ASSESSMENT OF SINGLE FIBRE FRAGMENTATION AS A METHOD FOR ACCELERATED MICROMECHANICAL TESTING OF GLASS FIBRE LAMINATES

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ABSTRACT

In the current paper the single fibre fragmentation, SFF, method is evaluated as test method for accelerated testing of the composite constituent properties. The SFF-method offers a possibility to characterise matrix, interface and fibre properties. The idea is to characterise the constituents change over time due to environmental loading and then use micromechanics to model composite properties of an arbitrary geometry. A modified two-step manufacturing method has been developed to fit the purpose of the accelerated tests. The experimental results are in agreement with other studies and show a large data scatter, making data interpretation difficult. The paper is concluded with a discussion about lessons learned from the present work and a judgement of the feasibility of the SFF and the modified SFF test for ageing studies.

1. INTRODUCTION

There is a potential to significantly extend the use of polymer composite materials in areas such as infrastructure, transport, naval etc. provided that cost effective, yet accurate, methods to assess the service life of composite structure are made available. This is possible to achieve only if all relevant mechanical and environmental loads are known and it is understood how the material response to these loads evolve over time. A lot of work has been done in this area [1,2] but current methodology based on material characterisation and modelling of a homogenised material response at coupon level is rather crude and it may be questioned whether the results may be generalised to other temperatures or geometries than those actually tested. Consider for instance methodology to characterise the influence of humidity or solvents on the long-term properties. Coupons are by tradition relatively large which causes a transient environment inside the material, a transient that is present for a long time, until saturation is reached. The material may also consist of constituents that degrade by different mechanisms with a different response to temperature or concentration changes. It is therefore desirable to characterise and model the response on constituent level to environmental and mechanical loads. This would enable development a fully flexible method that may make use of micromechanics to calculate the response on an arbitrary geometry to arbitrary loads. Micromechanical testing offers a possibility to characterise the behaviour of the constituents in a composite and together with micromechanical models can then the strength evolution of a macroscopic laminate be assessed. Micromechanical test methods combined with micromechanical modelling offer several potential advantages for accelerated testing:

1) The constituents are isotropic (glass fibres) which significantly reduce the test matrix as compared to a complete characterisation of an orthotropic laminate.
2) Small dimensions minimise the time during which transient conditions are present and speeds up the condition time.
3) There is a possibility to separate solvent transport and ageing to different time scales by tailoring of the specimens (e.g. very small thicknesses).
4) Different degradation mechanisms and rates in the constituents (fibre, resin and interface) may be captured and quantitatively modelled

So far little work has been published on accelerated micromechanical testing for the purpose of quantitative modelling; one example is though Schutte *et al*. [3-6]. The single fibre fragmentation (SFF) test was selected as micromechanical test method for this work. The tests may provide data both on how fibre strength parameters as well as interface strength evolve with time in a certain environment. Both of these properties are important parameters that control the longitudinal tensile strength of composite laminates. The present paper presents an effort to modify the SFF method to fit the purpose of accelerated ageing tests. A set of data deduced for one material combination is presented and compared with previously published results and results obtained with different test methods. The paper is concluded with a discussion about lessons learned from the present work and a judgement of the feasibility of the SFF and the modified SFF test for ageing studies.

2. EXPERIMENTS

E-glass fibres extracted from the uniaxial fabric L600-E10 from Devold AMT is used in all experiments. The glass fibres in this fabric are Owens Corning A111 an Advantex glass fibre (boron free glass). For the SFF test the fibres were embedded in the Norpol Dion 9500-501 Rubber modified epoxy vinylester resin from Reichold with 2% Norpol peroxide No. 11 (MEKP) as initiator.

Very thin, ~0.5 mm, samples containing one single fibre extracted from bundles of the L600 fabric were manufactured by moulding between two glass plates. One needs to be aware that when taking out one fibre from a bundle the weakest fibres are sorted out since fibres already broken or broken at the extraction is thrown away. The thickness was chosen after sorption tests on pure matrix. The tests demonstrated that a 0.5 mm thickness gives time to saturation in water less than 48h, which is judged as sufficiently short to assume negligible influence of the sorption time and thus enables separation of the time scales for sorption and ageing. Thinner specimens also caused problems with wrinkling due to the moisture induced swelling of the resin. After demoulding, cutting to appropriate specimen dimensions and a 4 h postcure at 60ºC the samples were immersed in de-mineralized water for conditioning and ageing. In this study results are presented for 14days, 30days (one month) and 230 days ageing at room temperature (RT). The prepared specimen formation is visualised in Figure 1.

