

Microwave Curing of Carbon-Epoxy Composites: Penetration Depth and Material Characterisation.

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Abstract

Microwave heating has several major advantages over conventional conductive heating when used to cure carbon-epoxy composites, especially in speed of processing. Despite this and many other well-known advantages, microwave heating of carbon-epoxy composites has not taken off in industry, or even academia, due to the problems associated with microwave energy distribution, arcing, tool design and (ultimately) part quality and consistency, thus leading to a large scepticism regarding the technique/technology for heating such type of materials. This paper presents some evidence which suggests that with the correct hardware and operating procedure/methodology, consistent and high quality carbon-epoxy laminates can be produced, with the possibility of scaling up the process, as demonstrated by the micro- and macro-scale mechanical test results. Additionally, the author proposes a methodology to practically measure the maximum microwave penetration depth of a carbon-epoxy composite material.

Keywords

- A. Polymer-matrix composites (PMCs)
- B. Mechanical properties
- B. Interface/interphase
- E. Cure

1. Introduction

The production of quality parts, lower cost and time has been a priority for manufacturing companies, and increasingly so in today's very competitive global market, particularly for companies in developed countries where costs are generally higher. Additionally, the increasing demand of composite-intensive aircraft such as Boeing's 787, Airbus' A350 and Bombardier's C-series, as well as the expansion of composites into applications which were previously considered unsuitable (e.g. automotive, electronic packaging, etc.), has meant that increased productivity at a lower cost is key.

The production of parts made of composites typically requires the purchasing of costly materials – cost of carbon fibre is estimated to be better than 500x greater than that of steel [1] – followed by a lengthy and energy-intensive heating process. When producing parts made of polymer matrix composites (PMCs), the low thermal conduction/diffusivity of the matrix leads to an inherent limitation in cycle reduction using conventional heating methods, thus a 24 hour cure cycle is sometimes necessary for curing thick parts. One possibility to reduce production time and its associated costs is to use alternative heating methods such as microwave (MW) heating. The advantages of MW heating are well-known [2-5], but there are some major challenges remaining, such as even energy distribution and consistency, arcing, tooling design and part quality. These challenges need to be addressed before MW heating/curing can be considered for (structural) industrial applications.

In the present investigation, carbon-epoxy composites were cured in a highly homogeneous MW field, employing a suitable heating/curing methodology – which differs from the work reported previously by other authors as described by the discrepancies in the results obtained which are explained later. These samples were then tested under different loading conditions and the performance was evaluated against conventionally cured composites. Additionally, the importance of MW penetration depth is presented, and a practical method for measuring this property is introduced. The main objectives of the current work are:

- Present the current state of the art in MW curing of carbon-epoxy composites, and clarify the discrepancies in the physical and mechanical test results obtained by previous investigators.
- Provide a methodology to measure MW penetration depth in composites.
- Assess the mechanical properties of MW cured composites under tension, compression, in-plane shear (IPS) and indentation loading, and compare the results with conventionally cured samples.

- Propose an explanation for any differences in the mechanical properties of MW cured and conventionally cured composite materials.

Many papers have been published in the field of MW heating of materials, such as cement [6], rubber [7], polymers [8-12] and polymer composites [13-23]. The current summary will only focus on MW heating of carbon fibre reinforced polymer (CFRP) composites, more specifically carbon-epoxy composites, as these present some specific challenges (e.g. arcing, selective heating, etc.) other types of materials (e.g. thermosetting polymers, thermoplastics, glass-reinforced polymers) may not experience, and thus possibly the reason why there are relatively few publications in this topic/material. As mentioned in §1, there are some discrepancies in the results produced in the past [13-22], which is believed to be due to:

- i) Differences in hardware design: MW systems require careful design, as achieving a high MW field homogeneity is critical to achieve a highly homogeneous heating throughout the material. The MW systems used in the past were relatively simple systems, and therefore it would seem unlikely these could avoid cold/hot spots across the sample, as evidenced by the scepticism that has existed and exists even today regarding MW heating of (CFRP composite) materials.
- ii) Different methodologies used to define the process cycle: Most of the aforementioned MW systems lacked temperature control, thus the processing methodology was typically ‘x’ MW power for ‘y’ time. Such a heating profile would have produced a variation in heating rate as a function of time, and a fixed dwell temperature would have been unlikely. This would have inevitably led to a different cure cycle to the one which thermosetting resins are normally designed to follow. Additionally, in conjunction with point i), different MW applicator designs, and/or waveguide design (or lack of) probably had a different effect on the material (even if they were set at the same MW power) due to the different MW field distributions present in the cavities, i.e. the reproducibility of the heating process and subsequent results are unlikely.
- iii) Exposed carbon fibres cause arcing: Arcing causes three very undesirable effects; a) detrimental damage on the material, b) vacuum bagging becomes unfeasible thus leading to high void content, c) health and safety implications. The probability of arcing is greater in inhomogeneous MW systems.
- iv) Mechanical tests carried out on samples with non-standardised dimensions: In the past, samples produced using MWs were typically less than one wavelength (i.e. 125mm), and smaller than the dimensions recommended by test standards such as ASTM D3039 [24], ASTM D6641 [25] and

ASTM D3518 [26], possibly due to the difficulty in obtaining a highly homogeneous MW field over the specimen volume. The fact that testing of MW cured composites were only carried out for tension, interlaminar shear (using short beam shear) and flexure tests (i.e. tests which do not require specific test jigs and can be done with relatively small/short coupons) is an indication of the serious difficulties past researchers experienced to produce large(-r) samples. Therefore, it may seem logical that tests under compression loading for example were not carried out, even when compressive properties are possibly, together with fracture, two of the most important mechanical properties of (composite) materials. As an analogy, when CFRPs are cured in a conventional oven and undergoes excessive thermal runaway for example, the material is thrown away rather than being tested, since the material has undergone an unsuitable cure cycle and the material is not in an 'acceptable' condition. Likewise, knowing that MW heating of CFRPs in the past was neither consistent, homogeneous, nor followed a suitable procedure, it is difficult to assume the results in the literature are accurate or consistent.

Having these points in mind, Kwak et al's [3] study may have been the first publication which described a suitable methodology to heat CFRPs using MWs, producing laminate sizes large enough to follow the relevant mechanical test standards with a high degree of confidence, reliability and consistency. This has been a significant step forward as the results presented in the past were highly scattered, and little work was done on process reliability [4]. Kwak et al's subsequent study [23] was possibly the first publication that produced a thick (50mm+) CFRP laminate with MWs using the procedure in [3]. A similar study was carried out by Wei et al [21], where a laminate with dimensions of 76x76x38mm – again, dimensions of less than one wavelength – was heated using MWs, however MW was used for post-curing only.

When assessing the main outcomes of the work carried out in the past by other investigators (Table 1) [13-22], it can be seen that in terms of T_g , Fang and Scola [14] reported an increase, Papargyris et al [18] reported no significant changes, and Paulauskas [22] reported a decrease by using MW heating. In terms of mechanical properties, various authors [14,15,18,19] reported similar or increased values, whereas Paulauskas [22] reported a decrease with MW curing. In the most recent publications related to testing of MW cured composites, Kwak et al [3] reported similar T_g , similar 90° tensile strength, and an increase in 0° tensile strength by MW curing. Kwak et al [23] later demonstrated that the fracture toughness G_{IC} indicated an apparent linear increase with fibre-matrix interfacial shear strength (IFSS), where the MW cured G_{IC} was greater than the oven cured G_{IC} due to an increase in IFSS.

2. Experimental

2.1 Materials and Equipment

The materials and MW equipment employed in the present study are consistent with those used in [23], i.e. 600g/m² uni-directional (UD) out-of-autoclave (OoA) carbon fibre reinforced epoxy from Gurit, which has a PAN (polyacrylonitrile)-based carbon fibre with an elastic modulus of 255GPa, tensile strength of 4.3GPa, fibre density of 1.8g/cm³, and cured ply thickness (CPT) of 0.6mm [27]. Four plies were laminated to produce 2.4mm thick laminates for the tension, compression and in-plane shear samples. Two plies were first debulked in a vacuum table for 30 minutes, and the two halves were then further debulked for an additional hour. There was a small difference in this final step between the samples to be cured conventionally and using the MW. In the latter case, epoxy tape was used to cover the edges of the laminate to avoid exposed carbon fibres, whereas in conventional curing this was not required. The samples were introduced into the oven and MW respectively after final debulking.

The laminates produced for the tension and compression tests had a stacking sequence of $[0^\circ]_4$, whereas the laminates produced for the in-plane shear tests had a stacking sequence of $[\pm 45^\circ]_S$.

The VHM 100/100 MW (VHM) equipment (in TWI Middlesbrough, UK) is from Vötsch Industrietechnik GmbH [28], which has 12 magnetrons (two on each side of the hexagon) operating at a MW frequency of 2.45GHz, and has been shown to produce a highly homogeneous MW energy distribution, as described in [3,23,28]. MW field homogeneity is believed to be achieved due to a combination of cavity geometry, waveguide design and magnetron firing sequence, thus leading to a highly complex field where the MW fields are constantly changing, and hence why the orientation of fibres in this case, i.e. relatively simple, thin laminates, is believed to not have a great influence on the heat distribution as there is a small chance of creating standing wave patterns.

In terms of operability, a fibre optic temperature probe is connected to the VHM computer, in conjunction to its S!MPATI software, it is possible to provide temperature feedback, thus allowing the programming of the cure cycle as one would do in a conventional oven/autoclave.

A frequency of 2.45GHz is known to provide a good compromise between MW heating and penetration depth of dielectric materials in the available MW frequencies for industrial, scientific and medical (ISM) use. The low cost to produce magnetrons at this frequency is also an advantage.

