Influence of internal temperature development during manufacturing on thick laminates compression fatigue properties

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Abstract. Thick laminates (above 6 mm) are increasingly present in large composite structures such as wind turbine blades. Blade designs are based on static and fatigue coupon tests on 1-4mm thick laminates. However, a thickness effect has been observed, showing significantly shorter fatigue life in thick laminates. Different factors are suspected to be involved in the decreased static and dynamic performance of thick laminates. These include the effect of self-heating during fatigue testing, scaling effects, and the influence of residual stresses due to temperature gradients during manufacturing. This work studies the influence of the temperature gradients during resin infusion on the through-thickness fatigue properties in thick laminates. Coupons from thick laminates cured at different curing rates have been tested in fatigue. The work reports the compression fatigue properties of a thick laminate and relates the results with the curing rate. Safety factors between 1.23 and 1.60 regarding the influence of the curing cycles in thick laminates are reported.

1. Introduction

Voids, resin rich areas, dry spots and flaws are inevitably formed during the manufacturing process. Also, the different parameters involved in the manufacturing process such as the curing cycle, and the infusion process are likely to influence the final mechanical properties. This paper focusses on the relationship between curing rate and compression fatigue mechanical properties.

Lee [1] elaborated a series of expressions that can be used in conjunction with cure models to evaluate the effects of the curing cycle on strength values and moduli of the composite after manufacturing. A relation between the heat transfer rates and the interlaminar shear strengths (ILSS) was observed by Davies [2], lately by Liu [3] for epoxy resin in carbon-reinforced composites and recently Esposito [4] reported changes in the ILSS properties with the overheating of a glass fibre reinforced polymer.

While for thin laminates, curing temperatures do not show large gradients through the thickness, in thick laminates strong temperature gradients are observed [5]. Effectively, this is related to a curing cycle variation through the thickness, which can be expected to become more pronounced in thicker laminates. These gradients are caused by the exothermic nature of curing process and thermal diffusion problem dependent on the laminate thickness and thermal conductivities [6].

White and Kim [7] developed and studied the technique of staged curing. In this technique, two or more thin (<5 mm thick) prepreg laminates are manufactured at once with a similar curing cycle per staged laminate. Later, Ciriscioli [8] reported a technique to determine mechanical properties of thick
pre-pregs using a porous film in between. This technique was applied to compare the properties of laminates of different thicknesses at the middle thickness position.

With the help of the sub-laminate technique (a technique to extract sub-laminates with the help of peel-ply layers from thick laminates), it was previously reported [9] that mechanical properties vary through the thickness, a strong correlation was found between static strengths and heating rates during the matrix hardening. The influence of the manufacturing and laminate thickness was not reported in detail [10,11].

The present work analyses scaled compression fatigue properties, and compression fatigue properties of composites manufactured at different heating rates using the sub-laminates technique. Where the variation of the fatigue properties through the thickness is related to the variation of the heating rates during the curing.

2. Materials and methods

The present work analyses S-N curves from plates manufactured using the sub-laminate technique [11], as well as S-N curves obtained from thickness scaled compression tests carried out for 4, 10 and 20mm thick coupons [10]. In both cases laminates were vacuum infused with an epoxy resin commonly used for wind energy applications (Hexion RIM135). The core material was a UD glass fibre type E of 600 gr/m² non-crimp fabric, and in the case of the scaled compression tests biaxial glass fibre type E of 400 gr/m² non-crimp fabric was used for the tabs. Fibre content was measured for each sub-laminate and plate with averaged fibre weight ratios between 68 to 76% (see table 1), void content was below 0.1%, average densities of 1.945 gr/cm³ and DSC glass transition temperature (Tg) was around 80-85°C.

### Table 1.

| Name series | Thickness position of sub-laminate | Thickness initial plate | Thickness coupon gauge section | Width gauge section | Cross section area | Number of coupons | Fibre weight ratio, FWR [%] |
|-------------|-----------------------------------|-------------------------|-------------------------------|---------------------|-------------------|-------------------|---------------------------|
| SubA-C1     | 4.19                              | 55.65                   | 3.74                         | 14.39               | 53.8              | 13                | 71.3                      |
| SubA-C3     | 28.97                             | 55.65                   | 3.87                         | 14.74               | 57.0              | 11                | 71.0                      |
| SubA-C4     | 39.26                             | 55.65                   | 4.27                         | 14.53               | 62.0              | 6                 | 71.3                      |
| SubB-C1     | 4.00                              | 64.00                   | 4.07                         | 14.35               | 58.4              | 14                | 74.7                      |
| SubB-C3     | 26.40                             | 64.00                   | 3.99                         | 15.18               | 60.5              | 10                | 74.9                      |
| SubB-C6     | 60.00                             | 64.00                   | 4.01                         | 15.48               | 62.0              | 10                | 76.2                      |
| SubC-C6     | 60.00                             | 64.00                   | 3.93                         | 15.36               | 60.3              | 7                 | 73.2                      |
| Th4mm       | -                                 | 8.00                    | 4.00                         | 10.00               | 40.0              | 16                | 68.9                      |
| Th10mm      | -                                 | 20.00                   | 10.60                        | 25.00               | 265.0             | 11                | 70.7                      |
| Th20mm      | -                                 | 32.00                   | 20.00                        | 55.00               | 1100.0            | 9                 | 71.3                      |