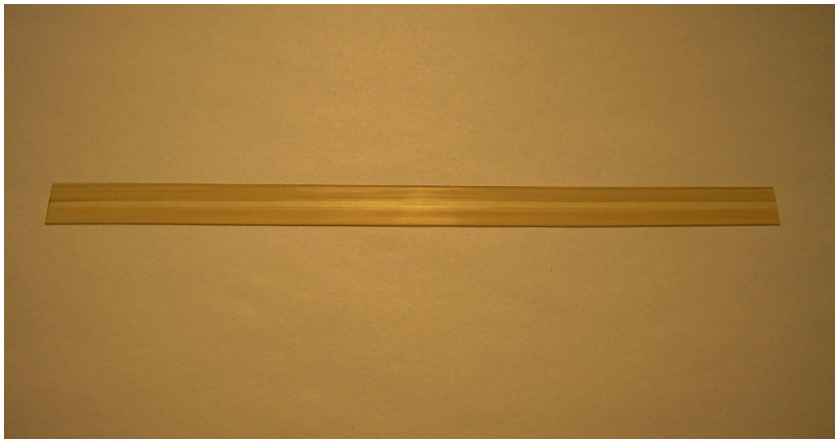

Figure 1. Specimen formation after demoulding and cutting.

After ageing the specimens was removed from the water bath and cleaned. To prepare the SFF specimens an additional layer of neat resin was cast on each side of the thin aged single fibre samples by placing the samples in the middle of two glass plates, 2 mm apart, and pouring resin in the cavity. Individual specimens were then cut out from this thicker plate and post cured. Each specimen was polished to final dimensions to remove any edge flaws that would ruin the test. The final step in the sample preparation was bonding of end tabs. These steps gave SFF specimens of traditional dimensions (100 x 6 x 2 mm^3). The overmoulding step was necessary since SFF tests on 0.5 mm specimens result in premature failure of the whole specimen as soon as the first fibre fracture occurs and no experimental data could be achieved. Tests without tabs where also made but the clamping force, different clamping forces was tested, generated stress concentration and a premature failure close to the tab occurred. The final specimen formation is visualised in Figure 2.

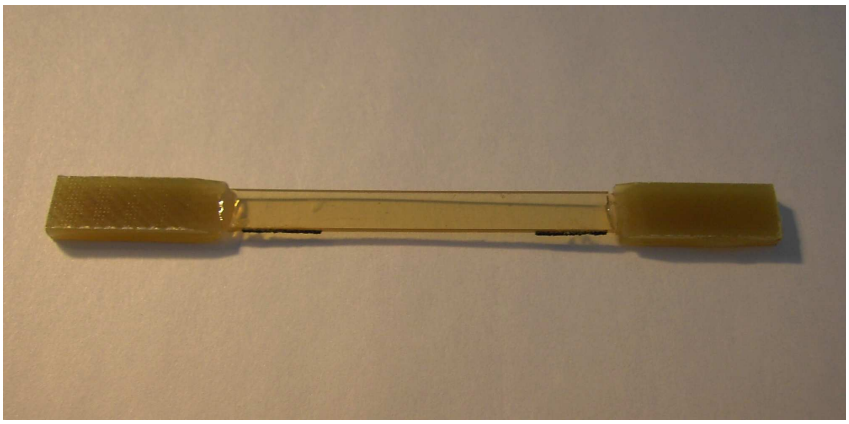

Figure 2. Final specimen formation after overmoulding, cutting, polishing and bonding of end tabs.

A similar overmoulding method was used by Jacques and Favre [7] to facilitate SFF tests with brittle resin systems. The manufacturing process is a very tedious work and introduces a number of steps which all are sensitive to operator skill and experience.

During testing it was observed that only a few specimens got fibre fragmentations and that not all of them reached fragmentation saturation. This is however known to be one of the major draw-backs of the single fibre fragmentation method.

