DIRECT COMPARISON OF NOVEL UNIDIRECTIONAL SANDWICH COUPON DESIGNS FOR ACCURATE TENSILE FAILURE STRAIN DETERMINATION OF CARBON FIBRE EPOXY MATERIAL

Gergely Czel[1,2]

[1]Department of Polymer Engineering, Faculty of Mechanical Engineering, Budapest University of Technology and Economics, Műegyetem rkp. 3., H-1111 Budapest, Hungary
Email: czel@pt.bme.hu, Web Page: http://www.pt.bme.hu/munkatarsak.php?l=a
[2]MTA-BME Lendület Sustainable Polymers Research Group, Műegyetem rkp. 3, H-1111 Budapest, Hungary

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Abstract
Three different novel sandwich coupon designs comprising continuous unidirectional protective layers were directly compared with standard coupons to assess their potential to fully eliminate stress concentrations near the gripped regions. The gripping conditions were optimised using different grade sandpaper tabs at the end of the sandwich coupons. The sandwich type coupons yielded statistically significant increase in their average failure strain compared to that of the baseline tabbed coupons. However, the three sandwich coupon types did not show significant differences.

1. Introduction

Carbon fibre reinforced epoxy (CF/EP) is the primary choice for several high performance applications because of its excellent strength-to-weight ratio. Due to the complicated inhomogeneous and anisotropic structure of this relatively new material family, coupon-level tensile testing of unidirectional (UD) plates is crucial to provide reliable input for numerical modelling. However, tabbed prismatic test coupons recommended by standard test methods ASTM D3039/D3039M and ISO 527-5 tend to fail prematurely at the gripped sections due to stress concentrations. The standards recommend thickness direction tapering of the end-tabs, but this approach is prone to debonding of the tapered sections of the tabs due to the lack of gripping force. Alternatively, the tapered ends may be created by inserting extra plies into the end of the coupons [1], but this technique is rather complicated and, therefore, not widely adopted. A possible approach to reduce the stress concentration around the gripped zone is to widen the coupon towards the ends [2]. However, this has to be done carefully due to the UD fibre structure, as not highly stressed parts tend to split off from the main body of the coupons. Butterfly and long butterfly coupons have large radii in the transition zones between the prismatic and widening regions of the coupons. Non-prismatic coupons are still prone to splitting, especially if machining is not done with extra care. Czél et al. proposed co-cured interlayer hybrid (or sandwich) tensile test coupons (see Figure 1) to eliminate stress concentrations [3]. Consistent gauge-section CF/EP layer fractures with high failure strains near that of fibres quoted by the manufacturer were reported. A 2D finite element analysis was also presented to explain the protective role of continuous UD GF/EP layers co-cured together with the CF/EP plies. Fazlali et al. [4] proposed arrow-shaped tabs to reduce the stress concentrations and compared the new coupon type to standard prismatic, co-cured sandwich and butterfly designs. The author and Fazlali et al. noticed that the CF/EP layer in co-cured sandwich coupons can have significant transverse undulation. Co-curing the CF/EP and the protective GF/EP layers at elevated temperature gives rise to thermal residual strains between the layers, which must be compensated for during the evaluation of the CF/EP layer failure strain. In order to improve the sandwich coupon design by addressing the issues mentioned, a systematic experimental programme was initiated using the same raw materials with three different layer integration techniques. A comparison with standard tabbed coupons is also a vital part of the study.

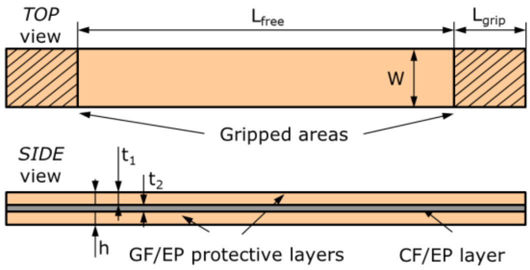

Figure 1. Schematic of a sandwich coupon with continuous protective layers

2. Experimental

This section presents the materials, manufacturing methods, equipment and results of the experimental programme.

