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Cryogenic fatigue and stress-strain behavior of a fibre metal laminate

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Abstract

This paper reports on the cryogenic fatigue life of Al 2024 / Stycast 2850 FT composite sandwiches loaded under cyclic strain, as well as on the strength of their constituent materials at 77 K. These Fibre Metal Laminate (FML) specimen serve as a model for an alternative class of cryogenic structural materials that might be used e.g. in downstream LNG applications. FMLs, such as the GLARE ™, are already used in the aeronautic industry, where they provide better damage tolerance, corrosion resistance and lower specific weight. Their cryogenic performance, however, is yet to be understood. Preliminary results show that the metal/filled-epoxy combination presented here withstands repeated cool-down to 77 K. Moreover, its cryogenic fatigue life is at least 20 times longer than at room temperature. These observations are consistent with the measured stress-strain behaviour of the metal and the epoxy, as well as with the shear strength of the bond between them. The Youngs modulus, yield strength and tensile strength of the Stycast 2850 FT roughly double when cooled down to 77 K. In addition to this, the bond strength with the GLARE-type coated Al increases significantly. These preliminary experiments indicate that cryogenic FML are technically feasible.

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Keywords: FML, fatigue, Stycast 2850 FT, cryogenic, LNG;

1. Introduction

Fiber Metal Laminates are used in the aeronautic industry, such as the GLARE™ concept, which is an aluminium reinforced epoxy. FML can have better damage tolerance, corrosion resistance, lower specific weight and higher yield strength compared to their constituent materials (Sinmazçelik et al. (2011), Wu and Yang (2005)). By combining several materials in a sandwich structure, the strong points of each material can be used, while a material's weaker property might be compensated by application of a ply of a different material. This way the strong points of several materials can be combined to achieve an optimal structural material for cryogenic applications. A small increase in

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mechanical properties of GLARE is shown by Hagenbeek (2005), upon cool-down to 216 K. However, their cryogenic performance has yet to be understood.

The intention is to analyse the possibility of using FML as a high-strength low-weight material alternative for e.g. LNG applications. One of the main issues for these materials is the mismatch between the constituents coefficients of thermal expansion, which results in high thermal stresses in these materials upon cool-down. A possible solution to this problem might be the use of alternative metal/epoxy combinations. Stycast 2850 FT is an epoxy that is specifically designed to resist thermal shock and is used succesfully in a number of cryogenic applications, such as potting of superconducting magnet coils. In this investigation, a preliminary series of experiments is carried out to compare the mechanical behaviour of these model specimen at room temperature and at 77 K. The experiments performed in this feasibility study are fatigue measurements, in which bi-layer sandwich specimen are subjected to cyclic bending strain; axial stress-strain measurements and experiments on the shear strength of double lap joints.

2. Experimental Details

2.1. Fatigue life

For the measurements of the fatigue life, bi-layer metal/epoxy specimen are loaded under cyclic bending, effectively probing delamination behaviour under mixed mode I/II loading. A DC motor mounted on the top of the cryostat insert is connected via a gearbox and transmission system to a vertical push rod, which moves up and down at a maximum frequency of 7 Hz. The push rod is connected via a pivot to one of the arms of a symmetrical cantilever system shown in figure 1a. As the push rod moves down, the cantilevers are pushed together and the specimen-substrate assembly is bent outwards. The sandwich consists of an Al 2024 base plate with the same length and width as the substrate, on which a shorter top plate is glued with Stycast 2850 FT epoxy. This assembly is glued on top of an aluminium substrate with the same epoxy and screwed onto the cantilever arms. Three strain gauges are glued to the specimen-substrate assembly to monitor the local strain ϵ as schematically shown in figure 1b. The first gauge is attached to the back of the substrate to verify the overal behavior of the assembly. The second gauge is connected to the specimen to act as a reference signal. The third gauge is positioned close to the edge where the delamination is expected to initiate. As the delamination front propagates under the strain gauge, a part of the strain gauge will not be under strain as illustrated in figure 1c. The response of the stain gauges is given by $\epsilon_i(t) = \epsilon_{i,offset} + \Delta\epsilon_i sin(\omega t)$, with ω the frequency, is measured with three lock-in amplifiers. The strain amplitudes $\Delta\epsilon_i$ are recorded accurately and continuouesly. Since the strain gauges have a length of 6.5 mm, the gradual progression of the delamination front under gauge 3 is registered as a gradual decrease of the signal amplitude where the strain amplitude measured at the other strain gauges stay approximately constant.