2.2 Curing Methodologies

The oven cured laminates were produced using a single-sided aluminium mould by means of OoA vacuum bagging (at -1.0bar) using typical consumables and curing methodologies used in the composites industry. The MW curing methodology was consistent with that presented by Kwak et al [3,23]. The most noticeable differences in terms of process methodology compared with studies from other authors [13-22] was the use of epoxy tape to shield the edges of the CFs to avoid arcing. Additionally, temperature feedback was used to control MW power and maintain the programmed cure cycle. The 18 laminates produced are described in Table 2.

300x300x2.4mm laminates were produced for the tension, compression, and IPS tests. Two laminates were produced for each test case, i.e. one oven cured, and one MW cured. The heating rate used was 2°C/min and 10°C/min for the oven and MW cured laminates respectively.

2.3 Microwave Penetration Depth

MW penetration depth is defined as the depth at which the MW magnitude decreases to e^{-1} (i.e. ~0.37) of its original magnitude. The establishment of the MW penetration depth of a material at a specific MW frequency is of much importance as this will determine whether the material under investigation will heat evenly through the thickness of the material. The theoretical penetration depth calculation of an electrically conducting material is based on Eq.1 [29].

$$D_p = \sqrt{\frac{1}{\pi \cdot f \cdot \mu \cdot \kappa}} \quad (\text{Eq.1})$$

where 'f' is frequency [Hz], 'μ' is magnetic permeability [H/m] and 'κ' is electrical conductivity [S/m].

The author has only identified two publications by Cuccurullo et al [30,31] in which an attempt was made to measure this parameter/property using a practical approach. The methodology relied on using a thin-walled (0.5mm) metallic cylinder filled with water. A thermal imaging camera was used to measure the temperature gradient along the side of the enclosure as the water in the container was heated using MWs. As the enclosure wall was very thin, and was made of a material with a high thermal conductivity, the temperature gradient caused by MW heating was rapidly and easily picked up by a thermal imaging camera. This approach is useful for assessing the MW penetration depth of conventional materials, however some modifications are required to be used on materials such as CFRP composites.

The author has proposed an alternative (Fig. 1), which comprised an aluminium vessel, two poly-tetra-fluoro-ethylene (PTFE) sheets, a thermally and electrically insulating support, a glass bowl with one litre of water, aluminium tape, and an aluminium sheet with (and without) a hole.

The aim was to heat the water by MWs passing through the perforated hole only. The MW power and heating time (1kW for 10 minutes) was set to provide a significant increase in water temperature, whilst not encouraging heating due to conduction/convection. The CFRP laminates were placed between two PTFE sheets that were used to support and maintain the shape of the laminates. PTFE was used due to its very low dielectric properties and therefore it was deemed safe to assume little MW absorption/heating by the PTFE sheets. Temperature probes were located at two different locations on the laminate (T1 and T2), and at two different locations within the water vessel (T3 and T4). The change in water temperature was recorded during the ten-minute cycle. Various tests were done with a range of ply thicknesses until the change in water temperature became minimal. Two reference tests (i.e. one with a solid aluminium plate and one with the perforated plate with no CFRP) representing the minimum and maximum MW penetration cases were also carried out in order to gain confidence in the methodology.

2.4 Mechanical and Physical Characterisation

2.4.1 Tension

The tensile strength and elastic modulus were determined based on ASTM D3039M [24]. The test specimens had dimensions of 230x25x2.4mm, and the cross-head speed was 2 mm/min. The tensile strength was calculated from Eq. 2 [24].

$$\sigma_{t,max} = \frac{P_{t,max}}{A} \quad (\text{Eq.2})$$

where $\sigma_{t,max}$ is the maximum tensile strength (MPa), $P_{t,max}$ is the maximum failure load (N) and A is the cross-sectional area (mm²). The elastic modulus (i.e. Young's Modulus) was calculated from Eq. 3 [24].

$$E_t = \frac{\Delta\sigma_t}{\Delta\varepsilon_t} \quad (\text{Eq.3})$$

where E_t is the tensile elastic modulus (GPa), $\Delta\sigma_t$ is the change in stress (MPa), and $\Delta\varepsilon_t$ is the change in strain. The 'Δ' range was between 1000-3000με. Strain values were obtained through one bi-axial strain gauge (axial and lateral readings) on one face of each coupon. Glass fibre reinforced polymer (GFRP) composite end-tabs were bonded using a room temperature cure adhesive on the 0° and 90° tensile test coupons. The adhesive had a chamfer on the gauge section to reduce stress concentration due to the tabs. An Instron 8500 with a 500kN load cell, and an Instron 5567 with a 30kN load cell was used for the 0° and 90° tensile tests respectively. The results of a minimum of six coupons (out of ten coupons) with acceptable failure modes were considered and analysed.

2.4.2 Compression

The compression strength and modulus were determined based on ASTM D6641 [25]. The test specimens had dimensions of 140x12x2.4mm, and the crosshead speed was 1.3mm/min. The compression strength was calculated from Eq. 4 [25], and the compression modulus was calculated from Eq. 5 [25].

$$\sigma_{c,max} = \frac{P_{c,max}}{A} \quad (\text{Eq.4})$$

where $\sigma_{c,max}$ is the maximum compressive strength (MPa), $P_{c,max}$ is the maximum failure load (N) and A is the cross-sectional area (mm^2).

$$E_c = \frac{\Delta\sigma_c}{\Delta\epsilon_c} \quad (\text{Eq.5})$$

where E_c is the compressive elastic modulus (GPa), $\Delta\sigma_c$ is the change in stress (MPa), and $\Delta\epsilon_c$ is the change in strain. The strain range used for the calculation of E_c was between 1000-3000 $\mu\epsilon$. Strain values were obtained through one bi-axial strain gauge on one face of each coupon to monitor buckling/bending effects. Glass fibre reinforced polymer (GFRP) composite end-tabs were bonded using a room temperature cure adhesive. The small gauge length meant that producing a chamfered edge with the adhesive was not possible. An Instron 5567 with a 50kN load cell was used for the compression tests. The results of a minimum of six coupons with acceptable failure modes were considered and analysed.

2.4.3 In-plane shear

The IPS strength and shear modulus properties of the material were determined based on ASTM D3518 [26]. The test specimens had a balanced and symmetric ($\pm 45^\circ$)_{2S} layup, with dimensions identical to those used for the tensile tests (i.e. 230x25x2.4mm), and the cross-head speed was 2mm/min. The IPS strength was calculated from Eq. 6 [26].

$$\tau_{12} = \frac{P_{max}}{2A} \quad (\text{Eq.6})$$

where τ_{12} is the maximum IPS strength (MPa), P_{max} is the maximum failure load (N) and A is the cross-sectional area (mm^2). The IPS chord modulus of elasticity was calculated using Eq. 7 [26].

$$G_{12}^{chord} = \frac{\Delta\tau_{12}}{\Delta\gamma_{12}} \quad (\text{Eq.7})$$

where G_{12}^{chord} is the IPS modulus (GPa), $\Delta\tau_{12}$ is the difference in applied shear stress (MPa) between two shear strain points, and $\Delta\gamma_{12}$ is the difference in shear strain between two shear strain points. The ' Δ ' range was between 1500-5500 $\mu\epsilon$. Strain values were obtained through one bi-axial strain gauge on one face of the coupon. Glass fibre reinforced polymer (GFRP) composite end-tabs were bonded using a room temperature cure epoxy adhesive. The adhesive layer had a chamfer on the gauge section. An Instron

8500 with a 30kN load cell was used for the IPS tests. The results of a minimum of six coupons with acceptable failure modes were considered and analysed.

2.4.4 Failure mode assessment

Scanning electron microscopy (SEM) was used to assess the differences in failure modes between the oven and MW cured composites tested under 90° tensile. The areas of interest included degree of matrix remaining on the fibre surface, fibre bridging/disbonding and matrix fracture surface.

2.4.5 Indentation

Indentation testing is a proven technique used to assess a material's hardness, as demonstrated by the standardisation of such technique in international test standards such as ASTM D2583 [33], ASTM E2546 [34] and ISO 14577-4 [35]. The difference between standard indentation methods and the more recent instrumented methods is mainly on the size and displacement of the indentations, which can be 'a few' nanometres. Additionally, with the use of highly sophisticated and sensitive equipment, the indentations can be carried out in a very controlled manner, thus increasing the reliability compared to standard tests. The biggest advantage of small-scale indentation is that very small and local changes in material properties, such as the matrix in between the fibres of CFRPs, can be accurately measured.

During nano-indentation testing, a load versus displacement graph was plotted as the indenter contacted the matrix. It was then possible to calculate the reduced modulus (or indentation modulus) E_r (GPa) using the unloading portion of the curve, and since E_r is a combination of the modulus of the matrix and the indenter material, the elastic modulus E of the specimen can be obtained by using Eq. 10 [36]:

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_{in}^2}{E_{in}} \quad (\text{Eq.10})$$

where E (GPa) and ν are the matrix elastic modulus and Poisson's ratio respectively, and E_{in} (GPa) and ν_{in} are the same properties of the indenter material. For a diamond indenter tip, E_{in} is 1141GPa and ν_{in} is 0.07 [37]. The Poisson's ratio of the epoxy was assumed to be 0.35 [38]. The indentation was load controlled to 5mN, thus creating indentations ~1000nm deep. This equates to an indentation imprint of ~4µm diameter (when drawing a circle around the three peaks).

A total of 360 indentations were carried out for the oven and MW cured laminates (i.e. 180 indentations each). The tests were carried out using load control, with a load limit of 5mN.