2.1. Materials

The materials considered for design and used for the experiments were CF/EP and S-glass/epoxy (GF/EP) prepregs from Hexcel made with IM7 intermediate modulus (IM) carbon (Hexcel) and Flite Strand S-glass (Owens Corning) fibres. Both prepregs had the aerospace grade 913 epoxy resin (Hexcel) for full compatibility. Araldite 2011 (Huntsman) two-part epoxy adhesive was used to bond the end-tabs and the GF/EP and CF/EP plates for specific coupon types at room temperature. The basic properties of the applied fibres and prepreg systems can be found in Tables 1 and 2.

Table 1. Properties of the applied fibres based on manufacturer's datasheet values

Fibre type	Manufacturer	Diameter	Elastic modulus	Strain to failure	Tensile strength	Density	CTE[a]
		[µm]	[GPa]	[%]	[GPa]	[kg/m^3]	[1/K]
Flite Strand S-glass	Owens Corning	9	92	3.6-4.4	3.3-4.1	2450	$3.4 \cdot 10^{-6}$
IM7 carbon	Hexcel	5.2	276	1.9	5.7	1780	$-6.4 \cdot 10^{-7}$

[a]Coefficient of thermal expansion

Table 2. Cured ply properties of the applied UD composite prepregs

Material designation	Manufac-turer	Nominal fibre areal density[a]	Fibre volume fraction[a]	Nominal cured ply thickness[b]	Elastic modulus[b]	Failure strain
		[g/m^2]	[%]	[mm]	[GPa]	[%]
S-glass/ 913 epoxy	Hexcel	305	49	0.25	47.1	3.9[c]
IM7 carbon/ 913 epoxy	Hexcel	220	50	0.25	139.3	1.6[d]

[a]Based on the manufacturer's data
[b]Estimated using manufacturer's data
[c]Conservative average experimental value obtained with similar material for design purpose
[d]Manufacturer's data for 60% fibre volume fraction

2.2. Coupon design

The thickness of the CF/EP layer was kept at 1 mm for consistency with current material testing standards. For simplicity, the thicknesses of the GF/EP protective layers were also set to t_1=1 mm, resulting in a 3 mm thick sandwich laminate construction with a simple UD lay-up sequence: [SG_4/CF_4/SG_4] where SG stands for UD S-glass/EP and CF stands for UD IM7 CF/EP prepreg plies. Design criteria for UD sandwich coupons were identified earlier [3] to ensure immediate delamination after the first fracture of the CF/EP layer (Eq. 1).

$$G_{II} = \frac{\varepsilon_{2b}^2 E_2 t_2 (2E_1 t_1 + E_2 t_2)}{8E_1 t_1} > G_{IIC} \tag{1}$$

Where: E_1 is the elastic modulus of the GF/EP layers in the loading direction, E_2 is the elastic modulus of the CF/EP layer in the loading direction, t_1 is the thickness of one GF/EP layer, t_2 is the thickness of the CF/EP layer as shown in Figure 1. A G_{IIC}=2.0 N/mm, which was obtained with similar materials and *co-cured* sandwich construction, was considered as an estimated parameter. Substituting the material and geometric parameters in Eq. 1 yields a G_{II}=21.7 N/mm at the expected failure strain of the CF/EP layer, which is significantly higher than the estimated G_{IIC} therefore, immediate delamination is expected, so the proposed construction (see Table 3) is suitable.

Table 3. Sandwich laminate design selected for testing (Key: SG -S-glass fibre, CF- IM7 carbon fibre, subscripts indicating the number of plies in a block)

Sandwich configuration [Lay-up sequence]	Fibre areal densities of the constituent plies	Nominal thickness *h*	Calculated G_{II} at CF/EP nominal failure strain (1.6%)	Predicted elastic modulus
	[g/m^2]	[mm]	[N/mm]	[GPa]
[SG_4/CF_4/SG_4]	[305_4/220_4/305_4]	[1/1/1]	21.7	77.5

