

SCALING EFFECTS OF GLASS FIBER REINFORCED EPOXY IN FATIGUE

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Abstract

The fatigue performance of a composite material is influenced by many factors including the loading mode, geometry, and process-induced microstructure. New work in resin transfer molding (RTM) has yielded methods to produce thick-sectioned composites with per-piece cycle times below 1 minute. The materials produced with RTM offer a distinct competitive advantage over prepreg, but carry an unknown fatigue performance. A baseline study is conducted with a commercial glass textile and a commercial snap-cure epoxy to document the fatigue performance of this material from the RTM process. Several length scales from a coupon up to full component level are tested and evaluated to produce complete S-N curves up to 10^6 cycles. SEM and μ CT testing are used to deduce the microstructure and fatigue failure modes. The results of this work have direct impact toward the material selection and design of composite structures in fatigue, namely automotive leaf spring suspension.

Background

Composite leaf springs have been in commercial use for quite some time. The Chevrolet Corvette has used a transverse leaf spring since 1981 and the Mercedes Sprinter since 2005. In both these examples, the fiber reinforced plastic (FRP) has been an epoxy/glass prepreg. The viability of this material has been well-validated in these suspension applications over the many years of operation. Now, with the advent of newer processing methods, resin transfer molding presents an opportunity to further reduce the manufacturing cycle time limitations of prepreg. RTM, however, requires slightly different material inputs, changes to the process, and thus needs to be validated.

Filament winding is another method to produce large, thick-sectioned composite components, and takes advantage of lower materials costs similar to RTM. Here, the cycle time is longer still than prepreg so this process tends to be reserved for lower production volume applications.

In addition to understanding the performance of leaf spring elements fabricated using resin transfer molding, a secondary challenge is to understand how that performance scales with element thickness since it is desirable to test small samples that are easy to manufacture. Not addressed herein but also important is to generate data that can validate the failure criteria of the material models used to design thick-sectioned composites in fatigue applications.

The fatigue performance of a composite material is influenced by factors including part geometry and mode of loading. Part geometry results in different material defects/internal features, such as fiber waviness (undulation) [1-3] and ply drops [4-5] within composite structures. Also, different fatigue loading modes have been investigated, for example tension [6], compression [7], impact [8] and flexure [9]. The flexural loading mode is of particular interest as it is similar to the loading mode of the leaf springs in service. Therefore, the technological

challenges are largely centered on the understanding of the relationship between flexure fatigue performance of glass fiber/epoxy composite materials and their microstructure, at both coupon and component levels.

Previous work with a new epoxy matrix for high-volume leaf spring production with HPRTM demonstrated excellent chemical resistance and complete life cycle assessment [10]. Recently, the economics of resin transfer molding have also been presented [11]. The present work aims to complete the performance validation of the composite system at several material thicknesses.

Experimental Procedure

Sample Fabrication

Coupon-level panels were fabricated at the Fraunhofer Project Center in London, Ontario using their Krauss-Maffei Rimstar 8/4/8 HPRTM unit. Panels, 5.5 mm thick, were created from seven layers of Saertex U-E-1200 glass fabric, using an epoxy-compatible sizing, and without binder. The system was Hexion's EPIKOTE™ Resin TRAC 06150 with EPIKURE™ Curing Agent TRAC 06150 cured for 5 minutes at 120 °C.

Component-sized samples were fabricated at Hexion's Technology Research and Application Center in Duisburg Germany with their Cannon HPRTM system and a specially designed 30mm-thick-section HPRTM tool. The same textile and resin system were used, however, the epoxy was cured for 10 minutes at 100 °C and was followed by a 30 minute post-cure to ensure both methods of fabrication resulted in identical materials having reached the same degree of cure. In both cases, samples were manufactured under vacuum to the target fiber volume fraction in the range of 55-59% using industry best practices.

Fatigue Testing

Fatigue testing was performed at the University of Western Ontario using an Instron 8804 servo-hydraulic load frame rated for up to 500kN. The load frame was outfitted with grips and a load cell rated for up to 250kN static and 236kN dynamic load. Aluminum I-beams, bolted to the grips, were used to support the steel testing fixture, as pictured in Figure 1. In this way, the overall test fixture stiffness far exceeded the material in the test setup but remained adjustable for the many different tests conducted. The specific steel loading cones were adjusted based on the size of the sample being tested both in terms of placement and radius.



