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FATIGUE ANALYSIS AND RESIDUAL STRENGTH EVOLUTION OF HIGH-PERFORMANCE UNIDIRECTIONAL CARBON-FIBRE COMPOSITES FOR HYDROGEN STORAGE APPLICATIONS: CHARACTERISATION USING SPLIT-DISK RING TESTS

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Abstract

Facing climate change and pollution challenges, cleaner energy sources are essential, making hydrogen a key focus due to its clean, versatile nature. The work presented in this paper takes place in the frame of the CAVENDISH project of the European Union and focuses on the mechanical assessment of a latest-generation high-performance carbon-fibre composite material for its application in hydrogen storage in the aviation industry. The adopted study approach consists in the evaluation of the residual strength of the material after a fatigue history. First, the material in ring form is loaded in tension-tension fatigue using the split disk method until a defined number of cycles. Following the fatigue, a quasi-static tensile test is carried out on the ring specimen until failure. This method allows the comparison of the apparent hoop strength before and after its fatigue life. An increase of 11% is observed over the tested number of fatigue cycles. This effect is attributed to a change in the multiaxial stress ratio in the gauge section, resulting from cyclic-loading-induced geometric evolution (ovalisation) of the rings. Although this increase in apparent hoop strength does not necessarily correspond to an actual increase in intrinsic material strength, it is nevertheless highly reassuring regarding the fatigue life of the material. Regarding the stiffness, non-destructive vibration tests show a 5% loss in the elastic hoop modulus of the tested rings after fatigue. Further mechanical testing and data analysis is underway to form a full view of the material's behaviour.

Keywords: *Carbon fibre; fatigue; residual strength; mechanical testing; filament winding.*

1 Introduction

In the context of climate change and pollution issues, it has become imperative to switch to cleaner energy sources. This transition has spurred significant scientific research across various fields, with hydrogen emerging as a key area of focus in recent decades. Hydrogen is a promising energy source due to its clean and versatile nature [1]. This transition involves not only a shift in energy sources but also requires the development and testing of new materials and technologies to ensure their viability and sustainability in various hydrogen-related applications.

Hydrogen storage requires specialised tanks suited to different pressure and temperature ranges. The evolution of pressure vessel technology from Type I to Type IV highlights the industry's search for lighter and more durable storage solutions. Type IV vessels reinforced with composite materials such as carbon fibre have become essential in applications such as aviation, to improve the efficiency of hydrogen storage whilst maintaining structural integrity.

The growing adoption of hydrogen technologies is accompanied by the need for reliable and robust materials capable of withstanding the unique operating conditions of hydrogen. Composite materials, which are lightweight and strong, are excellent candidates for hydrogen storage and transport infrastructure. Carbon fibre-based composites, in particular, are commonly used in high-pressure hydrogen storage tanks due to their exceptional mechanical properties. However, it is essential to ensure the safety aspects of using these high-performance materials.

Mechanical testing plays a vital role in assessing the durability and safety of carbon fibre-based composites used in hydrogen applications, and in ensuring their ability to withstand the specific challenges of extreme pressures and temperatures posed by hydrogen storage. Various testing methods, coupled with non-destructive testing techniques, are essential for evaluating the performance of these composites under different potential service conditions.

This article presents the work carried out as part of the CAVENDISH (Clean Aviation) project with the aim of predicting the durability of a UD T1100/UF3376 composite material, in particular the residual strength after fatigue loading followed by a quasi-static test. A statistical approach is then used as the basis for assessing the risks associated with the material's fatigue strength, with a focus on how the residual strength evolves as a function of the number of cycles undergone.

2 Materials and methods

This study focuses on a high-performance carbon-fibre-reinforced composite material of the latest generation, the T1100S/UF3376 from TCR Composites. The data-sheet provided by Toray, the fibre manufacturer, gives the tensile strength of the fibre itself at 7,000 MPa, and its elastic modulus at 324 GPa. The data-sheet of the UF3376 resin gives its tensile strength at 97.2 MPa and its modulus at 3.3 GPa. These values result in a theoretical tensile strength of the composite material at 4,226 MPa and a theoretical longitudinal modulus at 195 GPa, as per the rule of mixtures, considering a fibre volumetric fraction of approximately 60%. The Toray data-sheet for a T1100-reinforced composite with a 3960 epoxy resin system at 60% of fibre volumetric fraction, however, gives the tensile strength of UD flat rectangular coupons at 3,444 MPa, falling at 81.5% of the theoretical value.