2.4.6 Degree of Cure, Void Volume (V_v) and Fibre Volume (V_f) Content

A Perkin Elmer DSC 6000 was used to identify the material's T_g and degree of cure. Differential scanning calorimetry (DSC) was carried out across the laminates at three different locations, and three samples per location. The degree of cure was calculated by comparing a reference enthalpy value obtained from a semi-cured (the term 'semi-cured' describes the prepreg's stage of cure) sample with a second enthalpy value obtained from a cured sample. The sample was heated from 20°C to 250°C at a rate of 10°C/min. Using the heat flow curve obtained, the peak area was calculated between points A and B using a sigmoidal baseline. Points A and B are locations where the heat flow curve flattens to produce a horizontal tangent. The 'delta H (enthalpy)' value obtained was ~191 J/g. This was the reference value chosen for the calculation of degree of cure.

A second enthalpy value was obtained by analysing a cured sample under the same dynamic analysis as the reference sample. In order to obtain the degree of cure of sample 'x', the delta H value measured from the sample 'x' was compared to the reference sample's delta H value (Eq. 11).

$$\text{Degree of cure } (x) = \left(1 - \frac{\text{Delta H } (x)}{\text{Delta H } (\text{Reference})} \right) \times 100 \text{ (Eq.11)}$$

Optical microscopy and image analysis was used to determine the V_v and V_f values based on ASTM E2109-01 (2007) [39]. Three sections of each of the laminates were prepared, and six non-overlapping images were taken, i.e. a total of 18 images were used from each laminate.

3. Results

3.1 MW Penetration Depth

The results of the change in water temperature as a function of laminate thickness can be seen in Table 3 and Fig. 2. The average and standard deviations are from three tests. The reference tests showed a high (~19°C and ~11°C) and a low (<0.3°C) temperature change for the R1 (perforated aluminium plate only, i.e. full MW penetration) and R2 (solid aluminium plate only, i.e. no MW penetration) tests respectively. The change in temperature as a function of laminate thickness showed approximately an exponential decay pattern, thus agreeing well with the theory described in §2.3. The theoretical MW penetration depth based on Eq.1 is 1mm or less than 0.1mm assuming $\mu=1.26 \times 10^{-6} \text{H/m}$, and $\sigma=2 \times 10^4 \text{S/m}$ or 100S/m at a frequency of 2.45GHz depending on whether the electric field is parallel or orthogonal to the carbon fibres [29]. In order to compare the theoretical and practical D_p values, it is first necessary to multiply the theoretical value by 1.37 (or multiply the practical value by 0.63) due to the e^{-1} relationship, i.e. the

practical method to measure the MW penetration depth is a maximum, whereas the theoretical value is e^{-1} , and thus the difference is 0.11mm since $1\text{mm} \times 1.37 = 1.37$, or $2\text{mm} \times 0.63 = 1.26$.

3.2 Degree of Cure, Void Volume (V_v) and Fibre Volume (V_f) Content

The oven cured sample's degree of cure for a 45 minute dwell at 120°C was ~95%, thus agreeing with the values provided by the manufacturer. The MW cured samples' degree of cure ranged between 82% (30min at 120°C) and ~97% (45min at 120°C). The largest increase (~9%) was observed between 30 and 35 minute dwell at 120°C. 40 minutes at 120°C in the MW achieved the oven sample's degree of cure. A 40 minute MW dwell at 120°C provided very similar longitudinal and transverse tensile performance compared with the oven cured samples. The 45 minute MW dwell at 120°C also produced similar results. The oven cured samples obtained a V_f and V_v of ~46% (deviation of 4.5) and 0.8% (deviation of 0.8), whereas the MW cured samples obtained values of ~41% (deviation of 4.5) and 2% (deviation of 1.2) respectively.

3.3 Mechanical Testing

3.3.1 Tension

The tensile test results (Fig. 3) showed that the maximum longitudinal ultimate tensile strength (UTS) of MW cured samples was similar compared to oven-cured ones, which is similar to what was presented previously by Kwak et al [3]. On the other hand, the average transverse UTS of oven cured composites was slightly greater (~11%) than the highest average of the MW cured composites.

Little change in longitudinal and transverse elastic moduli was observed across all the samples regardless of the curing method and curing cycle. There were no indications of large variations in strength or modulus across the laminate for both the MW and oven cured samples (Fig. 4).

3.3.2 Compression

All four sets of MW cured samples showed greater average ultimate compression strengths (UCS) compared with the oven cured samples, with the MW cured samples showing a slightly greater standard deviation (Fig. 5). Sample S7 (40min at 120°C MW cured) showed an average UCS of 1159.6MPa compared with sample S21's (45min at 120°C oven cured) average of 857.6MPa, i.e. an increase of ~35%. As with the modulus values obtained under tensile loading, no significant changes were observed.

3.3.3 In-Plane Shear

The average IPS test results (Fig. 6) showed little variation in in-plane shear strength (IPSS) (~9%) and IPS modulus (~2%) regardless of the heating method.

3.3.4 Nano-indentation

The elastic modulus of oven and MW cured laminates were calculated from the reduced/indentation modulus using Eq. 10. There was little variation in results irrespective of the heating method. The difference between the minimum (oven cured) and the maximum (MW cured) laminate was 13%. The average oven and MW cured elastic modulus was 4.7 and 4.9GPa respectively. By applying the rule of mixtures to the elastic modulus values obtained in §3.3.1 for a V_f of 50%, the elastic modulus of the matrix is 4GPa, i.e. 15% lower than the values obtained using the indentation method.

3.4 Failure Mode Assessment

Some similarities and differences were observed between the conventional oven cured and MW cured samples when the failure mode was assessed using SEM. Representative SEM images of the 90° tensile tests for the oven and MW cured with 40 and 45 minute dwell time can be seen in Fig. 7. Some of the areas of interest have been highlighted in the figures with labels (i), (ii) and (iii), representing fibre disbonding, matrix remaining on fibre surface, and matrix roughness, respectively. Only some of these features have been highlighted in the figures for clarity purposes.

Visual SEM inspection of the fracture surfaces show that although both oven and MW cured samples suffered from apparent fibre disbonding, this was much clearer in the oven cured samples. Additionally fibre-matrix interfacial failure was predominant in the oven cured samples as observed by the limited matrix remaining on the fibres. MW samples however showed both a high and low degree of matrix remaining on the fibres even on the same sample. Samples S16 (45min at 120°C) and S6 (40min at 120°C) showed fibres that were almost fully coated. A cohesive failure mode (i.e. matrix failure) was more predominant on MW cured samples, however some clean fibre with little (or no) matrix was also observed, suggesting the existence of a multi-phase failure in the MW cured sample. Overall, the degree of matrix roughness between the samples is similar, however MW cured samples showed both a rough matrix fracture surface with indications of highly deformed matrix material and ribbons (i.e. signs of ductile failure) and a smooth matrix fracture surface (i.e. brittle failure) even in the same sample.

4. Discussions

The MW penetration depth tests suggest that approximately a maximum of 2mm will heat through the thickness of the UD SPARPREG CFRP. This, however, can be multiplied by two if using a tooling material that has low MW absorption, since the VHM system has magnetrons all around the chamber. Additionally, it could be argued that for this same reason, the thicker the laminate, the greater the heat generated by the MWs interacting with the sides of the laminate, thus there may be a thickness at which the penetration depth value becomes less critical. Compared to the theory, the difference of 0.11mm (i.e. 11%) is small. The direction of the waves (i.e. parallel or orthogonal) to the fibre direction has less relevance in the VHM due to its field homogeneity. The temperature increase of 0.3°C observed in 'R2' is believed to be due to resistive heating on the aluminium casing due to the MWs. It is commonly said that

metals reflect MWs, and this is a safe assumption for most cases, however for experiments such as the one described here, it is noticeable that some interact within the metal 'skin', causing heating.

In terms of MW curing, Lee and Springer [17] was only able to cure UD laminates, however in the present study both UD and $\pm 45^\circ$ laminates were successfully cured, similar to Wei et al [21], although Wei et al's laminates were only 76x76mm. The low V_f and high V_v values obtained from MW cured composites (high V_v also reported by Nightingale and Day [20], although this was possibly due to the complete lack of vacuum) is possibly due to; i) limitations in debulking due to the epoxy tape, and; ii) the five times greater heating rate than the oven cured samples, which would have greatly reduced the time until vitrification of the matrix, further limiting the time to remove the trapped air. This is more severe if we take into account that the temperature at the fibre-matrix interface is greater [15].

The 0° tension test results showed little difference regardless of the heating method, as also shown by Kwak et al [23], which is unsurprising since this is a fibre-dominated test, thus raising a question of why Paulauskas [22] reported a decrease of 10% with MW cured samples. The matrix (and/or interface) dominated 90° tension test showed that the MW cured samples produced slightly lower strength values than the oven cured ones, thus producing similar results to Paulauskas [22], but the difference is within experimental error, especially when taking into account the limited number of coupons (i.e. six), and the highly sensitive nature of this test to factors such as voids and other defects. It is believed that the increased IFSS of the MW cured samples were counter-balanced by the increase in V_v .

The 0° compression test results showed the greatest difference between the oven and MW cured samples. The hypothesis is that the brittle fibre-matrix interface held the fibre in place for longer, thus delaying the fibre buckling/kinking process. Due to the selective heating nature of MWs (i.e. predominant heating from carbon fibres to the matrix, potentially providing a higher temperature at the fibre-matrix interface), it can be deduced that all MW cured coupons produced greater compressive strengths because the matrix close to the carbon fibres had, assuming an Arrhenius relationship, a relatively high degree of cure.

Therefore it can be further deduced that the further increase in compressive strength observed with increasing dwell time by the MW cured coupons was due to the increase in degree of cure of the matrix away from the fibres after the interface reached a high degree of cure. This is quite a remarkable result taking into account the greater V_v content in MW cured samples should in theory produce lower compression strengths. The significant enhancement in compression strength may indicate that MW cured

composites should perform well in compression after impact tests. These results could have a large impact in industries where CFRPs are normally preferred due to its advantages in compressive properties. The increase in 0° compression strength of MW cured samples could lead to changes in design allowables, which in turn could lead to thinner sections, thus offering cost and weight reduction.