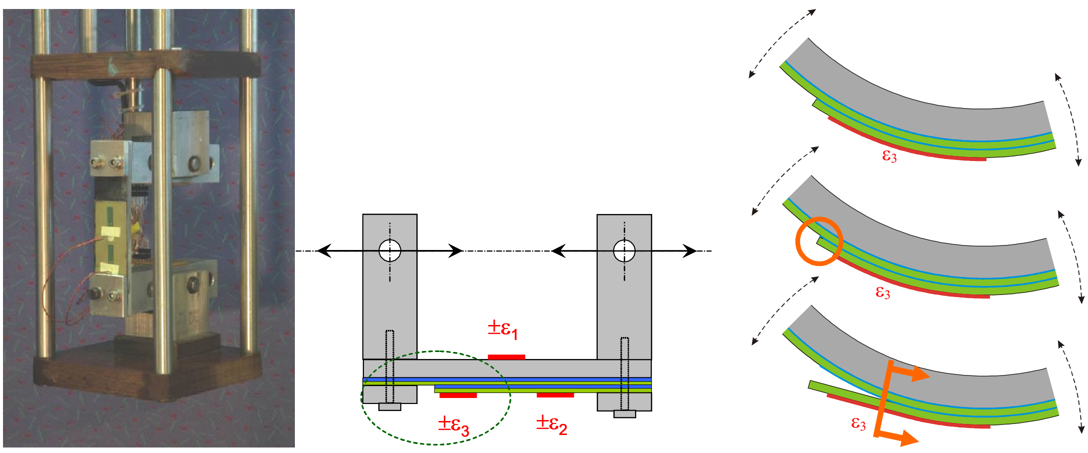

Fig. 1. The fatigue test setup with (a) the bottom of the insert, (b) the schematic of the test setup and (c) illustrates the delamination measured by the strain gauge.

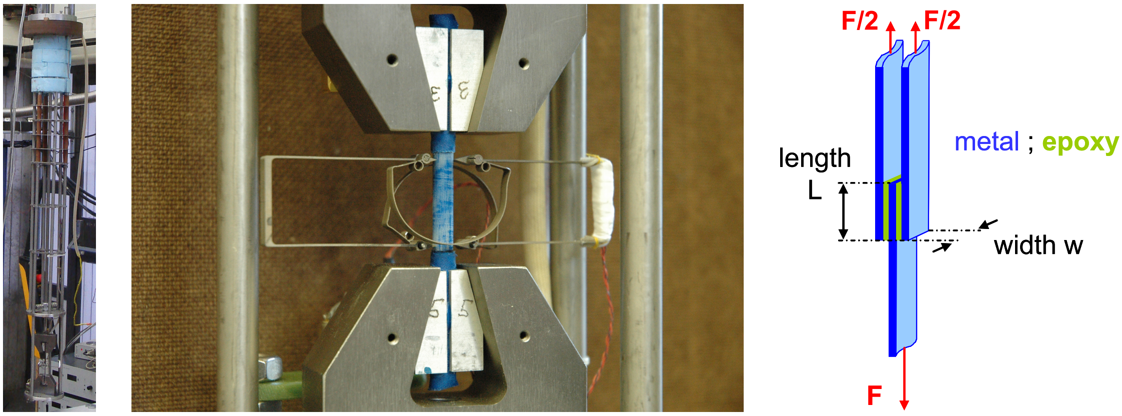

Fig. 2. Test setup with (a) the insert for stress-strain and shear strength, (b) the clamps with a stress-strain specimen and (c) the schematic setup for shear strength.

The fatigue life of seven specimens were tested within the frame of this investigation. The first four specimens (type 1) consist of a base (25x105 mm^2) and top (25x70 mm^2) plate fabricated from 0.3 mm-thick Al 2024 with a GLARE-type coating on both sides and are glued together with Stycast 2850 FT blue epoxy. The substrates (25x105 mm^2) were machined from 4 mm thick Al 51ST. The remaining specimens (type 2) have the top plate directly glued to the substrate of AL 2024 with GLARE-type coating by Stycast 2850 FT black epoxy. The epoxy layer of specimen 6 and 7 were reinforced by untreated BOPP (biaxially oriented polypropylene) and woven fibre-glass mat respectively.