Tubes of an internal diameter of 120 mm and a length of about 500 mm are manufactured by filament winding using the pre-impregnated material. The pre-impregnated tow comprises 12K carbon fibres and has a width of approximately 4 mm and a thickness of about 0.3 mm. Four layers are wound in the circumferential direction with a winding tension of 45 N. The thickness of the tubes is about 1 mm. The wound tubes are cured at 121 °C during 4 h according to the recommendations of the supplier. Individual ring specimens are cut out of the manufactured tubes using a diamond saw-blade at a width of 10 mm, and their edges are sanded. The gauge cross-sectional area is the sum of 2 cross-sectional areas measured at 2 points of the ring diametrically opposed. For each gauge section, 3 measurements are made and their average is considered for the calculation of the cross-sectional area. Figure 1 shows a cross-section slice of a ring coupon obtained by X-ray micro-tomography (RX Solutions). The fibre distribution throughout along the thickness is not uniform, the inner part of the ring (orange arrow) having a denser fibre distribution than the outer part (blue arrow). This is likely linked to the manufacturing process in 2 ways, namely the winding tension and the cure cycle, where the wound carbon fibres heat up and contract on the mould, pushing the still-liquid resin outwards.

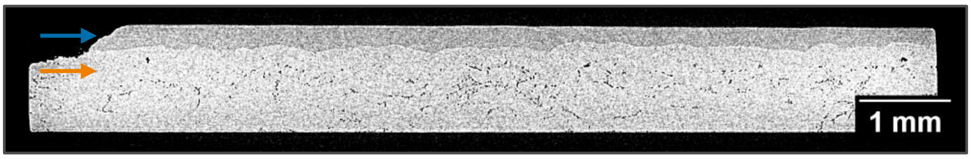

Figure 1. Cross-sectional X-ray micro-tomography slice of a cut ring coupon with the upper left corner broken off during cutting, the orange arrow pointing at the fibre-dense area and the blue arrow at the resin-rich area.

The apparent quasi-static hoop strength of the material is determined on the basis of split-disk tensile tests on rings based on the ASTM D 2290 method [2] using an INSTRON 8501 hydraulic tensile testing machine fitted with a 100 kN load cell. However, the ring coupons are not notched to avoid longitudinal splitting during the test. The tests are performed at a quasi-static crosshead speed of 3 mm/min. A total of 7 rings are tested to determine the apparent quasi-static hoop strength. The experimental setup is shown in Figure 2 with a CAD model for better reading.

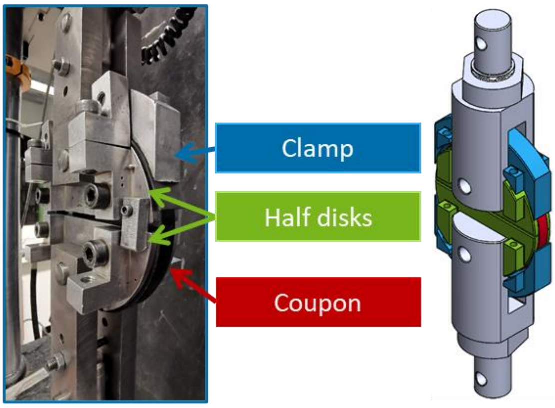

Figure 2. Split-disk experimental setup and 3D CAD model for better reading.

A fatigue test campaign is conducted on rings at ambient temperature to simulate the service life of a hydrogen storage tank. An INSTRON 8501 hydraulic tensile testing machine fitted with a 100 kN load cell is used for the tests. In total, 35 specimens are tested in fatigue. A thermocouple registers the temperature at the outer surface of the ring during the fatigue test. Force-controlled tests subject the rings to a tensile load consisting of an initial cycle at 70% of the average value of the material’s apparent quasi-static hoop strength. The first cycle is then followed by a number of cycles at 55% of this average value at a frequency of 3 Hz and a ratio R of 0.1, ranging from 1,000 to 50,000 cycles for different specimens, as shown in Figure 3.a. The applied maximum and minimum forces are calculated prior to testing, taking into account the nominal cross-sectional area of the rings to be tested in the gauge zones.