Each of the series was tested in fatigue with a stress ratio R=10. Table 1 shows the average dimensions for each of the series as well as the thicknesses of the coupons origin plates. Three 60mm thick laminates were manufactured (A, B and C) with two different curing cycles (see table 1 from [11]). Plate A and B were infused at 25°C, the mould control was setup with an initial soak of 35°C and 380 minutes and post-cured at a mould temperature of 80°C for 800 minutes. In the case of plate C the initial soak mould temperature was 60°C. From such thick laminates, sub-laminates plates were
extracted at various thickness positions C1, C3, C4 and C6 (see table 1 and figure 1). Thus, series belonging to the sub-laminate tests are named as SubA, SubB and SubC. Therefore, each sub-laminate is associated with a certain through thickness position and a certain maximum heating rate (see table 2).

![60mm thick laminate](image1)  
![Sublamine extraction and coupons milling 4mm thick](image2)

**Figure 1.** Sub-laminates coupons plates manufacturing and coupons preparation.

![Th4mm](image3)  ![Th10mm](image4)  ![Th20mm](image5)

**Figure 2.** Thickness scaled compression tests setups and coupons, for 4, 10 and 20mm.

In addition, three laminates were manufactured to obtain compression specimens with gauge section thicknesses of 4, 10, and 20 mm, respectively. These plates were designated Th4mm, Th10mm, and Th20mm. From these panels, specimens were cut. These are ‘scaled’ specimens were designed to have maximum similarity between the geometries in terms of stress distribution, as well as maximum stress concentration near the gauge section [12,13].

Both the 4 mm thick coupons sub-laminates and thickness scaled tests were tested on an MTS servo-hydraulic 100 kN test frame, using the standard machine clamping system (see figure 2). Coupons of 10 and 20 mm thickness were tested on 400 kN and 1 MN test frames, respectively. For clamping, these were equipped with an assembly of steel plates and hydraulic clamping (in the case of the 1MN test frame this was hydraulic bolt tightening). Fatigue tests frequencies were limited to 2 Hz for 4 mm thick coupons to 0.5 Hz for 20 mm thick coupons to avoid a reduction of fatigue life due to the self-heating effect [14].

S-N curves were modelled according to ASTM E739 using a least squares regression with an average data sample of 10 coupons per series. A minimum of four different stress levels was tested.
Due to practical reason data was gathered up to $10^7$ cycles to failure, and extrapolated to $10^8$ and $10^9$ cycles to failure using the S-N curves models.

To analyse the scatter, confidence band lines were computed at 95% interval of confidence according to the mentioned standard. As reference stress, design values were obtained using S-N GL curve eq. 5.5.3.3 from GL guidelines [15] with a safety factor $\gamma_M = 2.21$, $\gamma_M = 1.63$ and a slope $m=10$. In addition a compression ultimate strength of 630 MPa and a tension ultimate strength of 849 MPa was used to compute the GL curve. Reserve factors were computed as the ratio between the test stresses values at 95% of confidence (from S-N curves models) and the design stresses from the GL curve at four different cycles to failure ($10^6$ to $10^9$).

### Table 2. S-N curve model according to ASTM E739. Slope and offset data, the coefficient of determination and maximum heating rate during the curing per S-N curve. Fatigue compression tests at stress ratio $R=10$.