Figure 1 – Fatigue testing setup for thick component testing

The testing machine actuator was capable of up to a 300mm stroke. The system could be operated in either load- or displacement-controlled cyclic testing, the former selected for the present work. The test protocol was based on ASTM D7774 and ASTM D790, but used samples with a constant (approximate) dimensional ratio of 1:3:12 thickness:width:span.

The 5.5 mm thick samples were all tested as fabricated, but many of the thick 30mm components were machined to thicknesses of 11mm or 22mm (multiples of the base thickness) and corresponding widths according to the target sample dimension ratio. The possibility of the induced damage from the machined surface affecting the test outcome was partially mitigated by always testing with the machined surface 'up' so that the tension side of the test specimen was always a pristine surface. The machining was also conducted with a slow finishing pass and sample polish so as to minimize this influence further.

Initial test screening indicated no observable heat generated at up to 10Hz testing rate after 50,000 cycles, but the rapid actuation at high loads tended to cause issues with the hydraulic cooling system. Thus, for cyclic load testing below 20kN, a 7Hz testing rate was selected to minimize the testing time, while 2 Hz was selected for tests at higher load. The data collection rate was set at 50 times the testing rate so as to always capture substantive data in each testing cycle.

To prevent undesired sample motion, small PTFE tabs on the lower supports were used to prevent the samples from rotating in their plane. Additionally, small pieces of flexible double sided tape prevented the samples from shifting along their long axis.

The loading cone radius was adjusted based on the sample thickness to yield a targeted tensile failure mode. For the 5.5 mm thick samples, a bare 20mm diameter steel loading cone was used. For all other sample thicknesses, a steel insert with a lower curvature 240mm in diameter was placed between the main cone and was separated from the sample with a glass reinforced nylon strip (Figure 2).

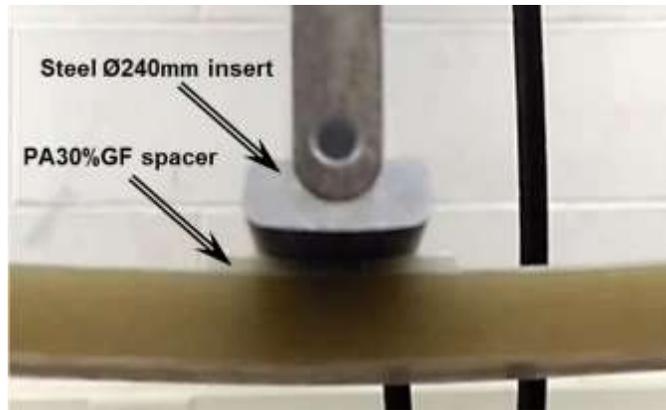


Figure 2 – Loading cone setup for thick sample fatigue testing

Results and Discussion

The quasi-static mechanical properties as measured for this material system are collected in Table 1.

Table 1 – Quasi-static properties of the glass reinforced epoxy

Parameter	Standard	Unit	Value
Fiber Volume Fraction	ISO 7822	%	56.6
Density	*calculated	g/cm ³	1.97
T _g (DSC Peak)	ISO 11357	°C	125
Flexural Modulus	ISO 14125	GPa	49.6
Flexural Strength	ISO 14125	MPa	1541
Interlaminar Shear Strength	ISO 14130	MPa	68.4

The quasi-static sample testing identified issues with the failure mode as a function of thickness. The bare loading cone on the machined sample surfaces of the thicker specimens almost always resulted in a compression failure (Figure 3) well below the expected shear strength of the sample. This was alleviated by the aforementioned steel insert, which increased the loading cone diameter as well as a reinforced polymer strip to help with the surface compliance. The polymer strip helped mitigate early compressive failure from imperfectly flat samples. As well, this helped reduce issues with the sample moving within the fixture during high cycle fatigue testing.