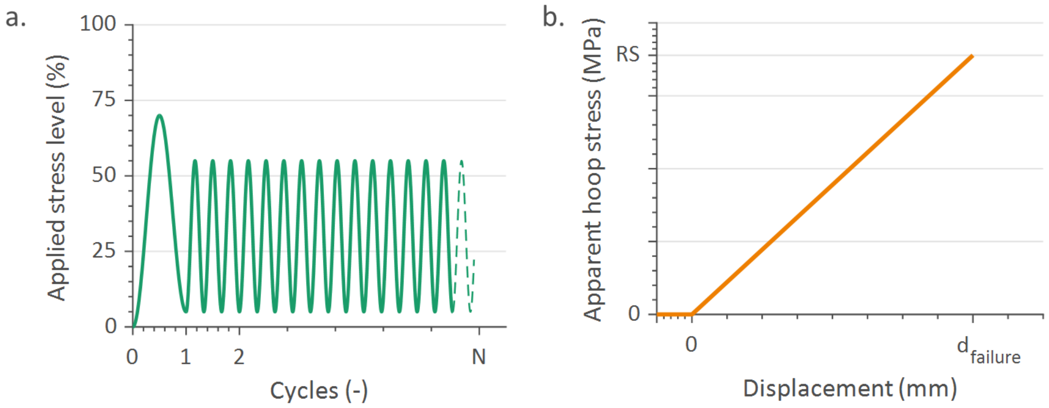

Figure 3. Fatigue test protocol (a) followed by quasi-static residual strength test (b).

Following the fatigue test, the ring is removed from the experimental setup and left at ambient temperature during one week. It is then loaded to failure in accordance with ASTM D 2290 (Figure 3.b) in order to evaluate the evolution of its residual strength as a function of the number of fatigue cycles undergone. A statistical significance test of the type ANOVA, or Analysis of Variance, is performed to compare the results of residual strength tests done on fatigue coupons to those of the quasi-static tensile strength tests done on pristine coupons.

One ring specimen is also tested using a non-destructive contact-free inverse vibrational method before and after fatigue to determine the evolution of the hoop stiffness based on finite element model updating and 3D velocity fields measurements (FEMU-3DVF) [3]. The test consists in hanging the ring using thin cables to simulate nearly free-free boundary conditions, and soliciting it over a large frequency range using a speaker. A vibrometer (Polytec PSV-QTec) detects the ring's 3D velocity fields response. The test is performed before fatigue at N = 0, at N = 5,000, and at N = 15,000 cycles. The vibrometer data is coupled with a finite-element model in COMSOL to back-calculate the ring's hoop elastic modulus using finite element model updating (FEMU). This model is based on the actual-geometry 3D model of the specimen obtained by X-ray micro-tomography.

3 Results and perspectives

The average apparent quasi-static hoop strength of the rings is measured at 3,001 ± 77 MPa. The coefficient of variation is thus at 2.5%, of a similar magnitude to recent round-robin studies investigating the tensile strength of UD T800-reinforced flat coupons [4]. Compared to the theoretical tensile strength for a flat specimen calculated using the rule of mixtures, the apparent hoop strength obtained experimentally falls at 71% of that value. The discrepancy between the 2 values comes from the dissimilarity of the compared coupons in terms of geometry and the difference in the measured mechanical property, namely the apparent hoop strength in rings in contrast to the longitudinal tensile strength in flat coupons. The tensile strength value of 3,444 MPa reported by Toray and obtained from UD flat rectangular coupons demonstrates the difficulty of realising the full potential of the T1100 fibre through composite-scale testing.

Table 1 compares the apparent hoop strength obtained for the T1100 in this study to the apparent hoop strength of other carbon-fibre-based epoxy-resin composites assessed using the same type of test. The values given in Table 1 are for ring coupons not identical in geometry to the ones tested in this paper. The hoop strength measured using a split-disk test is dependent on the size of the specimen both in diameter and thickness [5-6]. The apparent hoop stress for all the shown cases is measured at 73.65%, on average, of the theoretical value calculated using the rule of mixtures. Nevertheless, the T1100-reinforced material performs better than those with the T700 or the T1000, presenting an increase by 30% and 9% respectively in the apparent hoop tensile strength.

Table 1. Comparison of the apparent quasi-static hoop strength for the T1100-based composite material to other commonly used carbon fibres using the split-disk test (*: calculated from the weight volume fraction with assumptions made; **: arbitrary value chosen for ease of comparison with the rest of the results due to the precise value not being given in the reference work).