2.2. Stress-strain and shear strength

The stress-strain and shear strength experiment, is shown in figure 2. On top of the insert, an encoder-controlled DC motor is connected to a satellite spindle that moves a pull rod up and down. At the bottom of the pull rod, it is attached to a clamp, and a second clamp is connected to the base-plate of the insert as shown in figure 2b. In this experiment the specimen is elongated with a constant speed of 10 μm/s, corresponding to a specimen strain rate of approximately 0.02 %/s. The motor/spindle combination is able to exert a maximum pulling force of 20 kN. The force F is registered with a S-type load cell mounted between the spindle and the pull rod. The elongation of the specimen is registered over an undeformed gauge length of 25 mm by an extensometer that is spring-clamped over the central part of the specimen as shown in figure 2b.

The axial stress-strain properties of Stycast 2850 FT specimen are measured. Cylindrical rods of 5.8 mm diameter and 125 mm length were prepared. After mixing the epoxy with the hardener 24LV, the epoxy was allowed to outgas under vacuum and then vacuum-pulled in a hollow Teflon cylinder. After curing for 2 hours at 60 °C the rods are glued to aluminium grips. Some specimen are 'post-cured' by an extra heat treatment for 2 hours at 125 °C.

The bondstrength between metal plates (Al 2024 and stainless-steel 316) and Stycast 2850 FT are measured on the double lap shear adhesive test setup schematically shown in figure 2c, where one plate is glued between two identical plates with an overlap L of 2.5 or 5 mm and a width w of 7.5 mm and cured for 2 hours at 60 °C. The same clamps, insert and motor/spindle is used as for the stress-strain measurements. The shear stress can be calculated as $\tau = \frac{F}{2wL}$.

3. Results

3.1. Fatigue life

For each specimen, the number of cycles before failure and the delamination growth rate are determined. The measurement conditions and the results of this experiment are summarized in table 1. On this table, the number of cycles to failure is the moment when the crack propagation front reaches the strain gauge #3. Specimen 1 to 4 are used to reveal the influence of cryogenic cool-down. Two identical room-temperature experiments (specimen 1 and 3) were