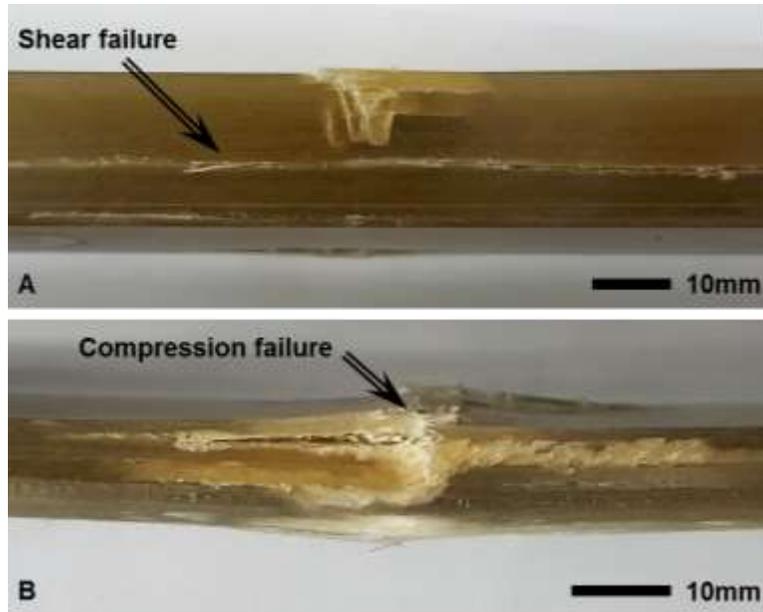


Figure 3 – Undesirable failure modes with different beam thicknesses a) 22mm b) 11mm

The thin samples always exhibited a tensile failure mode of the lower glass textile layers. Even with the loading cone adjustment to eliminate the early onset compression failure, as the sample thickness increased, the failure mode shifted toward a shear failure. The observed effect is a linear reduction to the flexure strength with respect to sample thickness as in Figure 4.

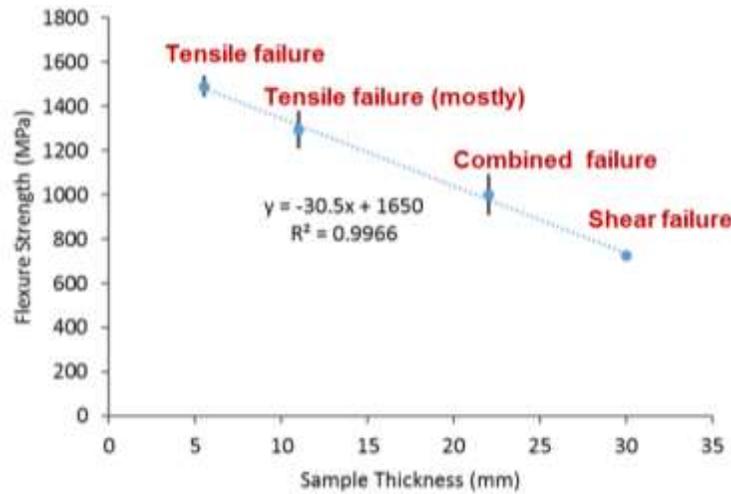


Figure 4 – Flexure strength and failure mode as a function of sample thickness for a fixed ratio, quasi-static 3-point bend test

This result is not expected. With carefully scaled samples and test setup, the flexure strength is expected to be a flat, horizontal line, effectively measuring the sample material modulus. Since coupons have shown better performance than components with the same fiber, matrix, and fiber volume fraction, it must be one or both of the presence of defects or stress state that causes this

decrease in observed strength of the tested components.

Micro CT through-thickness scans of different sample thicknesses were inconclusive and did not reveal the presence of any noticeable defects (inclusions, porosity, etc.) at a scan resolution of 20 μ m. The resin-rich layer between textile lamina in the thin samples was notably thicker, but this should have only served to degrade the thin sample strength rather than the components.

M-CT side-profile views of two fractures are presented in Figure 5. In both samples, the fiber is seen as well-aligned and impregnated with the epoxy without any obvious material or manufacturing defects. The thin sample shows a longer crack with sub-fractures within the observable fiber bundles. Direct imaging of the respective fracture surfaces did not yield any distinction between the fracture surfaces, nor were there present any unexpected features.