The ±45° tensile IPS tests showed consistent results independent of the heating method. The variation was also within experimental error. The little variation in all test results may be an indication that larger laminates can be produced with the same methodology.

The elastic modulus of the matrix for the oven and MW cured samples obtained using nano-indentation showed little difference, and the values were slightly greater than expected. This could be due to a fibre stiffening effect, where the close proximity of carbon fibres may potentially limit the 'flow' of the matrix from deforming as the indenter penetrates the material. Lower indentation depths may potentially minimise this effect, however at small depths the indentation may suffer from indentation size effects, where the results become very sensitive to the surface roughness caused by the preparation of the sample. When assessing the failure mode of the tested samples, there are some differences between the samples, albeit relatively minor, which is to be expected since the degree of cure values were all quite high, except for S12-5 where the degree of cure was 82%. Most noticeably the oven-cured samples showed relatively clean fibre surfaces with little matrix remaining compared with the MW cured fracture surfaces – which is similar to what was reported by Agrawal and Drzal [15], and Wei et al [21]. This is an indication of the tough(-er) matrix of the oven cured sample, since there is a tendency for fracture to occur preferentially at the fibre-matrix interface as the toughness of the material increases [40-42]. Multi-phase (i.e. brittle and ductile) failure regions were present on the same MW cured samples, which could be due to the selective heating nature of MWs, where the brittle region could be caused due to the predominant heating of the MWs from the carbon fibres to the matrix, and the ductile region could be due to the predominant conduction/convection heating. Therefore, using a technique such as DSC to identify the MW cured composite's degree of cure may not show the true state of the material, as the sample cut for investigation will possibly contain many interface/interphase regions and regions away from the interface/interphase, i.e. DSC shows an average degree of cure of the sample.

5. Conclusions

The literature review highlighted the main results obtained by previous researchers in the field of MW curing of CFRPs, but it is difficult to provide specific conclusions due to the differences in hardware used.

In addition, the importance of MW penetration depth has been emphasised and a methodology to practically measure/estimate this parameter on composite materials has been proposed and deployed.

The tension, compression, IPS and (local) elastic modulus properties of oven cured samples were compared against MW cured samples. SEM was used to investigate the fracture surface of transverse tensile tested samples.

Limited publications in MW heating of CFRPs were identified and the discrepancies in the results were due to unsuitable MW hardware and operational methodologies, therefore making it inappropriate to derive clear conclusions. The operational methodology reported by Kwak et al [3] provides a basic procedure that can be easily and consistently reproduced in the future. Following such procedure, the tensile and IPS properties showed little difference between MW and oven cured samples. The difference in mechanical properties in compression was significant, and the consistency in test results observed in MW cured composites was an indication of even heating through and across the laminate.

The selective heating nature of MWs (predominantly from the carbon fibres to the matrix in the case of CFRPs) created local variations in material ductility, indicating that the matrix is comprised of a two-phase system with a hard brittle region close to the fibre, and a more ductile and tougher region away from the fibre. It is believed this was the main reason why MW cured samples produced significantly increased compressive strength values. This is evidenced by the fact that; i) all MW cured samples performed better under compression than the oven cured samples, even the sample with 82% degree of cure, ii) indentation tests demonstrated there is very little difference in matrix modulus, and, iii) SEM shows significantly higher degree of matrix remaining after testing of MW cured samples.

The suitability of MW-heating of a material (particularly CFRPs) need to be investigated on a case-by-case basis, i.e. pre-work is required prior to attempting to process materials using MWs, since the global and local MW field will vary depending on factors such as the part's geometry, temperature, dielectric and conductivity properties.

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Fig. 1 Schematic for testing the MW penetration depth of CFRPs

Fig. 2 Change in water temperature as a function of laminate thickness

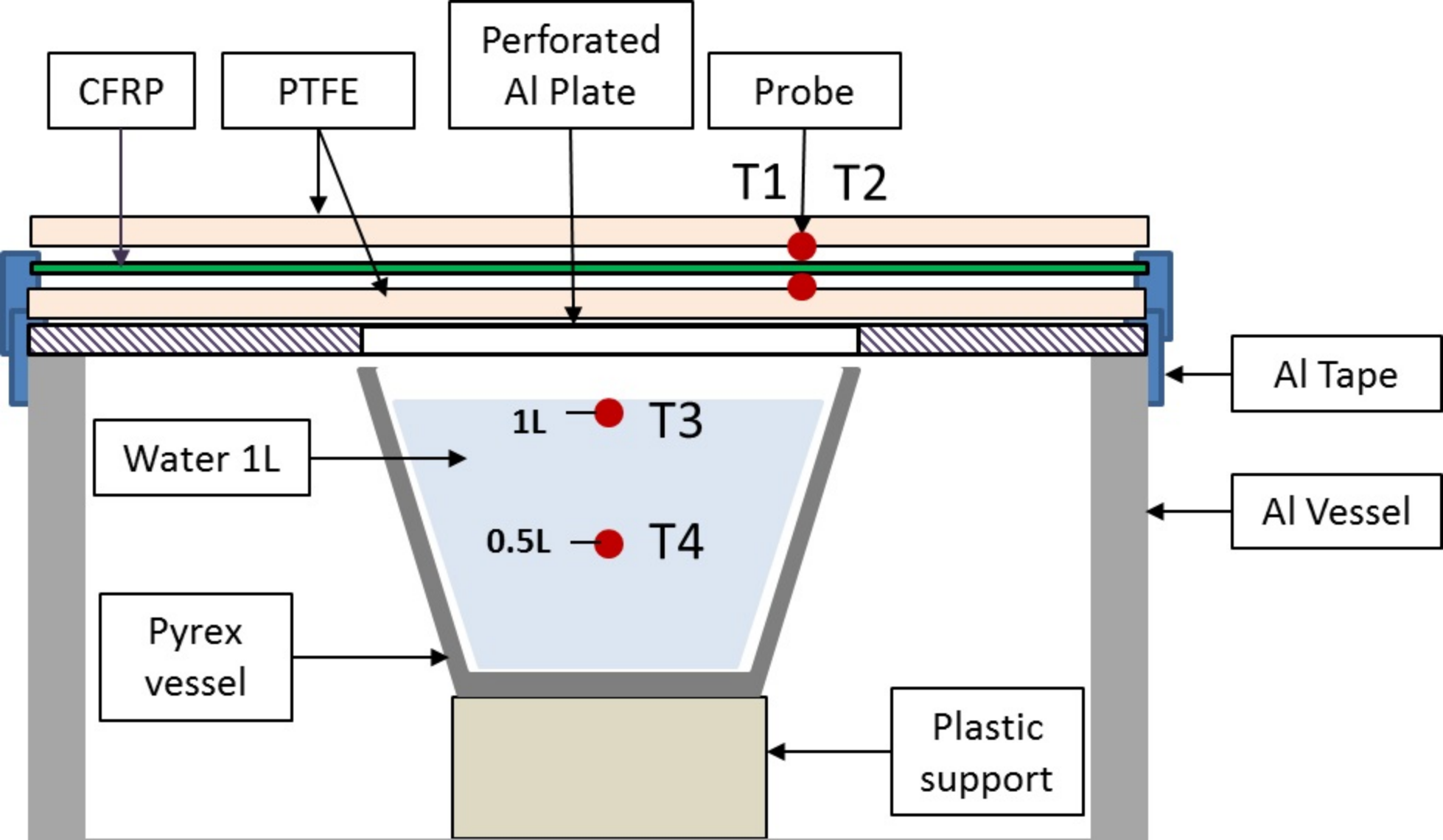
Fig. 3 Fig. 3 Longitudinal (Top) and transverse (Bottom) tensile strength (Left) and modulus (Right), and degree of cure, of MW and oven cured samples

Fig. 4 Fig. 4 Longitudinal tensile strength and modulus of MW and oven cured samples as a function of coupon location

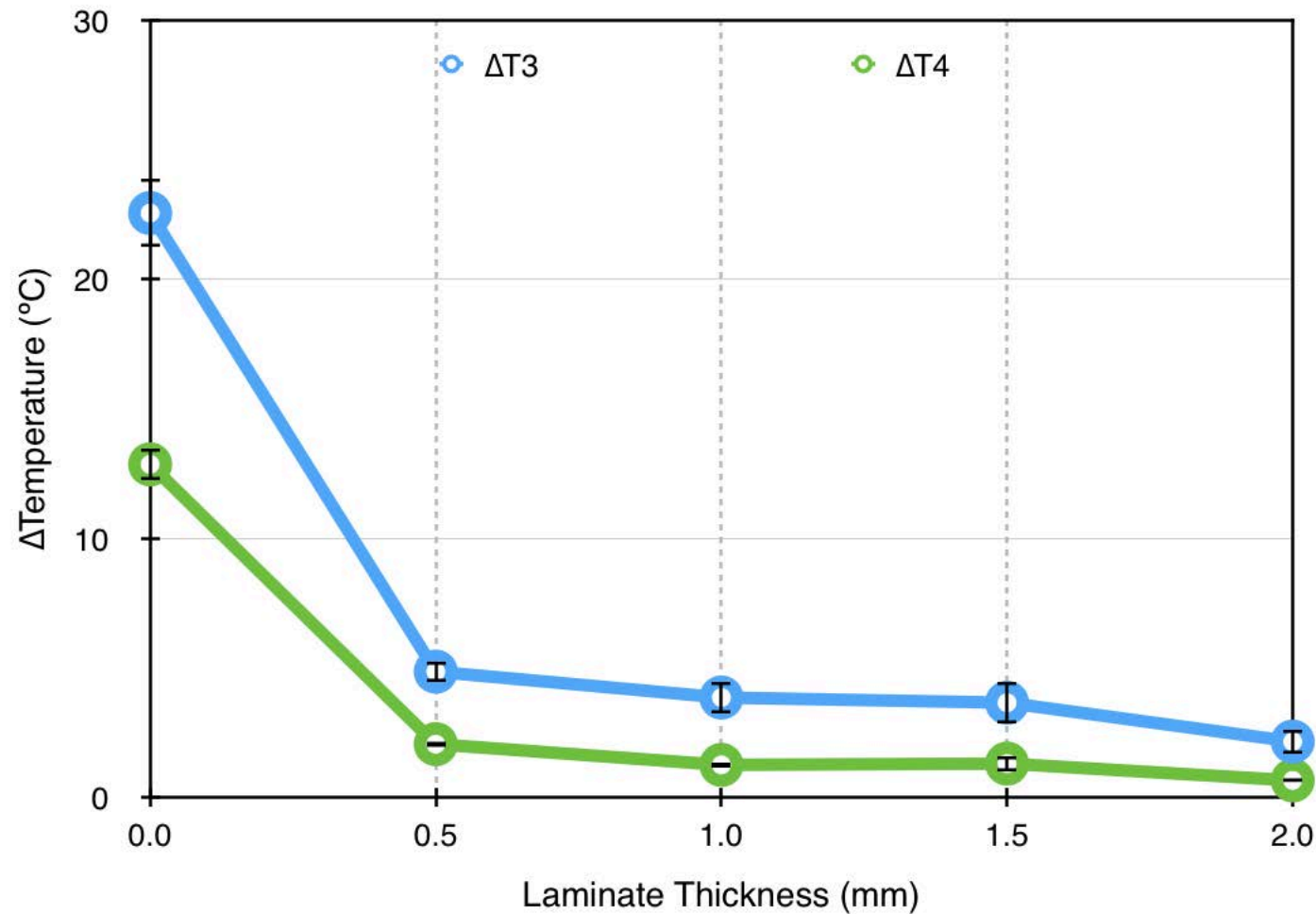
Fig. 5 Longitudinal compression, left compressive strength, right compressive modulus