Fibre	Fibre volume fraction [%]	Theoretical tensile strength [MPa]	Apparent hoop strength [MPa]	Reference
T1100	60	4,226	3,001	This paper
T700	62.9	3,064	2,071	[7]
T700	60	2,940	2,110	[8]
T700	62*	3,038	2,392	[5]
T700	60**	2,940	2,624	[6]
T1000	60**	3,822	2,755	[6]
M40	60**	2,646	1,934	[6]
M50	60**	2,472	1,913	[6]

In contrast to the results reported in the literature for carbon fibre composite laminated specimens [9-10], the initial results concerning the material's fatigue resistance (Figure 4) show that, on average, the material's apparent quasi-static residual strength after fatigue reaches 3,326 ± 380 MPa. The coefficient of variation after fatigue becomes 11%, due to the damage undertaken by the material during cycling. This amounts to an increase of 11% compared to the apparent strength of the virgin material, regardless of the number of cycles up to 50,000. For instance, after 1,000 cycles, the residual apparent hoop strength ranges from 2,706 MPa to 3,857 MPa. Similar mean values are obtained after 5,000 to 50,000 cycles, with a comparable order of magnitude in strength variation. The self-heating of the coupon during fatigue is registered at +25 °C on the outer surface of the ring.

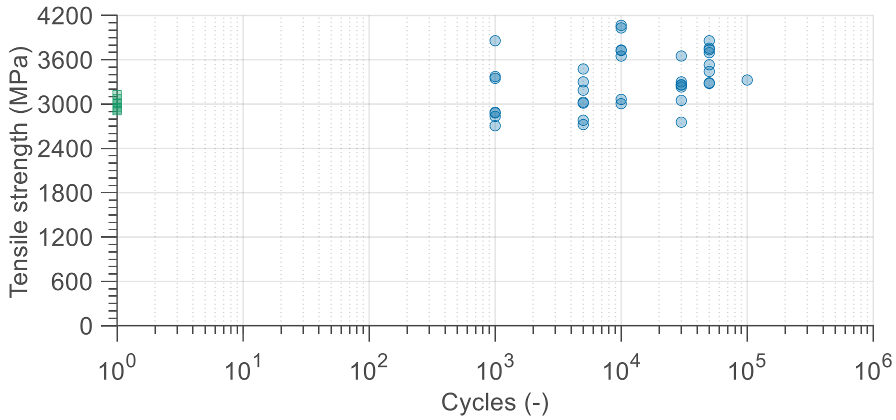

Figure 4. Quasi-static tensile strength and residual strength as a function of the number of fatigue cycles experienced (the different shades of blue are due to individual data-points overlapping).

This increase in the apparent hoop strength of the material after fatigue loading can be explained by the permanent geometric deformation of the tested structure, the ring, into an oval shape under the applied load. Figure 5.a shows the geometric difference between 2 ring specimens stacked, the bottom one after fatigue and the top one before being tested. This change in the geometry, mainly in the radius of curvature of the gauge section, has a strong impact on the ratio of stress multi-axiality. The main proposed hypothesis is that the bending-moment-to-hoop-stress ratio decreases after fatigue as the radius of curvature increases in the gauge section.

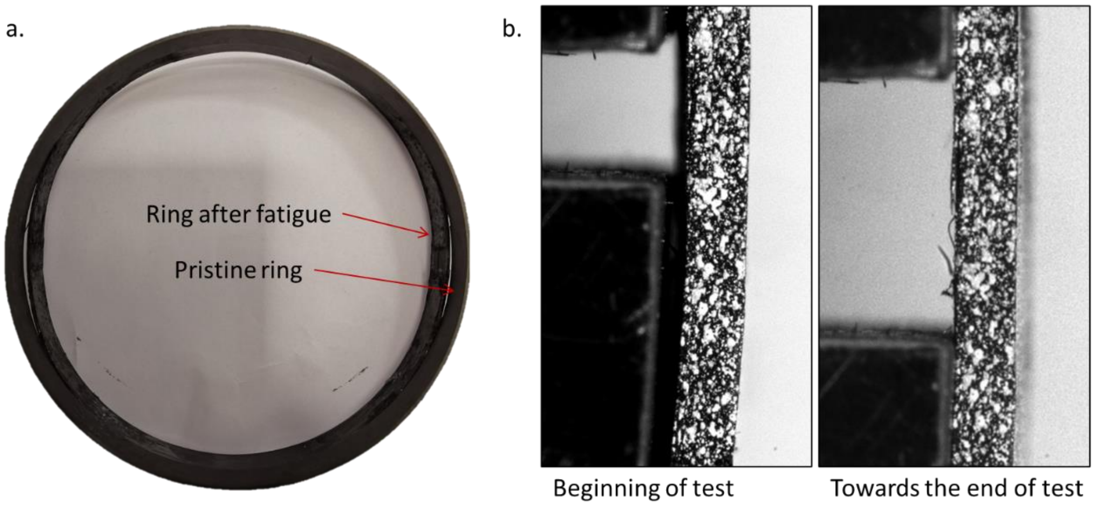

Figure 5. Visual geometry comparison of a ring after fatigue and a pristine ring showing the ovalisation of the former, with a clear difference in the diameter (a), and the thickness view of a ring at the beginning and before failure during a residual strength quasi-static test highlighting the straightening of the gauge section (b).

