig. 7), which supports the validity of the SCC approach (Eq. (5)). At ε_{max}=1.8% and for long loading times, a downward curvature of the $\ln(1/P_s(t))$ vs. $\log t$ curves can be noted. This deviation from linearity may be because an increased part of the remaining fibre surface defects are loaded close to the propagation threshold, i.e. in a domain when the subcritical crack growth rates predicted by Eq. (2) are overestimated. At lower strains, a similar trend would probably be observed, but for much longer times.

The kinetics of delayed fibre fracture also appears to be activated in a non-linear manner when the applied strain is increased. Such a characteristic is consistent with the trends which could be expected from the stress dependence of the subcritical crack growth rate during stage I: according to Eq. (2), the crack velocity within this range is proportional to the applied stress to the power n.

Whatever the applied strain, the slopes of the initial linear portions of the curves were roughly identical. From their average values, the ratio $m/(n-2)$ was estimated to be about 0.45±0.04. Similarly to the strength analysis, this value was found to be independent of the selected value for N_t. Using the above-determined value of the Weibull modulus (m=3.65), this result yields n=10, i.e. about half of the value (n=24) which was previously obtained from the tensile testing of unimpregnated E-glass fibre bundles in a humid environment [18].

3.3. Determination of the in situ subcritical crack growth rates

In order to confirm the intrinsic SCC behaviour of the fibres, an independent estimate of the value of the stress corrosion parameter, n, was obtained from the determination of the in situ v–K_I relationship. The latter was carried out by means of a methodology which was initially proposed by Fett and Munz [19] in the context of bulk glasses. In a previous investigation [18], we have successfully used this approach to determine the subcritical crack-growth rates within unimpregnated E-glass fibres under static loading. In the present study, the same scheme was applied to the analysis of the delayed failure processes within the elementary volume of an aged composite under relaxation loading.

As described fully in [18], the analysis is based on the combination of the initial fibre strength distribution and the lifetime distribution under relaxation. The former was determined from the monotonic tests carried out using the unaged composite (data reported in Fig. 2), while the lifetimes data were provided by the relaxation tests carried out using aged specimens (see Fig. 6). The treatment was repeated for the two limiting values of the number of fibres within the elementary volume under investigation. Whatever the selected value for N_t, the resulting v–K_I curves (Fig. 8) exhibit the typical features of SCC processes, namely a propagation threshold and a stage I. Within stage I, the experimental data yield a value of n=11, which is independent of N_t and very close to the evaluation derived from the slope of the $\log(\ln(1/P_s(t))$ vs. $\log t$ relationship. The good agreement

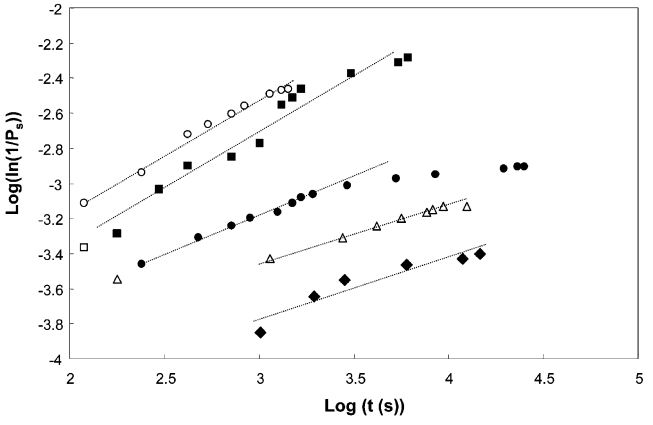

Fig. 7. Log–log plot of $\ln(1/P_s)$ against time, t, for the stress corrosion cracking of glass fibres within the aged composite: (◆) ε_{max}=1.5%; (△) ε_{max}=1.7%; (●) ε_{max}=1.8%; (■) ε_{max}=1.9%; (○) ε_{max}=2.0%.