| Name series   | S-N curve model (ASTM E739) | Max heating rate during curing\(^2\) |
|---------------|-----------------------------|---------------------------------|
|               | A  | B  | $R^2$ |                            |
|               | Least square offset | Least square slope | Least square coefficient of determination | [°C/h] |
| SubA-C1       | 48.3 ± 12.1 | -15.8 ± 4.5 | 0.787 | 25.09 |
| SubA-C3       | 61.5 ± 10.5 | -20.8 ± 3.8 | 0.916 | 55.87 |
| SubA-C4       | 35.7 ± 38.7 | -11.6 ± 14.2 | 0.432 | 64.94 |
| SubB-C1       | 42.4 ± 11.0 | -14.7 ± 4.3 | 0.753 | 16.08 |
| SubB-C3       | 101.2 ± 28.9 | -37.6 ± 11.3 | 0.828 | 37.43 |
| SubB-C6       | 60.1 ± 30.3 | -21.3 ± 11.9 | 0.583 | 48.62 |
| SubC-C6       | 34.8 ± 116.5 | -11.6 ± 46.6 | 0.048 | 150.68 |
| Th4mm         | 65.4 ± 7.4 | -23.2 ± 2.8 | 0.936 | 9.73 |
| Th10mm        | 66.4 ± 16.3 | -23.6 ± 6.3 | 0.840 | -\(^1\) |
| Th20mm        | 91.4 ± 42.0 | -34.7 ± 16.8 | 0.686 | 18.59 |

\(^1\) No data available
\(^2\) measured through the thickness during the curing

### 3. Results and discussion

Two different methodologies were considered to evaluate the influence of the manufacturing in thick laminates fatigue properties: thickness scaled tests and the evaluation of the mechanical properties through the thickness using the sub-laminates technique. To analyse the data, the approach followed was to gather experimental S-N curves with both methodologies for the same material specification. Coupons were manufactured under different conditions and with different thicknesses. The worst scenario in comparison with a design point such as the GL curve was analysed and the reserve factors were computed.

Since the maximum heating rates during the hardening have shown a strong influence in the ultimate strengths [11], the maximum heating rates were chosen as a manufacturing indicator of the curing cycle. Table 2 shows that the gathered series cover maximum heating rates from 9 to 150 °C/h. Therefore, a large range of possible manufacturing conditions can be found in a thick laminate due to the exothermic reaction and temperature gradients. According to the theory of the weakest link, the laminate component with the lowest fatigue performance will drive the degradation and failure. Therefore from the gathered S-N curves covering the full manufacturing range the worst performance will drive the design if not detailed considerations are made.
Figure 3. Coefficient of determination $R^2$ of S-N curves versus maximum local heating rates during the hardening.

Table 3. Stresses and reserve factors computed at different cycles to failure. Stresses computed according S-N curve 95% confidence band line and stresses computed according to GL curve (eq. 5.5.3.3, $\gamma_{ma} = 2.21, m=10$) [15]. Fatigue compression tests at stress ration R=10.

| Cycles to failure | $\sigma_{95\%}$ Stress at 95% confidence interval [MPa] | $\sigma_{GL}$ Stress at GL curve Design stresses [MPa] | Reserve factor $RF = \frac{\sigma_{95\%}}{\sigma_{GL}}$ [-] |
|------------------|---------------------------------|---------------------------------|---------------------------------|
| Name series      | $10^6$                          | $10^7$                          | $10^8$                          | $10^9$                          | $10^6$                          | $10^7$                          | $10^8$                          | $10^9$                          |                                   |
| SubA-C1          | 439                             | 379                             | 328                             | 283                             | 138                             | 120                             | 103                             | 89                              | 3.18                             | 3.17                             | 3.17                             | 3.17                             |
| SubA-C3          | 442                             | 396                             | 354                             | 317                             | 138                             | 120                             | 103                             | 89                              | 3.20                             | 3.31                             | 3.43                             | 3.55                             |
| SubA-C4          | 301                             | 247                             | 203                             | 166                             | 138                             | 120                             | 103                             | 89                              | 2.18                             | 2.07                             | 1.96                             | 1.86                             |
| SubB-C1          | 272                             | 232                             | 199                             | 170                             | 138                             | 120                             | 103                             | 89                              | 1.97                             | 1.94                             | 1.92                             | 1.90                             |
| SubB-C3          | 322                             | 303                             | 285                             | 268                             | 138                             | 120                             | 103                             | 89                              | 2.33                             | 2.54                             | 2.76                             | 3.00                             |
| SubB-C6          | 314                             | 281                             | 253                             | 227                             | 138                             | 120                             | 103                             | 89                              | 2.27                             | 2.35                             | 2.44                             | 2.54                             |
| SubC-C6          | 224                             | 184                             | 151                             | 123                             | 138                             | 120                             | 103                             | 89                              | 1.62                             | 1.54                             | 1.46                             | 1.38                             |
| Th4mm            | 345                             | 313                             | 283                             | 256                             | 138                             | 120                             | 103                             | 89                              | 2.50                             | 2.62                             | 2.74                             | 2.87                             |
| Th10mm           | 332                             | 301                             | 273                             | 248                             | 138                             | 120                             | 103                             | 89                              | 2.40                             | 2.52                             | 2.64                             | 2.77                             |
| Th20mm           | 268                             | 250                             | 234                             | 219                             | 138                             | 120                             | 103                             | 89                              | 1.94                             | 2.09                             | 2.27                             | 2.45                             |