2.3. Coupon manufacturing

Baseline coupons were made by manually stacking four 300x300 mm plies of IM7/913 prepreg with the 0º same orientation, placing them on a flat Al tool plate and curing them in an autoclave (Olmar ATV 1100/2000) for 60 min at 125ºC and 7 bar pressure (recommended by the manufacturer). 20 mm thick Al top plates were used to minimise the thickness variation of the cured plates. The same procedure and cure conditions were used for all other coupon types and prepreg materials as the matrix was the same 913 epoxy in them. 1.5 mm thick woven GF/EP end-tabs were bonded on the baseline coupons with epoxy adhesive, and the 15 mm wide coupons were fabricated with a diamond wheel cutter. This rigorous experimental study aimed to systematically compare all the feasible layer integration approaches with pre-impregnated composite sheets (prepregs) and a conventional tabbed coupon type as a baseline. After careful assessment of the practical aspects of manufacturing three-layer plates from CF/EP and GF/EP prepreg plies, the following layer integration approaches were applied:

Co-curing: This is the simplest approach when all the prepreg plies necessary to build the three-layer sandwich structure with the desired layer thicknesses were laid-up and cured in the autoclave in one operation. The layer interfaces usually get undulated, and thermal residual strains arise.

Curing-on: The CF/EP plate was laid up and cured separately in the autoclave, and then the necessary number of GF/EP prepreg plies were laid up onto the cured CF/EP plate and were cured in the autoclave in a second cycle. The layer interfaces are kept flat in this case.

Bonding: The CF/EP and two GF/EP plates of the designed thicknesses were cured separately. The three plates were then bonded together with epoxy adhesive at room temperature to make a sandwich plate with continuous UD GF/EP protective layers on the CF/EP layer. Special care was taken to keep one edge of all three plates as a reference for the fibre direction and position the plates with loose-fitting 3D printed buttons inserted in holes drilled through all layers with a diamond particle-coated tool. The adhesive was applied in narrow parallel stripes, which allowed the air to escape from the gap between the plates before they joined up, while the gap was closed by slow rolling of a heavy roller perpendicular to the stripes. The stack was loaded with a total weight of about 40 kg distributed by a 20 mm thick steel plate, and the adhesive was left to cure at room temperature for 24 hours. This room-temperature bonding eliminated the thermal residual strains for this coupon type. 15 mm wide coupons were cut with a diamond wheel from all sandwich plates.

2.4. Mechanical test procedure

Testing of the new type and *baseline tabbed* coupons was executed under uniaxial quasi-static tensile loading and displacement control at a crosshead speed of 5 mm/min (total test time 100-200 s) on a computer-controlled Zwick Z250 type 250 kN rated universal electro-mechanic test machine fitted with a regularly calibrated 250 kN load cell and 100 kN rated Instron 2716-003 type manual wedge action grips. The strains were measured optically with a Mercury Monet type video-extensometer using two 5 Mpixel resolution cameras to cancel the possible effect of out of plane displacements of the coupons. An approx. 50 mm gauge length and white dots on a black background were used for high contrast to make targets for the video-extensometer (see Figure 2). A minimum of ten coupons were tested of each coupon type. The failure strain and stress of the sandwich coupons were determined at the last data point before the major stress drop due to CF/EP layer fracture. The rest of the stress-strain curves was truncated for simplicity. The elastic moduli of the individual coupons were determined by fitting lines manually to the initial part of their stress-strain curves in the strain regime 0-0.3%.