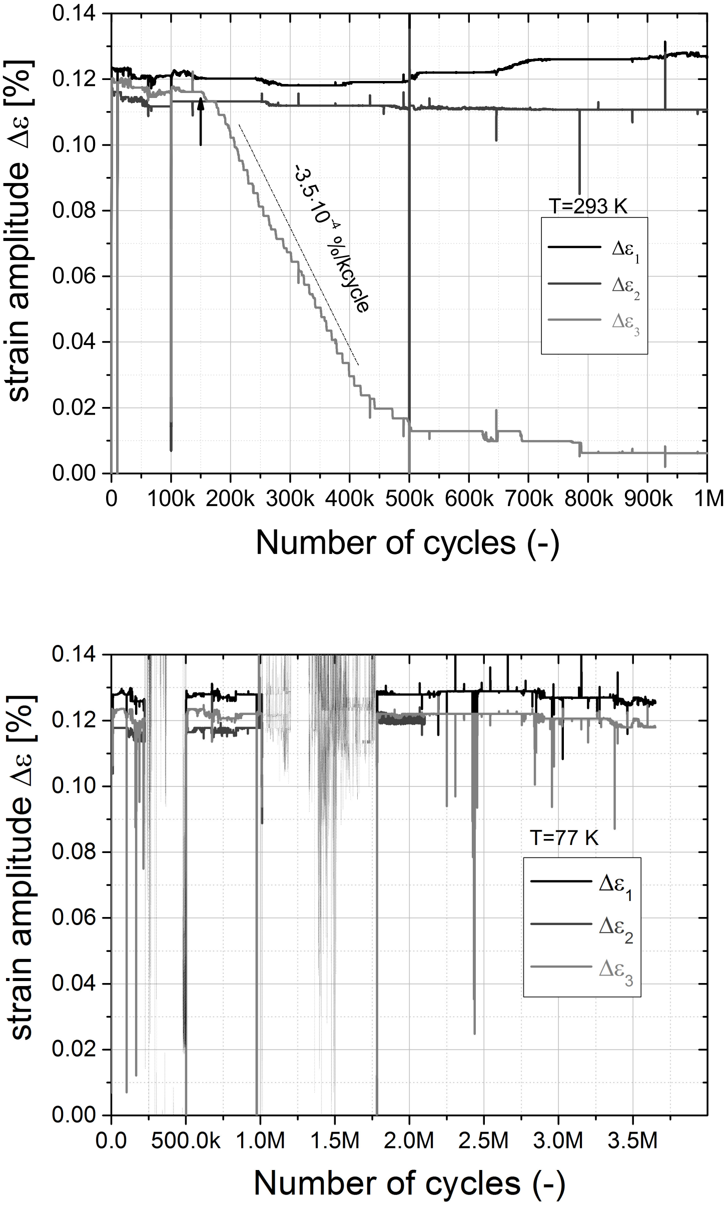

Fig. 3. strain amplitude measured on the surface (a) specimen 1 and (b) specimen 2, by the substrate-, reference- and delamination gauge, recorded against the number of strain cycles.

carried out to assess reproducibility. Specimen 2 was tested under identical conditions, except it is submerged in liquid nitrogen (77 K). The fatigue characteristics of specimen 1 and 2 are shown in figure 3. At T=293 K, delamination initiates after approximately ≈150.000 cycles and propagates gradually until it leaves the monitored zone (until the end of strain gauge 3) at ≈500.000 cycles. In contrast, at 77 K (figure 3b) no delamination and visible damage is observed up to the end of the experiment ($3.5 \cdot 10^6$ cycles). The fatigue life of this specimen increased by at least

Table 1. Summary of the fatigue results for the seven specimen measured.

Specimen #	Type	Temperature [K]	Remarks	ϵ_{offset} (± 0.01 %)	$\Delta\epsilon$ (± 0.01 %)	Cycles to failure [kcycles]	Propagation speed [μm/kcycle]
1	1	293	-	0.12	0.12	150	19
2	1	77	-	0.11	0.12	>3600	-
3	1	293	-	0.11	0.11	200	30
4	1	77	-	0.17	0.12	>7500	-
5	2	293	-	0.11	0.11	400	7
6	2	293	BOPP reinforced	0.12	0.11	1	-
7	2	293	Glass reinforced	0.13	0.11	17	40

a factor of 20. A second 77 K experiment (specimen 4) is performed, where the maximum strain is increased from 0.22 to 0.29 % by increasing ϵ_{offset} to obtain similar stress levels as those estimated to occur in the room temperature experiment. No damage is observed on this specimen after $7.5 \cdot 10^6$ cyles. Specimen 5 to 7 are initial room temperature tests of fiber reinforcement. Specimen 5 served as a reference for the setup without base-plate. Specimen 6 and 7 with untreated BOPP and a woven fibre-glass mat respectivfely showed an earlier failure at a higher delamination rate. The reason behind this is yet to be understood.

These measurements show that the Al 2024 / Stycast 2850 FT combination supports the differential thermal stresses build up during cryogenic cool-down. The fatigue life at 77 K of these Al 2024 / Stycast 2850 FT sandwiches increase significantly when cooled to 77 K, illustrating the feasibility of cryogenic FML based on these constituents. Reinforcing the Stycast layer by untreated BOPP or by woven fibre-glass matting proved to be unsuitable for FML reinforcement.

3.2. Stress-Strain and Bond Strength

In literature, there are relatively few results on the material properties of Stycast 2850 FT (Ekin (2006) and Emerson & Cuming (2004)). The axial stress-strain characteristics of two of the main envisaged FML constituents, Al 2024, Stycast 2850 FT blue and Stycast 2850 FT black, are measured at 293 and 77 K. The measured Young's moduli and tensile strength values of the Stycast specimens are shown in figure 4. These figures clearly show the increase of the Young's modulus and tensile strength of Stycast 2850 FT blue by more than a factor two. The same is true for the Young's modulus of the black epoxy. However, for the tensile strength this comparison is inconclusive, because those specimens failed at the bubbles in the epoxy and should be seen as a lower limit. This shows the effect of the epoxy quality on the performance. The result of the Al 2024 specimen with GLARE-type coating (results are not