3. Models

The SFF test and the data reduction were performed according to the procedure described in [8]. The two parameter Weibull distribution according to equation (1) has been used for the calculations.

$$P(\sigma) = 1 - \exp\left(-\frac{L}{L_0}\left(\frac{\sigma}{\sigma_0}\right)^{\beta} \right) \tag{1}$$

where σ_0 is the scale parameter associated with the characteristic length L_0, L the length for the measurements and where β is the shape parameter. The interfacial shear strength calculates from

$$\tau = \frac{\sigma_f r}{L_c} \tag{2}$$

where σ_f is the average tensile strength (average elongation/fibre Young's modulus), r is the fibre radius and L_c is critical length calculated from

$$L_c = \frac{4}{3}\frac{L}{number\ of\ \ fibre\ \ fragments} \tag{3}$$

The average fibre strength depends on the fibre length as

$$\langle\sigma\rangle = \sigma_0\left(\frac{L}{L_0}\right)^{-1/\beta}\Gamma\left(1+\frac{1}{\beta}\right) \tag{4}$$

where Γ is the gamma function.

4. RESULTS AND DISCUSSION

Figure 3-6 presents the Weibull scale and shape parameter, interfacial shear strength and the average fibre strength and how these parameters evolve over time. The specimens have been submerged in demineralised water at room temperature for different times. Eight specimens were tested at each time, three to five of those specimen survived to saturation or gave enough fragmentation for analysis. This gives a survival fraction of 38 – 63 %. A survival fraction this low indicates that a very large number of coupons have to bee tested to get statistical security, manufacturing of a large amount of specimen is time consuming as mention before.

The first observation is that there is a large data scatter. The large scatter in data appears to be a common problem for this type of testing and also other authors reports a scatter of similar magnitude, e.g. [5,8]. The large data scatter means that the interpretation becomes uncertain and large datasets will be required to quantify small changes over time. Note that the points placed on the y-axis are the references at zero time. One possible interpretation of the results is that all the properties studied, fibre scale and shape parameter, interfacial shear strength and average fibre strength is constant and do not change within the timeframe studied. However, Schultheisz *et al.* [5] found that for E-glass/epoxy aged for up to half a year at 75°C all properties show a clear reduction with time. They also chose to interpret their 25°C data as if there is a property reduction in time, even though the trend in the actual test data is not very clear. Some of the data scatter can probably be explained by the uncertainty in the method. It should also be pointed out that the present study concern boron free fibres

(Advantex fibres) that are known to be less sensitive to ageing in humid environments as compared to conventional E-glass fibres [9]. Hence we expect to observe a lower degradation rate than in previous studies that considered conventional E-glass fibres.

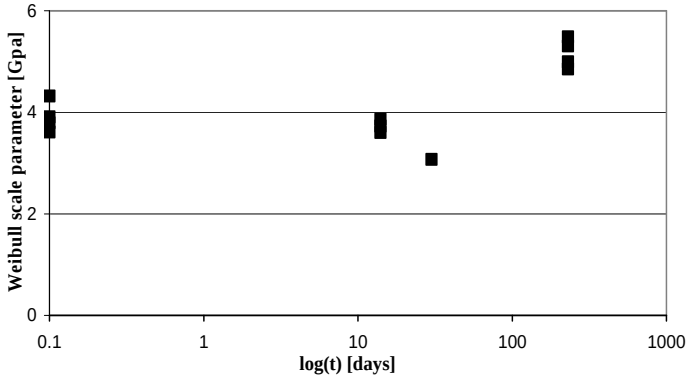

Figure 3. The change in Weibull scale parameter, σ_0,. Observe that the points on the y-axis are the references at zero time.

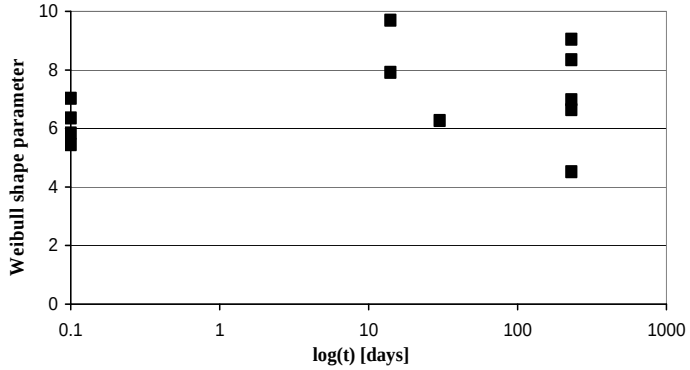

Figure 4. The change in Weibull shape parameter, β. Observe that the points on the y-axis are the references at zero time.