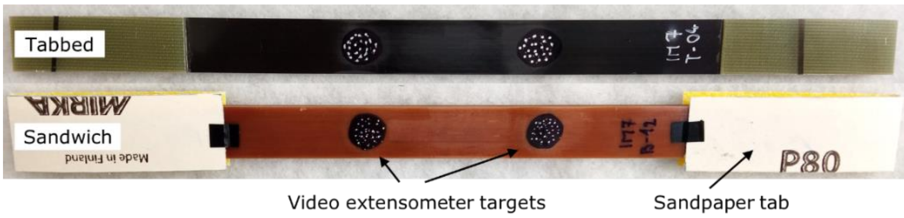

Figure 2. Baseline tabbed and sandwich coupon prepared for tensile testing

2.5. Optimisation of gripping conditions

The ends of the coupons were protected from the sharp nails of the grips using sandpaper tabs secured with small pieces of soft insulating tape. The length of the tabs was designed to hang 5 mm over the serrated part of the grips. Four different grit-size sandpapers were tested (see Figure 2), and the following conclusions were drawn: (i) too coarse papers (P40 and P60) severely damage the coupon surface and may initiate fracture of the GF/EP protective layer, (ii) too fine grades (P100) tend to have a thinner backing paper which lets the nails of the grip penetrate through and apply non-uniform pressure on the coupons and may cause splitting from the end, (iii) the most uniform pattern left on the tested coupons was achieved with grade P80 which indicates the most uniform distribution of gripping pressure, therefore it was judged optimal.

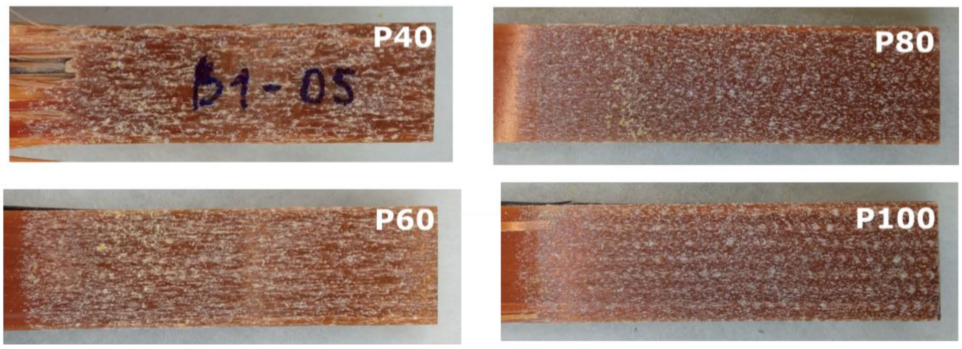

Figure 3. Marks left by different grade sandpaper tabs after testing until fracture of the CF/EP layer

2.6. Results and discussion

Figure 6. shows the stress-strain curves of the *baseline tabbed* coupon series with a second-order polynomial fitted to all the individual curves with regression.

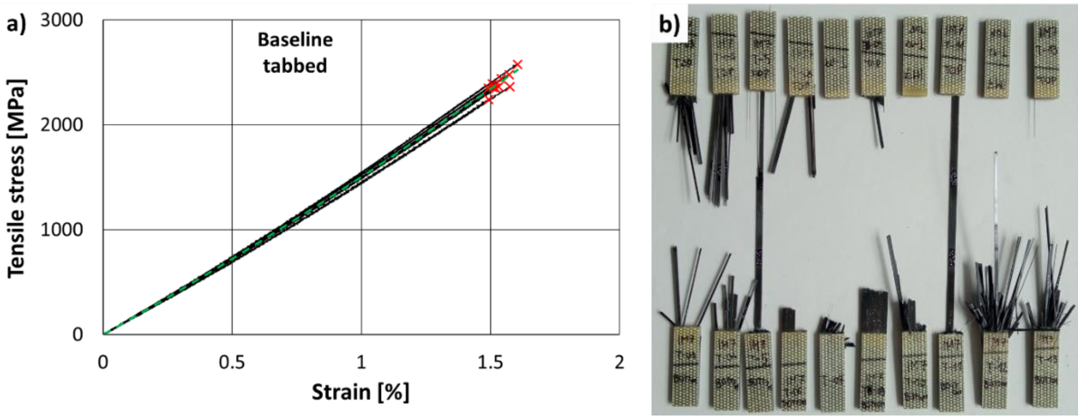

Figure 6. a) stress-strain curves and b) post-mortem images of the *baseline* coupons