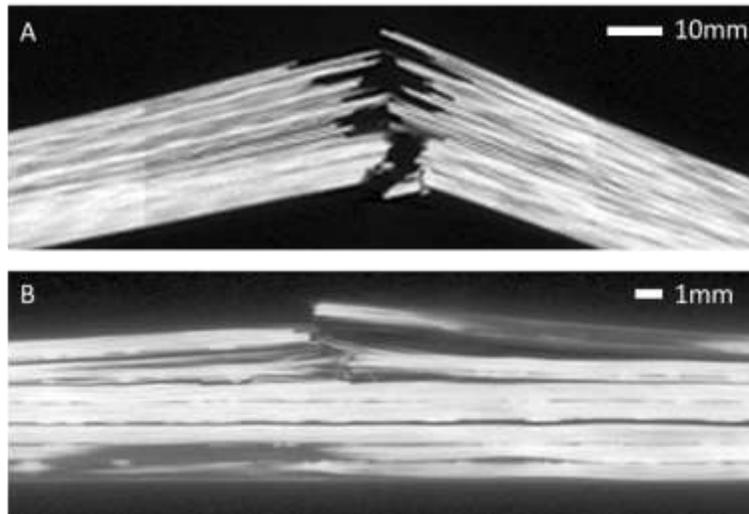


Figure 5 – M-CT side-profile fracture images of A) 22mm thick sample B) 5.5mm thick sample

However, SEM imaging of a polished end-on crack did provide some insight into the fracture morphology. In the thin samples, the cracks were predominately planar in nature, occurring at the resin-rich layer between lamina. In the thick samples, as presented in Figure 6, the crack path is seen to travel between several lamina with indifference to fiber bundles and resin-rich sections. In order for a crack to travel away from the plane of highest stress, it must be to reduce its total energy. It is then implied that multiple smaller cracks were formed and growing simultaneously before being linked together. Given that many of the neat-resin facing cracks are horizontal, while the fiber bundle cracks are vertical or on a roughly 45 degree angle, the cracks are interpreted to grow first between each lamina, then link up through the fiber bundles. Thus, the failure initiation is expected to be a function of the fiber-matrix interfacial shear strength. Again, the SEM imaging yielded no obvious defects in the microstructure.

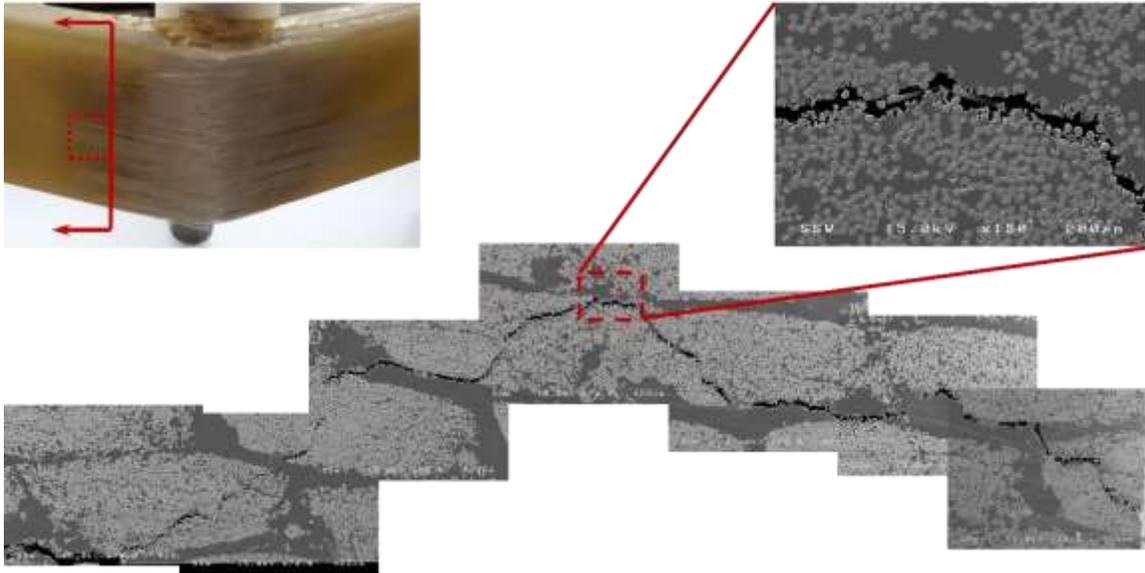


Figure 6 – SEM composition of images taken end-on from a single crack bundle from an 11mm thick failed sample