Fig. 6 In-plane shear strength and shear chord modulus of elasticity

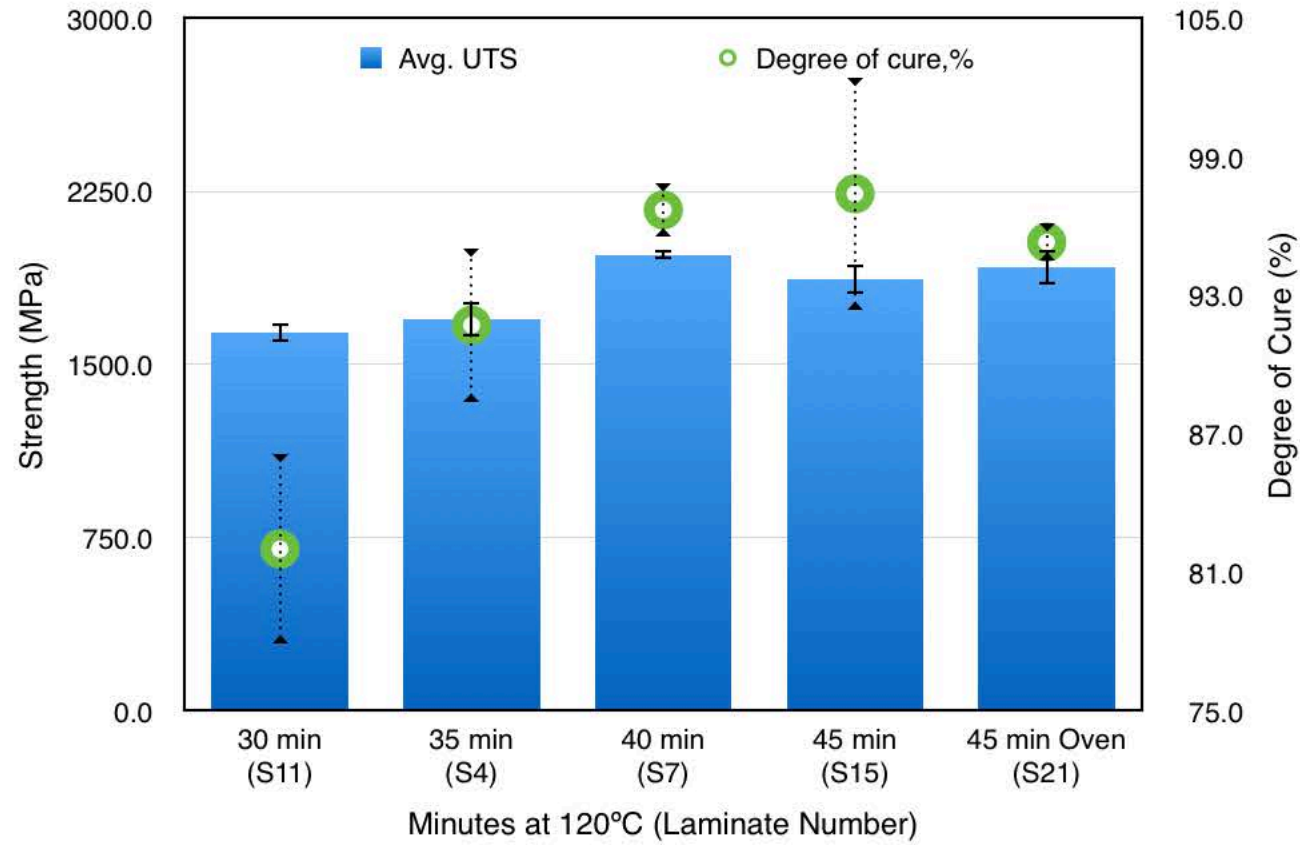
Fig. 7 Fracture surface SEMs of 90° tensile test samples (a) Sample S22-5, 45min at 120°C (Oven cured); b) Sample S16-5, 45min at 120°C (MW cured); c) Sample S6-5, 40min at 120°C (MW cured)) showing; (i) fibre disbonding, (ii) matrix remaining on fibre surface, and, (iii) matrix roughness.



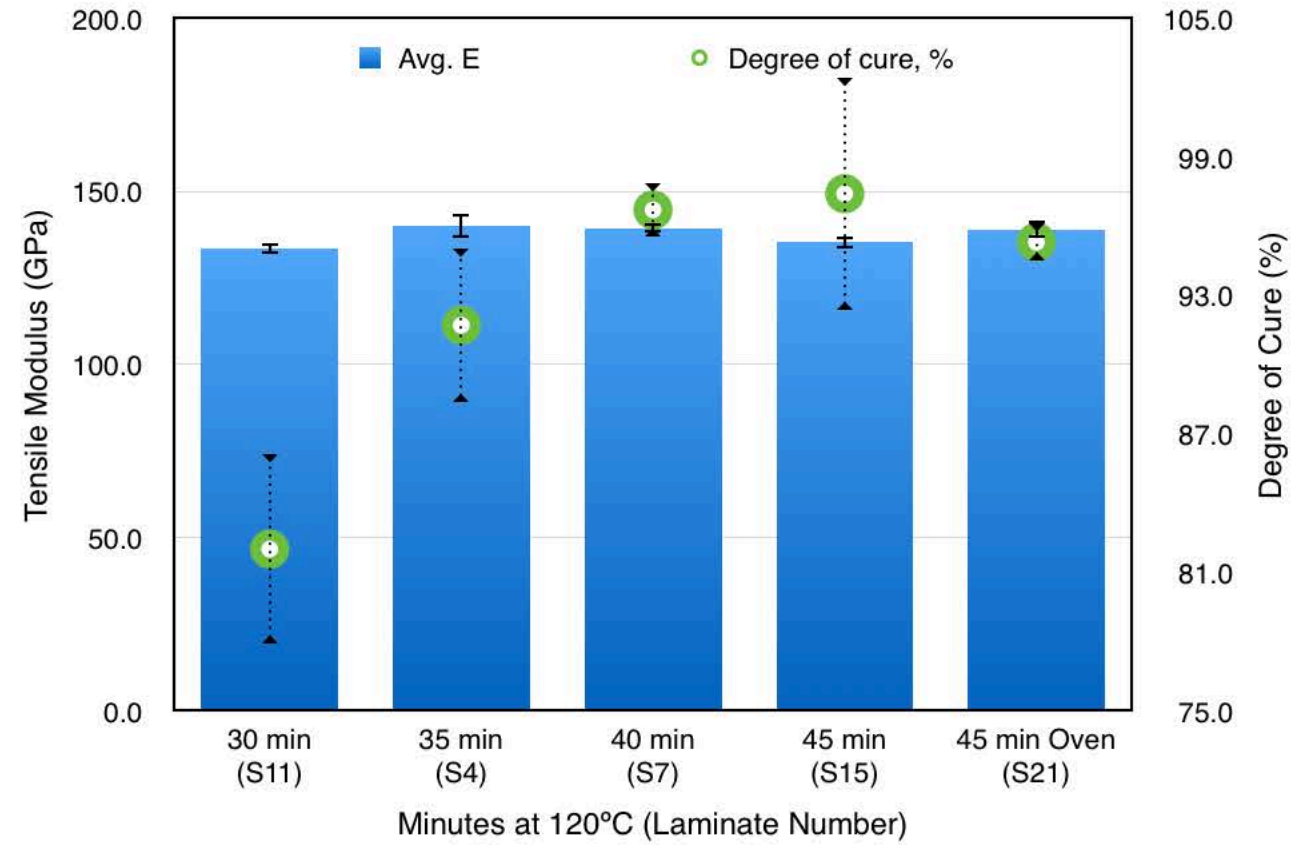
Change in Water Temperature as a Function of Laminate Thickness