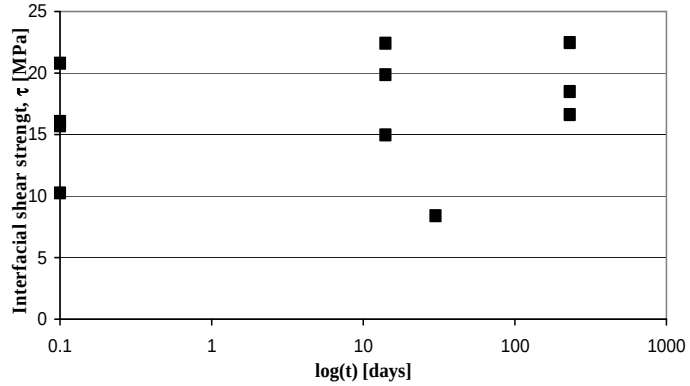

Figure 5. Interfacial shear strength over time. Observe that the points on the y-axis are the references at zero time.

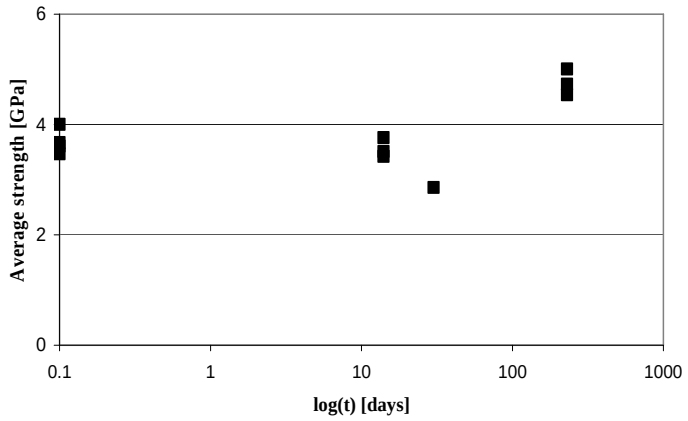

Figure 6. Average fibre strength over time. Observe that the points on the y-axis are the references at zero time.

5. CONCLUSIONS

The single fibre fragmentation test (micromechanical test) method has been evaluated for accelerated testing of environmental degradation of fibre and interphase properties. The ageing environment evaluated is demineralised water at room temperature. The single fibre fragmentation specimen preparation includes extraction of a single fibre from a bundle. This extraction is in favour for strong fibres in the bundle when weak fibres already will be broken or breaks during handling. This implies that tests will only be conducted on strong fibres in the bundles. The specimen preparation method for single fibre fragmentation has been modified in this work with a two-step casting procedure to enable fast conditioning and aging under known conditions. The modified method includes additional steps and thereby additional handling and adding insecurity at each step. The modified single fibre fragmentation specimens do not include the weakest fibres and introduce additional uncertainty of the method resulting in a very low survival fraction of the specimens.

The presently available test results on fibres extracted from a commercial E-glass non-crimp fabric and aged in demineralised water suggest that the fibre strength distribution does not change significantly at the conditions and within the timeframe used in the present study. The interpretation does however suffer from large data scatter, a scatter that seems to be inherent to the test method itself (in view of the similar scatter obtained by other authors). Additional studies with higher ageing temperatures and for longer ageing times are necessary before safe conclusions can be made. Especially the time evolution of the Weibull shape parameter for the fibre strength is of great interest, since a constant shape parameter will be a large simplification of the micromechanical calculations required to translate the micromechanical test results into property evolution of macroscopic laminates and structures. It proved however impossible to obtain such test data despite significant experimental efforts due to brittle failure of the whole test specimen before any significant fragmentation occurred.

The time consuming specimen preparation, documented large data scatter of the single fibre fragmentation test and the experimental problems with premature failure of aged and overmoulded specimens makes this method, in this form, unsuitable for accelerated testing. This conclusion is strengthened by the fact that to obtain statistical certainty a large number of specimens are needed which means that a huge amount of

time and costs have to be spent to get any results that can be used with confidence for life assessment exercises.

ACKNOWLEDGEMENTS

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