To see when the crack begins and how it affects the progressive failure of a GFRP material in fatigue, loop analysis was conducted; one such dataset is summarized in Figure 7. Hysteresis curves spaced equally on a logarithmic scale are plotted with two curves prior to failure. In this example, a 22mm thick sample under 34kN cyclic load has consistent load-displacement behaviour until prior to failure over the final 6000 cycles.

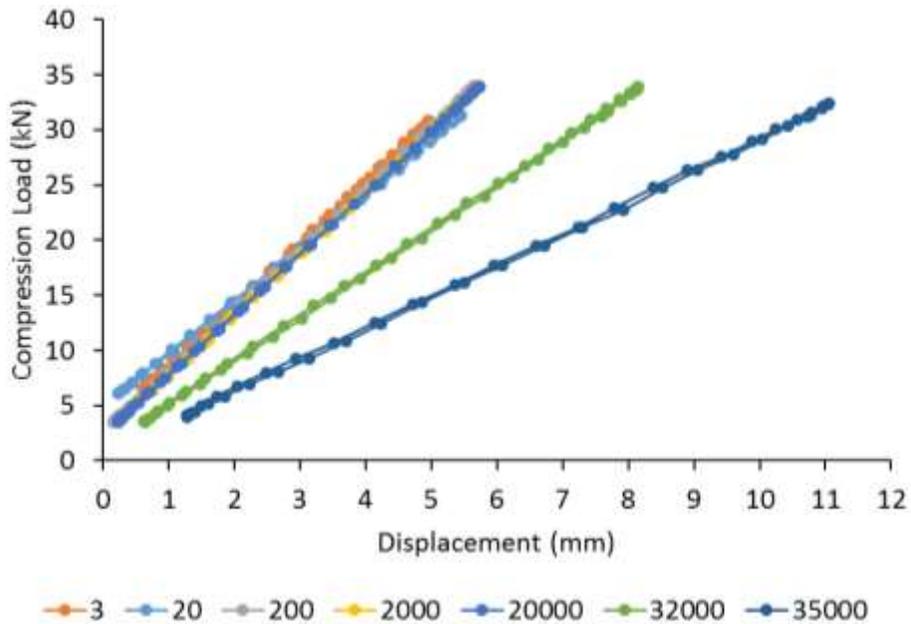


Figure 7 – Loop study 34kN, 2Hz, 22mm thick sample.

Individual curves before any damage onset have no statistically measurable energy loss indicating the previous assertion of no heat generation during testing is likely true. Also, given the

constant load-displacement slope indicates an elastic sample response. After a first fiber breakage is recorded, the overall low energy loss means that the crack growth is very slow and the load-carrying capacity is maintained for some time. Since the test is load-controlled, as the sample degrades with each cycle, the testing stroke is increased to reach the programmed load and eventually the machine can no longer keep up with the test parameters; the final ~5% of total cycles generally fail to reach the target load for a given test setup. Across all the tested samples, initial failure appears consistently at ~85% of the fatigue life.

The complete stress-cycles data is presented in Figure 8 for both the 5.5mm and 22mm thick samples. In all thicknesses tested, the fatigue curves presented as logarithmically related, though there is not sufficient data to form a statistical assertion. The observed fatigue endurance limit for this material, here defined as the stress below which the sample will reach 10^6 , is approximately 270 MPa. For a given number of fatigue cycles, the 22mm sample averages 75% of the strength of thin, 5.5mm sample.