Min values: 1.62 1.54 1.46 1.38

Table 2 shows the S-N curves models for the gathered data. For each series a least squares fit was computed as well as the coefficient of determination $R^2$ of such models. The coefficients of
determination are an indicator of the scatter and S-N curves confidence intervals, in other words the uncertainty of the fatigue failure. Table 2 and figure 3 shows that the coefficients of determination correlate with the maximum heating rates. This indicates that large heating rates require of larger confidence intervals and the fatigue failure uncertainty is larger. While significant heating rates during the curing enhance the ultimate strengths, in the case of fatigue, large heating rates increase the scatter and larger intervals of confidence are required.

![Figure 4](image-url)

**Figure 4.** General view of compression S-N curves. Plate A,B and C sub-laminates, curves from scaled compression coupons (4, 10 and 20 mm thick) and reference design curve according to GL guidelines (eq. 5.5.3.3, $y_{Ma} = 2.21$, $y_{Mb} = 1.63$, $\sigma_{compression} = -630 MPa$).

Figure 4 shows the compression S-N curves models from the sub-laminates series and the thickness scaled tests. Also, the GL curve is also plotted as a reference of the design level. The S-N curves belonging to plate A show higher average stresses for similar cycles to failure in comparison with plate B due to the influence of the fibre weight ratio FWR. Similar behaviour was observed for the ultimate compression strengths reported in a previous work [11]. Moreover, confidence band lines are also available in figure 4 showing larger intervals of confidence in the case of the series with significant local heating rates (see figure 3).

Moreover, figure 4 contains a graphical example of the reserve factor physical meaning, which is related to the distance between the experimental S-N curve at the 95% confidence band line and the GL curve design level for a given cycles to failure. In this way, the reserves factor were computed for all the series at different cycles to failure, where the lower cases are the most interesting ones since they represent the worst-case scenario. Table 3 shows the stresses at the 95% confidence band line, the GL curve values and the reserves factors. According to the different S-N curves gathered at various manufacturing conditions the lowest reserve factor found at $10^8$ cycles is 1.46 and the most optimistic scenario is 3.43. This indicates that the influence of the manufacturing in thick laminates can diminish the reliability of the material fatigue properties more than 200% under certain manufacturing conditions and heating rates. Moreover, taking into account this data when the fatigue performance is
also reduced by other factors such as ageing or manufacturing defects it might be plausible to reach reserve factors below 1.

The actual experimental data allows evaluating the safety factors to be used in the GL design curve due to the influence of the curing in thick laminates. For such purpose, the experimental data was compared with a GL design curve without any safety factors \((y_{Ma} = 1, y_{Mb} = 1)\) and the safety factor required to reduce the GL design curve below the gathered S-N curves (at 95% band lines) were computed (see figure 5). The computed safety factors oscillate between values of 1.23 to 1.60 at \(10^8\) cycles to failure for ultimate strengths in compression of 630 and 837 MPa respectively.

It needs to be remarked that GL curve was computed with an ultimate strength of 630 MPa provided by the thickness scaled tests [10] for a 4mm thin laminate. However, according to the sub-laminates quasi-static test results presented in [11], it is possible to achieve ultimate strengths as high as 837 MPa. If the GL design curve is computed with ultimate strengths of 837 MPa different design levels might be obtained levering the expected material performance. Therefore, since the curing cycle influence the thin laminate ultimate strengths, and no clear indications on this respect could be found. Therefore, is unclear which curing cycle should be used to evaluate the ultimate strengths to be used in the GL design curve.

Moreover, similar behaviour might be expected for resin related properties such as the shear, biaxial properties or properties in the transverse direction. Further research is on-going in studying other properties and in understanding the mechanisms that drive the influence of the manufacturing process in thick laminates fatigue properties, where it is intended to investigate the possible differences that might appear in the microstructure with different curing cycles.
4. Conclusions
Thickness scaled tests and sub-laminates tests reveal to be a useful approach to study the influence of internal heat generation during the manufacturing process on fatigue properties. Based on the data provided by thickness scaled tests and sub-laminates tests it was possible to analyse the full operational region in fatigue and determine the worst-case scenarios. The data revealed that reserve factors close to the design curves can be found for certain curing cycles and manufacturing conditions in compression loading. For the tested material, the gathered S-N curves allowed computing the safety factors between 1.23 and 1.60 regarding the influence of the curing cycles in thick laminates.

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