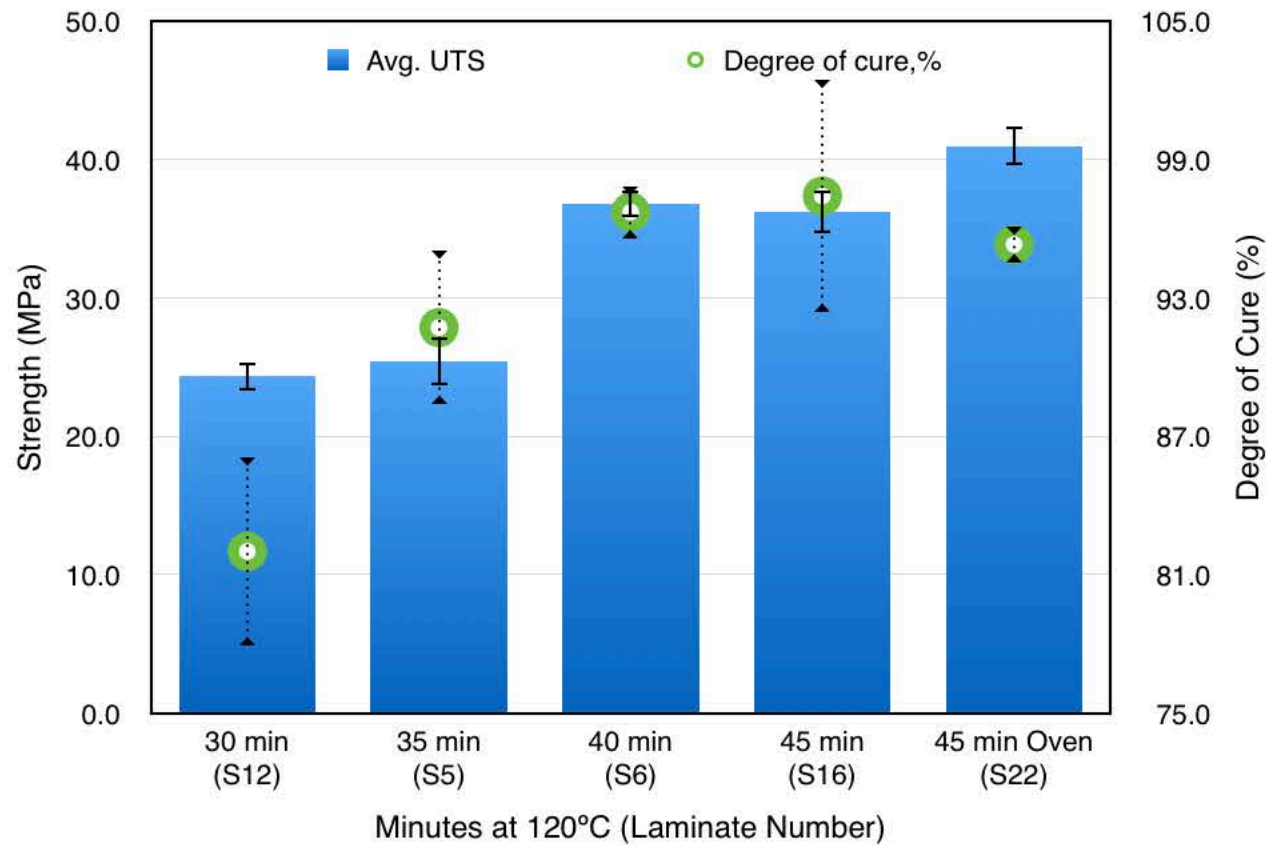
0° Ultimate Tensile Strength



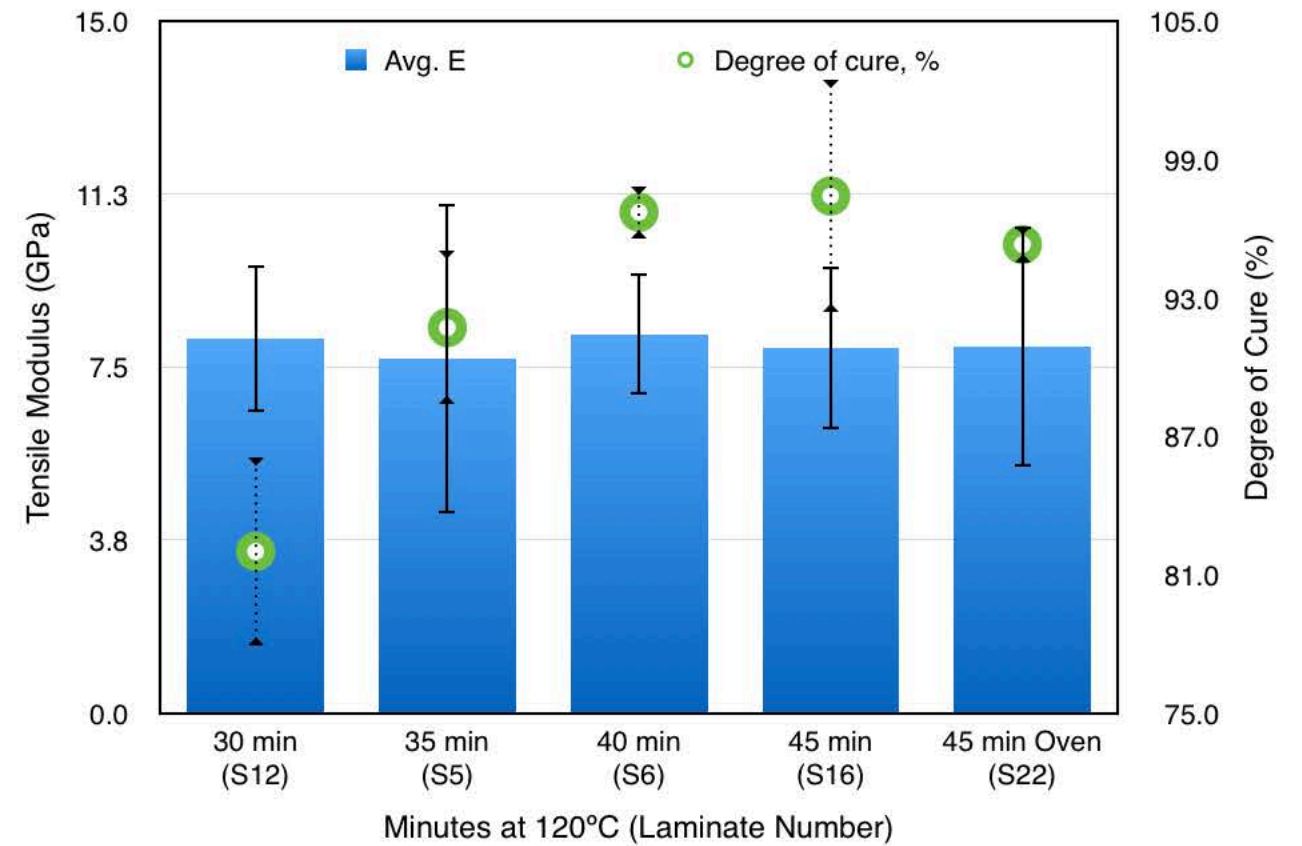
0° Tensile Elastic Modulus



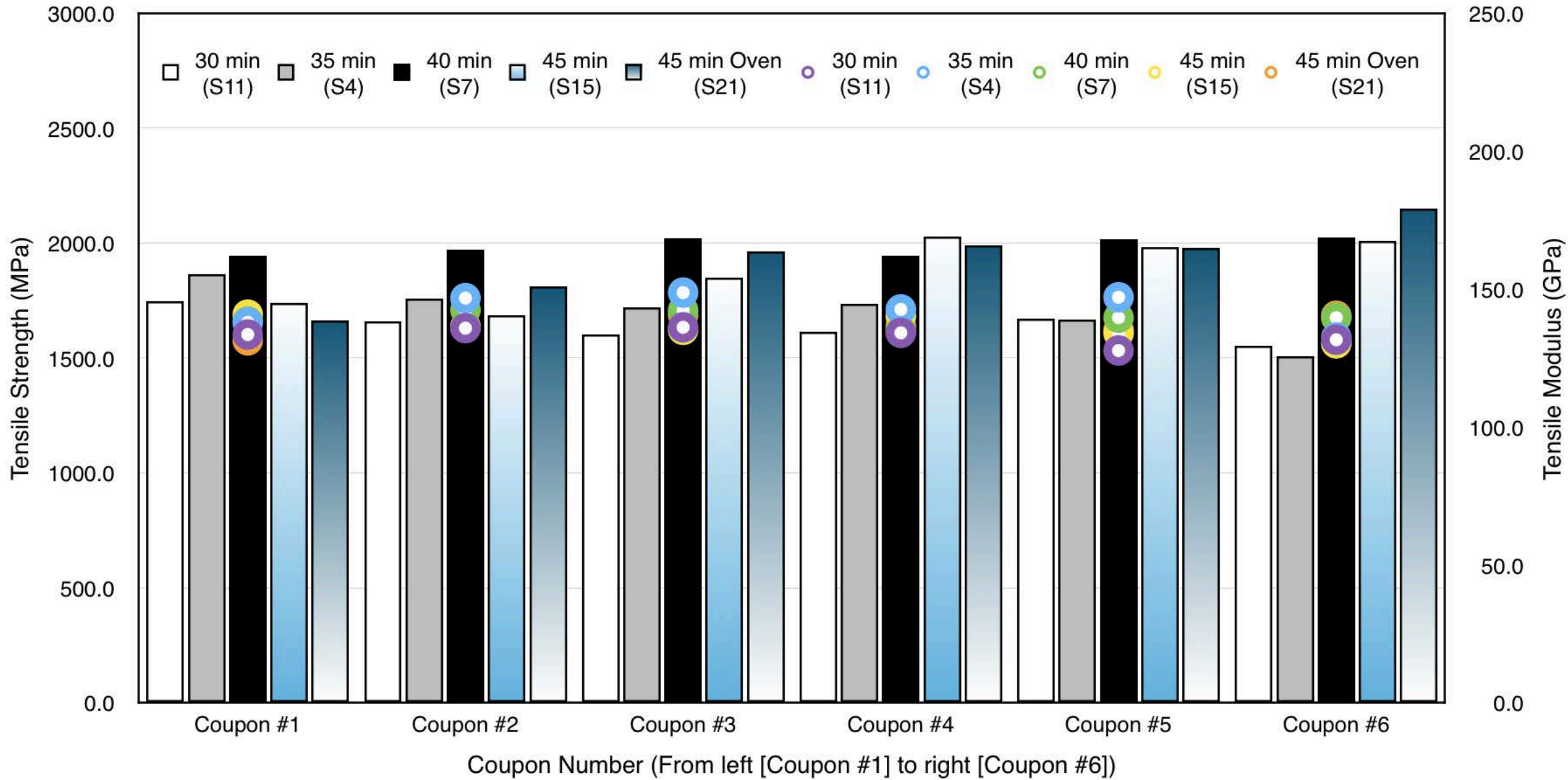
90° Ultimate Tensile Strength