The test graphs were consistent with limited scatter and showed visible non-linearity, as expected based on previous observations by the author and other researchers. The pure CF/EP coupon series provided a baseline for comparing the failure strains obtained with the novel sandwich coupons. They also enabled the failure stress estimation of the sandwich coupons, where direct determination of the stress in the CF/EP layer is not possible. The type of the function fitted to the stress-strain curves was selected by assuming a linear change of the elastic modulus with strain as proposed by Kumar et al. **Error! Reference source not found.**. This approach yields a second-order polynomial for the stress-strain relation. The parameters of the fitted function are summarised in Eq. 2.

$$\sigma = 114.2\varepsilon^2 + 1390.9\varepsilon - 2.930 \tag{2}$$

A good fit of the function to the test curves was confirmed visually and indicated by the high R^2=0.998. The small negative constant term is considered negligible (less than 0.2%) to the average stress at failure. The strengths of the CF/EP layer in the sandwich coupons can be estimated by extrapolating the polynomial (fitted to the baseline series) until the failure strain of the CF/EP (marked with a significant stress drop) in the selected sandwich coupon. Figure 6b shows the failed coupons and confirms that failure near the edges of the end-tabs was dominant, as expected for this series.

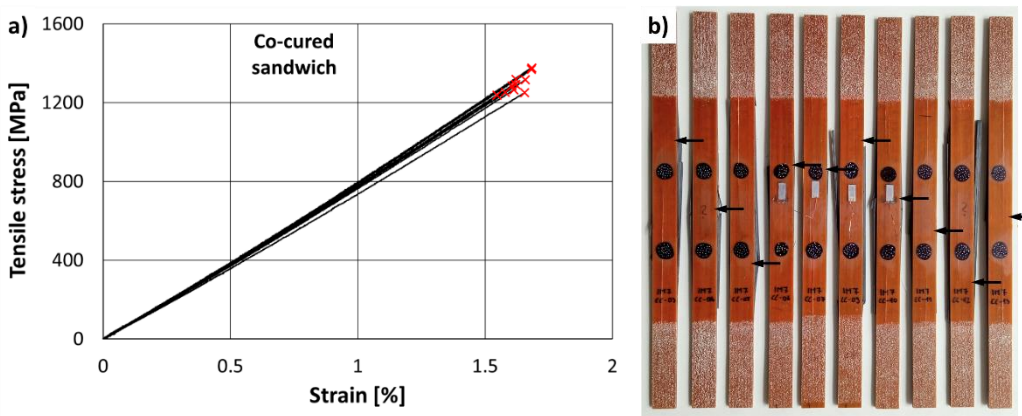

Figure 7. a) stress-strain curves (truncated at CF/EP layer fracture) and b) post-mortem images of the *co-cured* sandwich coupons with arrows showing the CF/EP failure locations

The stress-strain curves of the *co-cured* sandwich coupon series are presented in Figure 7a after evaluating the stresses using the full thickness of the sandwich coupons (please note the reduction in stresses compared to the baseline series). The test curves were consistent with limited scatter, and the non-linearity was reduced as expected, as the UD GF/EP protective layers were reported to show a linearly elastic tensile response. Figure 7b shows the appearance of the test coupons after CF/EP layer fracture and the failure locations based on visual observations. It is worth highlighting that the layers of the coupons remained bonded in the gripped areas therefore, handling and failure location determination was convenient, and all coupons failed within the gauge section.

The *cured-on* sandwich coupon series produced the most consistent stress-strain curves with little scatter, especially in the failure strain (see Figure 8a). Very similar results were obtained with these and the co-cured coupons. Failure locations were also found in the gauge section for all coupons (see Figure 8b).