The statistical significance of this observed difference is assessed across 35 fatigue points and 7 quasi-static points using an ANOVA test; the increase in the ring's mechanical properties following a fatigue load is statistically significant at a 95% confidence level.

An analysis of the test machine data, notably the machine displacement during the fatigue cycling, reveals an apparent stiffening behaviour in the material, as evidenced by a systematic decrease in the amplitude of the machine's displacement (Figure 6.a) for each specimen tested after a few hundred fatigue cycles. With the ovalisation of the rings, the gauge section experiences less bending during load application. The quasi-static tensile plots of rings before and after fatigue also reveal a stiffening of the structure characterised by the increase in the strength-displacement slope and a more linear curve compared to the one before fatigue cycling (Figure 6.b).

However, despite an increase in the ring's apparent stiffness during fatigue cycling, based on measurements of the peak-to-peak amplitudes of the machine's force and displacement signals, non-destructive measurements carried out using FEMU-3DVF show a decrease in the composite's elastic modulus in the fibre direction of approximately 5%. This indicates a moderate level of damage in the specimens at these loading levels.

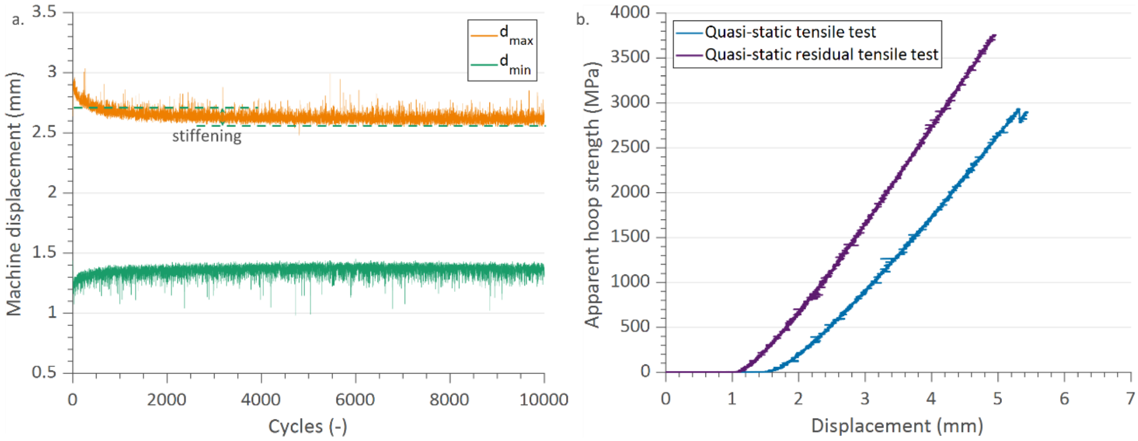

Figure 6. Evolution of the minimum and maximum machine displacements throughout the force-controlled fatigue loading of the material (a) and the comparison of the quasi-static tensile behaviour of a pristine ring and a ring after fatigue life during a residual strength assessment test (b).

Even if the observed 11% improvement in the measured apparent hoop strength of the tested rings does not necessarily reflect an improvement in the material's intrinsic properties – as is discussed subsequently in the FEMU results analysis, it is highly reassuring regarding its fatigue life. The material does not lose its performance after 50,000 cycles of fatigue.

The analysis of the collected data, which is currently underway, should provide a better understanding of the underlying phenomena, such as fibre realignment [11-12] and the potential relaxation of residual stresses, which occur in the material throughout its fatigue life and lead to the apparent stiffening of the test specimen and a significant increase in its apparent hoop tensile strength following fatigue. Finite-element simulation is also used to compare the stress distribution in both ring specimens, the one with a perfectly circular geometry, and the other with an elliptical gauge section due to its ovalisation.

Further tests will be carried out to obtain a satisfactory number of data points on which a statistical approach with a reliable level of certainty can be applied regarding the evolution of the material's apparent residual strength as a function of the number of cycles.

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