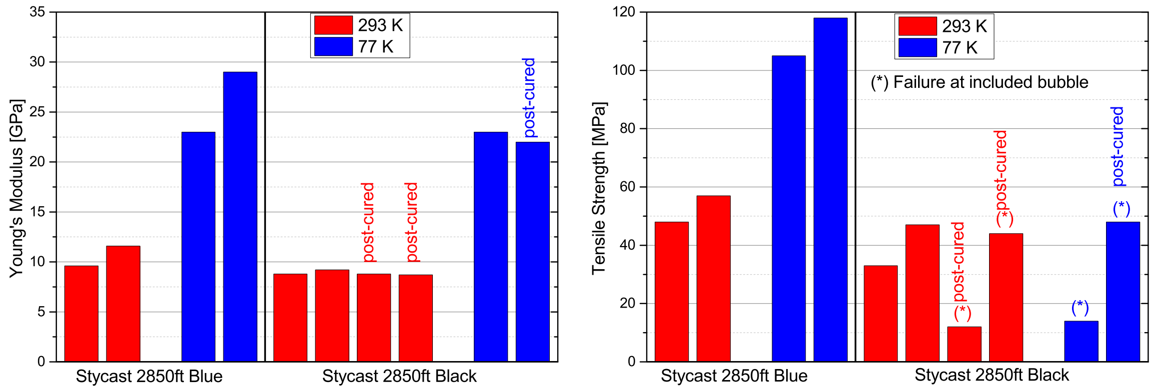

Fig. 4. Measured Young's modulus and tensile strength of the Stycast 2850 FT specimen. The blue version of the epoxy is shown on the left pane, the black one on the right. The room temperature experiments are shown in red, the experiments in liquid N_2 in blue.

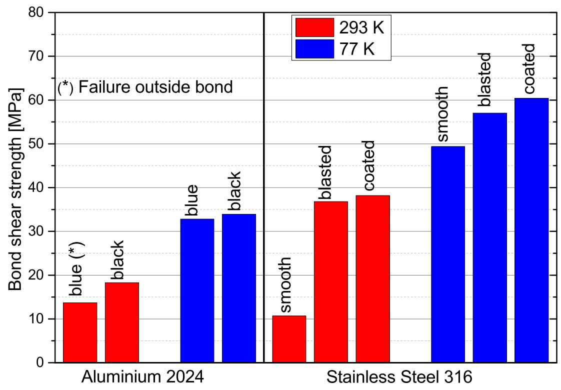

Fig. 5. Measured shear strength between Stycast 2850 FT and selected FML constituents. The room temperature experiments are shown in red, the experiments in liquid N_2 in blue.

shown here) show that these parameters increase after cool-down by 20-25 % as expected from data sheets (Kaufman (1999)).

The bond strength between Stycast 2850 FT blue and black with Al 2024, and between Stycast 2850 FT black with Stainless steel 316 subjected to three different surface treatments (smooth, sand blasted and GLARE-type coating) is tested both at 293 and 77 K. An overview of the results is shown in figure 5. Based on this limited preliminary data, it is too early to draw solid conclusions about the effectiveness of different types of surface treatments or epoxy choice. However, it can clearly be seen that the shear strength increases upon cool-down to 77 K for all tested specimen.

4. Discussion and Conclusion

A new experiment was realized to test the fatigue life of GLARE-type coated Al 2024 / Stycast 2850 FT sandwiches, serving as a model system for possible cryogenic fiber-metal laminate. Not only does this metal/epoxy combination withstand repeated cool-down to 77 K, its fatigue life increases by a factor of at least 20 at 77 K without any observable damage to the specimen. These observations are consistent with the stress-strain and bond strength experiments of the constituent materials of the tested sandwich structures. The Young's modulus, yield strength and tensile strength of Stycast 2850 FT roughly doubles at 77 K compared to room temperature, while the bond strength with the tested metals increases significantly. Although these results clearly illustrate the feasibility of cryogenic FML based on these constituents, they do not explain why their performance enhances upon cool down. To understand this behaviour in more details, a detailed study of crack nucleation and growth under combined thermal and external stress has been initiated, incorporating also different material combinations and surface treatments.

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