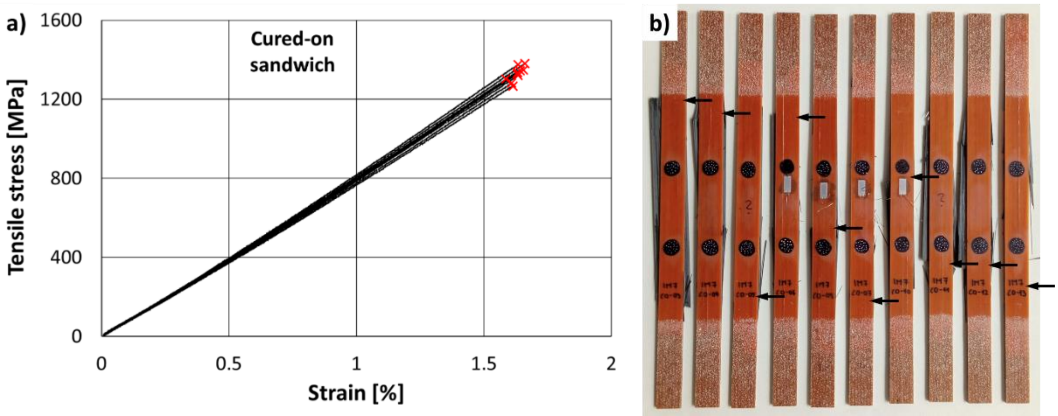

Figure 8. a) stress-strain curves (truncated at CF/EP layer fracture) and b) post-mortem images of the *cured-on* sandwich coupons with arrows showing the CF/EP failure locations

Figure 9a shows the stress-strain response of the *bonded* sandwich coupon series. The scatter of the data is also small in this case, but the stresses are generally reduced by the increased thickness of the coupons due to the adhesive layers. Only one CF/EP fracture was observed at the edge of the gripped section of the coupon (see Figure 9b). A significant amount of the adhesive was falling out of the sandwich coupons as small plates, possibly due to the dynamic effects of the CF/EP fracture and sudden delamination.

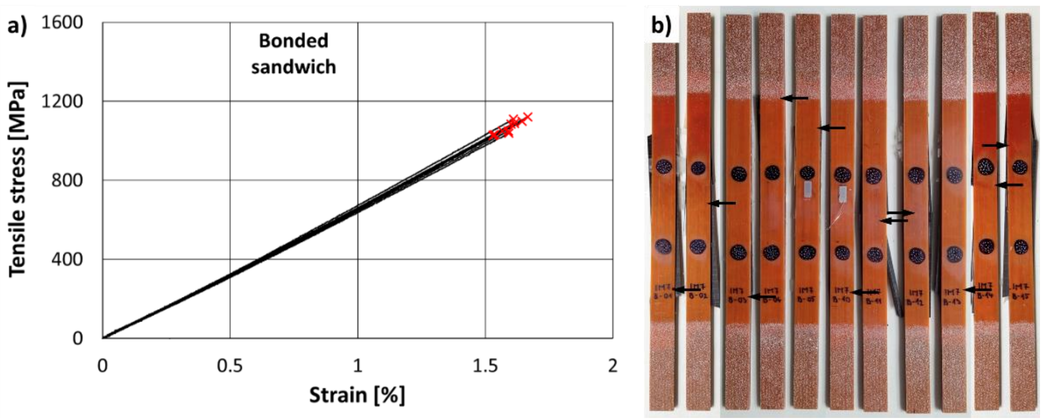

Figure 9. a) stress-strain curves (truncated at CF/EP layer fracture) and b) post-mortem images of the *bonded* sandwich coupons with arrows showing the CF/EP failure locations

As mentioned earlier, the *co-cured* and the *cured-on* sandwich coupons are affected by thermal residual strain at room temperature because, in these cases, the layers are integrated at the cure temperature of the composite layers (125ºC). Therefore, a nominal temperature change of ΔT=100K was considered when the CF/EP layer thermal residual strain $\varepsilon_{CF/EP}$=-0.022% was estimated following the method presented in [2].

Table 4. summarises the tensile test results of the four examined coupon types. The average elastic modulus of the CF/EP material measured with the baseline was slightly higher than the estimated value

(141.1 vs 139.3 [GPa] see Table 4). In contrast, the same parameter of the *co-cured* and *cured-on* sandwich coupons was slightly lower than predicted (74.2 and 75.6 vs 77.5 [GPa], see Table 4), but these small deviations are considered acceptable. The *bonded* sandwich coupons had significantly lower average modulus and stress at CF/EP fracture than the other sandwich coupon types due to their thickness being increased by the adhesive layers.