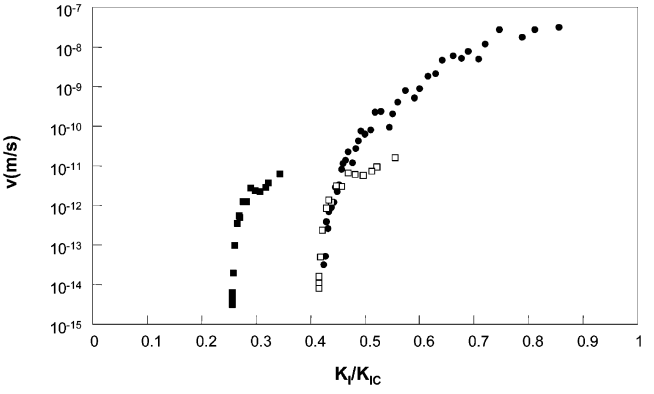

Fig. 8. Change in the subcritical crack growth, v, as a function of the ratio K_I/K_{IC}. (a) Glass fibres within the aged elementary composite volume: (■) N_t=450; (□) N_t=4500. (b) Data from tensile tests using unimpregnated E-glass bundles 50% RH (●).

between these two independent calculations of the crack growth parameter, *n*, further supports the consistency of the SCC approach during the early stages of fatigue damage accumulation.

The in situ v–K_I relationship for the aged composite can be compared to the data which have been obtained previously in the context of the tensile testing of unimpregnated fibres bundles exposed to 50% RH [18]. Compared to the unimpregnated fibres, the subcritical crack velocities within the aged matrix can be decreased by more than one decade during stage I (Fig. 8). This strong decrease in the crack growth rate is indeed reflected by the change in the stress corrosion parameter, *n*, from 24 to 11. Because of the uncertainty in the exact value of N_t, it is more difficult to draw any definite conclusions regarding the crack propagation threshold. There is, however, some indication that this threshold could be reduced in the composite by water immersion by comparison with unimpregnated fibres exposed to humid air.

The different physico-chemical environments encountered by an unimpregnated fibre exposed to water vapour and a fibre embedded within a water-aged epoxy matrix can probably account for the observed differences in the SCC behaviour. Within the aged composite, the local chemical degradation of the interphase may induce some specific changes in the pH of the surrounding media, which is known to have a strong influence on the subcritical crack growth rates in glasses [16]. Such physico-chemical changes probably represent the main contribution to the observed reduction in the stress corrosion parameter, *n*, which was found from the bundle relaxation tests to be relatively insensitive to the humidity level.

4. Conclusion

The microscopic analysis of aged composite specimens under flexural static fatigue conditions demonstrated that the fibre failure processes during the initial stages of the lifetime can adequately be described using a stress corrosion model based on the initial fibre strength distribution and the subcritical growth of pre-existing flaws on the fibre surface. This approach was restricted to the microscopic damage steps, i.e. before the occurrence of macroscopic cracks and/or delamination. Under such conditions, the damage development mainly involves the accumulation of microscopic fibre failures which act, to a first-order approximation, as non-interactive defects. The application of the SCC model required the determination of the Weibull modulus characterising the initial strength distribution and the stress corrosion parameter associated with stage I subcritical crack growth. The latter has been determined independently by two separate methods. The first was based on the time dependence of the fibre survival probability, while the second involved the combination of the lifetimes and the initial strength distribution. Both methods provided very similar results, which supports the overall consistency of the SCC model.

From the microscopic observations, it was possible to assess the in situ subcritical crack growth rates of the glass fibres in the real physico-chemical environment they encountered within the aged matrix. Separate SCC experiments carried out using unimpregnated fibre bundles demonstrated that the stress corrosion characteristics of the glass fibres within the aged composite can barely be extrapolated from the data provided by fibres exposed to humid air or liquid water. In the present case, it is worth noting that a strong reduction in crack growth rates during stage I occurred for the fibres embedded in the matrix, compared with the fibres in air. This result emphasises the definite effect of the physico-chemical properties of the aged interfaces on damage accumulation during the early stages of the fatigue life of glass/epoxy composites.

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