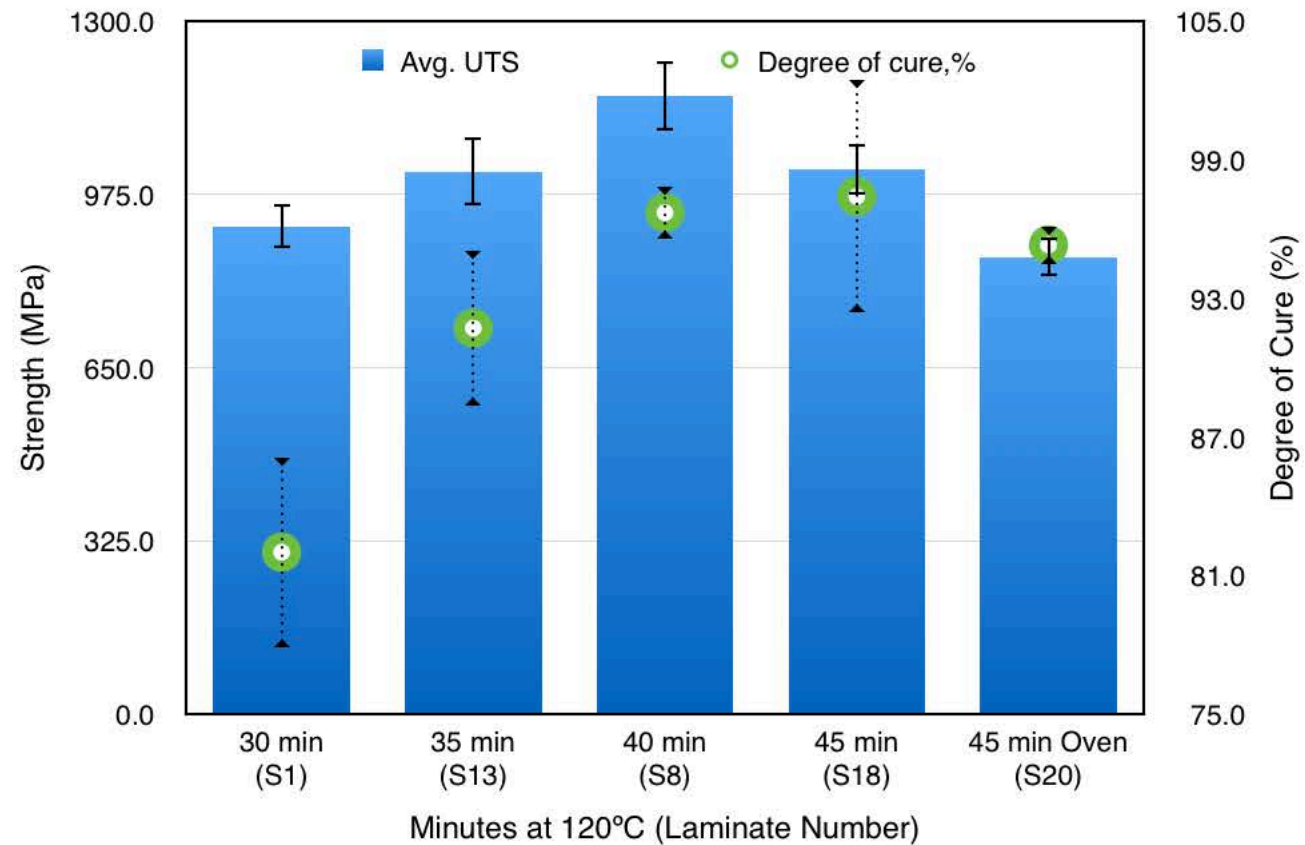
90° Tensile Elastic Modulus



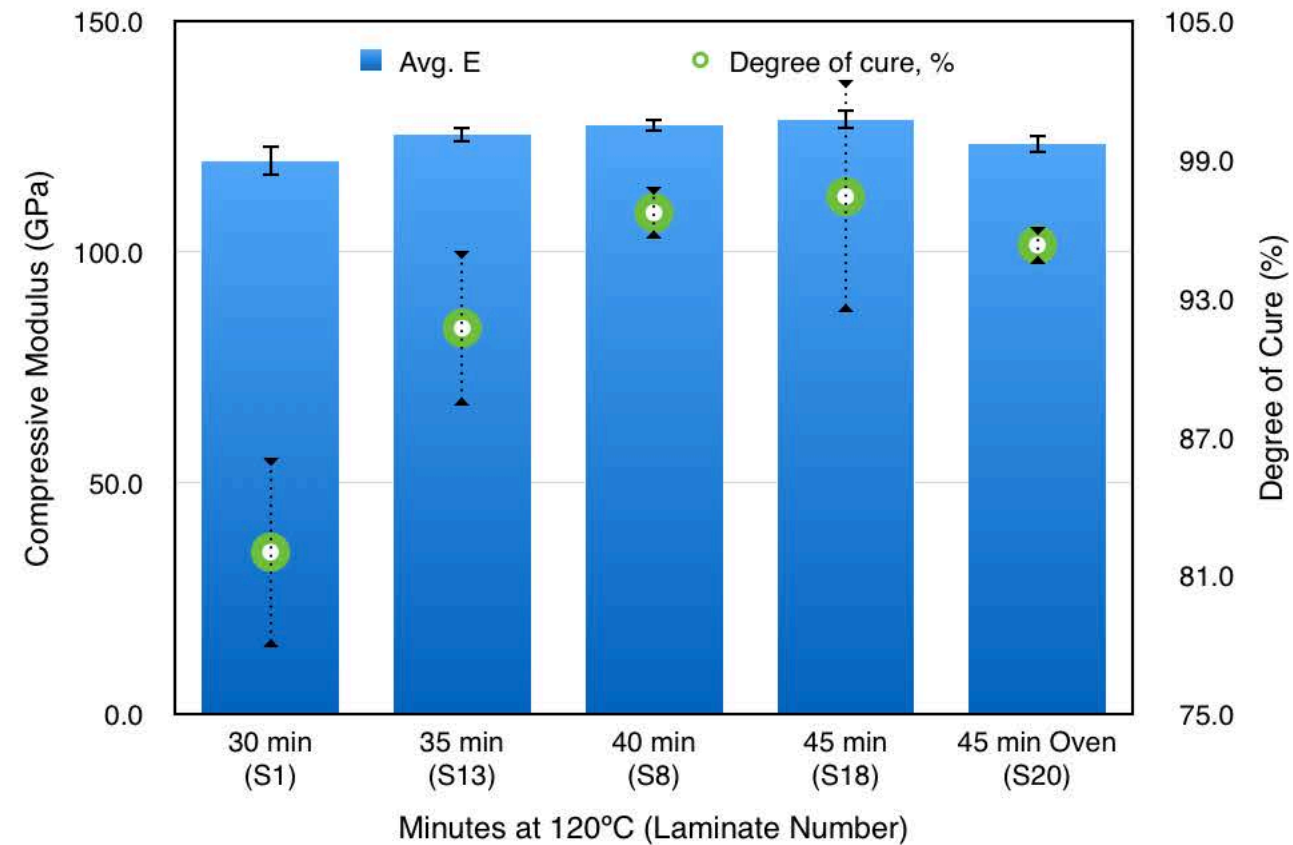
Tensile Strength (Columns) and Modulus (Circles) as a Function of Coupon Location Across Laminate