Table 4. Key average parameters of the tested coupon series (CoV- coefficient of variation in [%])

Coupon type	Width	Thickness	Initial elastic modulus	Measured failure strain	Failure stress (coupon)	Estimated CF/EP failure stress[a]	Thermal corrected failure strain
	[mm]	[mm]	[Gpa]	[%]	[MPa]	[MPa]	[%]
Baseline tabbed	14.98	0.98	141.1	1.535	2390.7	-	-
CoV	0.2	0.2	2.5	2.5	3.8	-	-
Co-cured sandwich	15.01	2.98	74.2	1.626	1296.3	2561.4	1.605
CoV	0.3	0.3	2.0	2.7	3.8	3.0	2.7
Cured-on sandwich	15.03	2.99	75.6	1.630	1328.1	2567.4	1.608
CoV	0.05	0.7	1.8	1.4	2.8	1.5	1.4
Bonded sandwich	15.03	3.61	62.6	1.589	1065.6	2496.4	-
CoV	0.1	1.2	2.0	2.8	3.3	3.1	-

[a]Calculated by substituting the failure strains in the polynomial of equation (2) fitted to the stress-strain curves of the *baseline* coupons.

Figure 10. compares the failure strain values measured with the four different coupon designs. In case of the *co-cured* and *cured-on* sandwich coupons, the strains are corrected for the thermal effect. Overall, the different coupon series yielded similar results with low scatter. The sandwich coupons showed higher failure strains than the baseline ones, but the difference was not high enough to judge the significance without statistical investigation. Therefore, two-sample Student's t-tests with two-tailed distribution were applied, considering unequal variances (also called Welch-test). The assessment indicated that all three sandwich coupon series resulted in statistically significant difference in the mean CF/EP failure strains compared to that of the baseline coupon series. However, the difference between the CF/EP failure strains obtained with the three different sandwich coupon designs was not statistically significant.

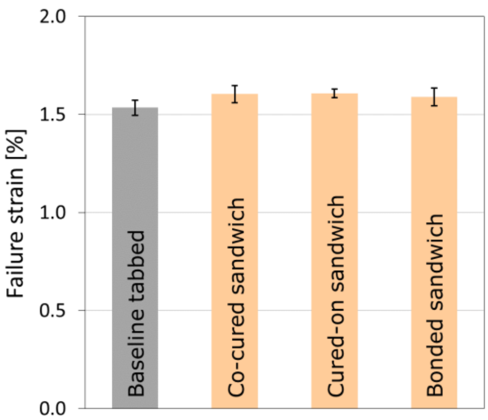

Figure 10. Comparison of the CF/EP failure strains obtained with the baseline and sandwich coupons

3. Conclusions

The following conclusions were drawn from the study of different coupon designs to accurately determine the failure strain of unidirectional carbon fibre reinforced epoxy (CF/EP):

- Novel unidirectional sandwich coupons with full-length glass fibre/epoxy (GF/EP) protective layers were developed using three different layer integration methods: i) *co-curing* all prepreg plies at once, ii) *curing-on* GF/EP prepreg plies to a solid CF/EP plate iii) *bonding* three cured composite layers together at room temperature.
- The gripping conditions of the sandwich coupons were optimised. P80 grit sandpaper tabs are recommended for the most uniform load distribution in the gripping zone.
- A method using a second-order polynomial function fitted to the stress-strain curves of the baseline coupons was presented as a practical means of estimating the tensile strength of the CF/EP material in the sandwich coupons.
- The failure strain values obtained with the sandwich coupon series showed a statistically significant increase compared to those obtained with the *baseline tabbed* coupons due to the elimination of stress concentrations near the gripped zones. Whereas the failure strain of the three different sandwich coupon series did not show statistically significant differences.
- The *bonded* sandwich coupons were outstanding as they ensured flat layer interfaces, full delamination between the layers upon CF/EP failure and did not require thermal residual strain correction. Therefore, this coupon type is recommended for further development.

Acknowledgements

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