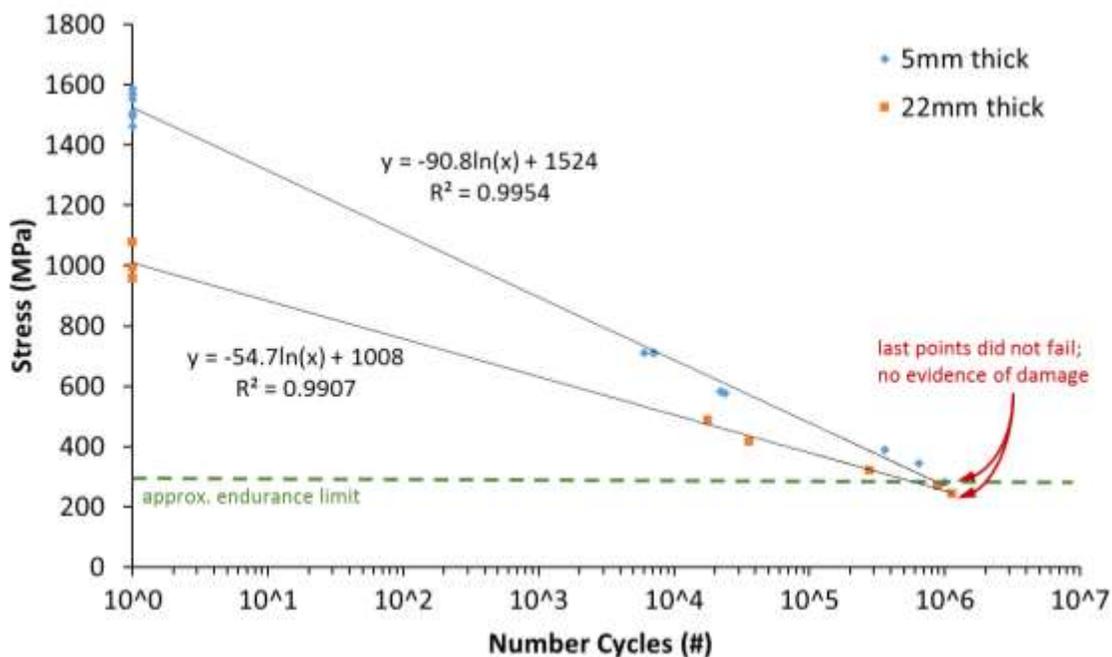


Figure 8 – Overall SN fatigue curves for the 5.5mm and 22mm samples with overlay endurance limit. Note the final points on both curves did not fail and the test were stopped around 10^6 cycles.

With the lack of observed defects and the large, consistent difference in fatigue stress, the stress state within each sample is suspected of being non-uniformly distributed. This non-uniform stress would be the result of non-linear load transfer layer-to layer in the microstructure. Until further testing can be conducted to validate the results, a key learning is that coupon fatigue testing does not accurately reflect component-level performance. However, despite the difference in performance, this consistent result indicates that the small coupons can be used to predict the performance of the components.

Using coupons to accelerate the development cycle, further optimization in the fabrics and process parameters have already yielded substantially increased fatigue performance (the data can be privately requested). This performance increase is now in the process of being validated at the component level and will be compared to the predicted performance using the results of

this study.

Anecdotally, by studying the load decrease at the end of each fatigue test, it is hypothesized that Miner's rule of cumulative damage applies to glass fiber reinforced epoxy and required further work to draw any conclusion.

Summary

A glass fiber reinforced epoxy composite, suitable for automotive fatigue applications, is tested in flexure at multiple length scales with consistent sample dimensional ratios. Many of the practical aspects of testing are presented along with novel failure analysis. The fatigue curves for two different sample thicknesses were generated up to 10^6 cycles and the fatigue performance is shown to be a function of the sample thickness. This unexpected result is thought to be linked to the internal state of stress as evidenced from the lack of observed defects and crack morphology.

The next steps will repeat some of this testing using more sophisticated measurement techniques to track the sample internal stress and better understand the measured performance difference in the scaled sample testing. Work is also being done to more closely align the test method to real-world conditions to replicate the stress state and load transfer as in automotive suspension elements.

Future work aims to compare materials across the major processing methods: prepreg compression molding, filament winding, and resin transfer molding.

Acknowledgements

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