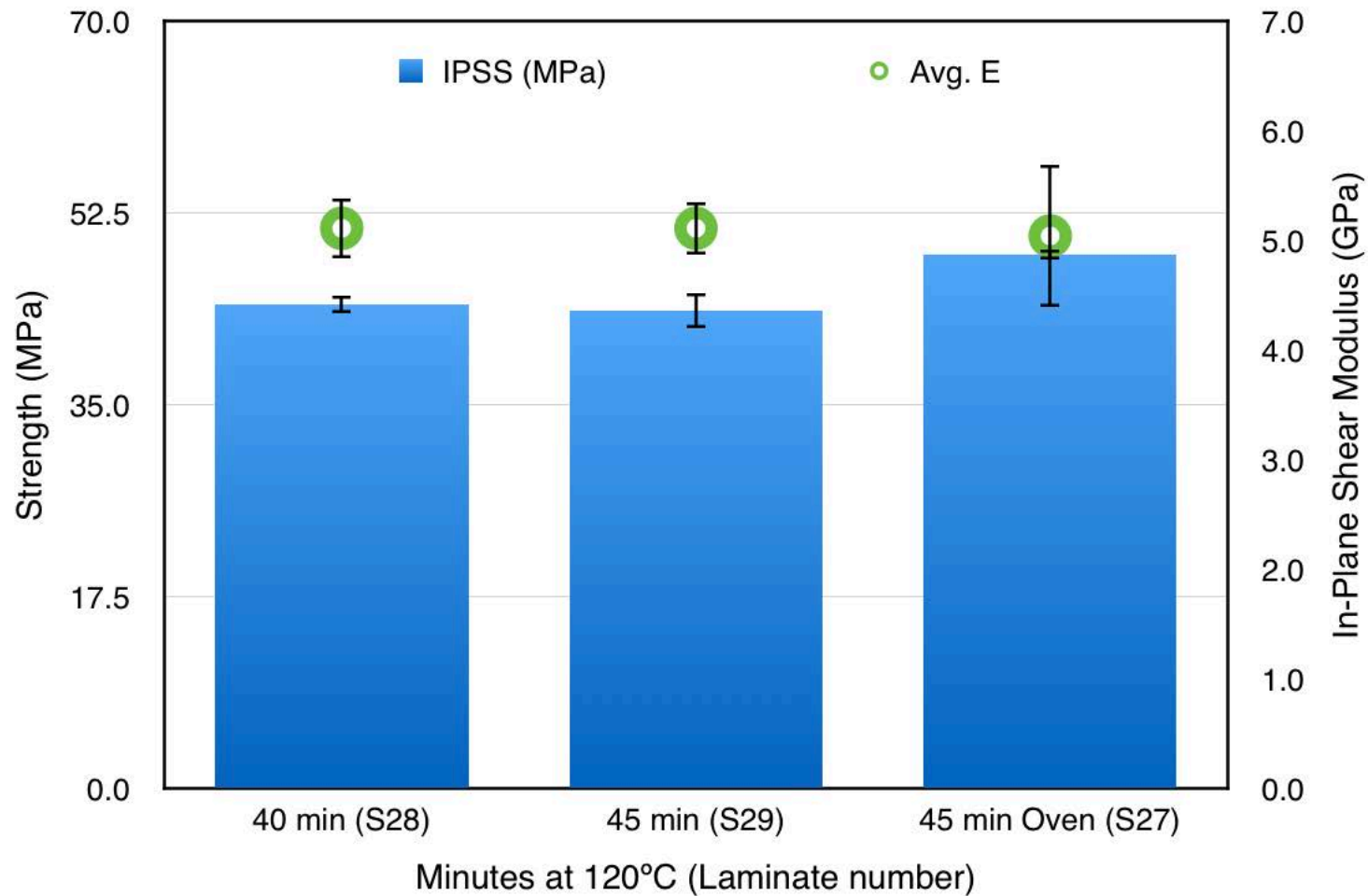
0° Ultimate Compressive Strength



0° Compressive Elastic Modulus



In-Plane Shear Strength and Modulus



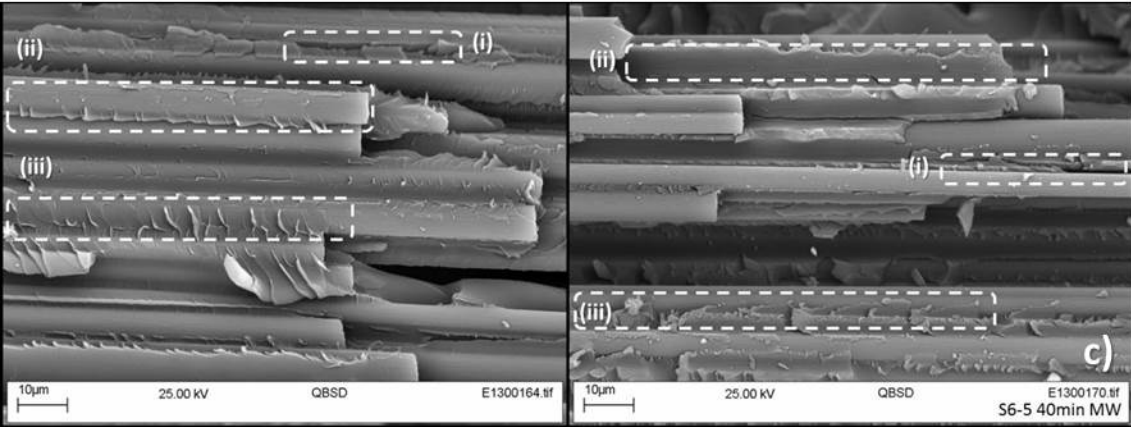
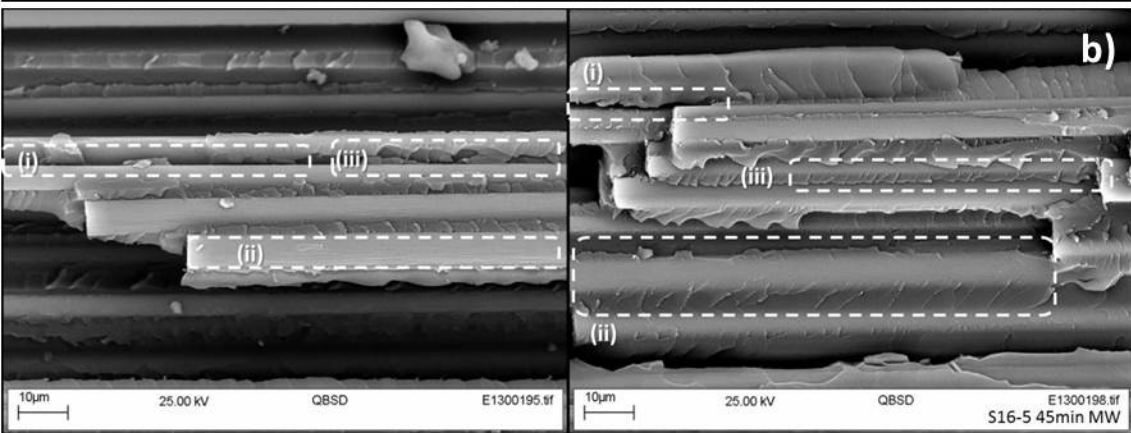
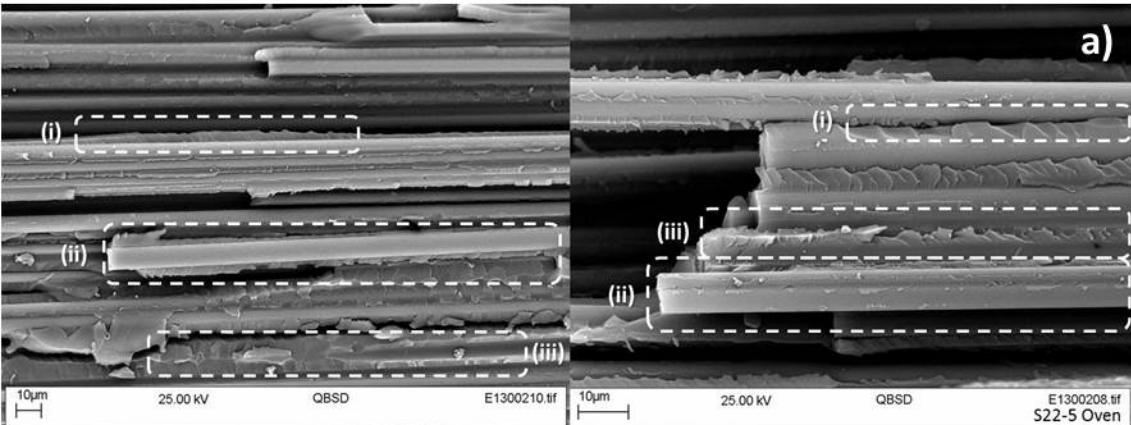


Table 1 Summary of past work on MW curing of CFRPs

Table 2 Information regarding the oven ('O-') and microwave ('MW-') cured laminates

Table 3 Change in temperature as a function of laminate thickness

Material Investigated	Properties Investigated	
	T _g	Mechanical Properties
Carbon-Epoxy [3]	Similar	0° and 90° tensile strength similar.
Carbon-Epoxy [14]	MW higher	At room temperature: No change in flexural strength and moduli. At 177°C: Flexural strength and modulus greater in MW.
Carbon-Epoxy [15]	-	MW increase fibre-matrix IFSS of CFRP (compensate for lack of pressure, increase in IFSS by 70%).
Carbon-Epoxy [17]	-	-
Carbon-Epoxy [18]	Similar	Flexural modulus and strength similar using normalised V _f . MW ILSS higher by 9%.
Carbon-Epoxy [19]	-	No pressure in MW gave similar strength as autoclave processed (100psi).
Carbon-Epoxy with PEI [20]	-	Flexural strength, flexural modulus and ILSS: Autoclave highest, MW post-cured autoclave worst, MW processed mid-range.
Carbon-Epoxy [21]	-	-
Carbon-Epoxy [22]	MW lower	At 5±1.0GHz: UTS @ 0° and 90°: MW cured samples lower by ~10%. At 13.8±1.0GHz: UTS @ 90°: MW lower by ~7%.
Carbon-Epoxy [23]	Similar	G _{1C} and fibre-matrix IFSS higher with MW

Table 1

Remarks
Little variation in T_g regardless of MW cure cycle employed.
Identical or higher curing degree obtained with MW. No substantial differences in curing kinetics.
Failure mode changed from interfacial to matrix (model composite, single fibre critical length test).
CFRP UD only. MD unsuccessful.
Ten different excitation frequencies ranging from 4 to 8 GHz. 50% cure cycle reduction. Low void content (2%). Improved fibre wetting and less fibre pull-out.
76x76mm laminate. Up to 32 plies possible; enhanced fibre matrix bonding
High void content on MW samples.
76x76x38mm laminate. Fully-cured cross ply laminate. Failure mode changed from mixture of interfacial and matrix to
Variable frequency microwave (5.0GHz centre frequency, range 1.0GHz; and 13.8GHz centre frequency, range 1.0GHz).
Practically linear relationship between G_{IC} and IFSS. Oven-cured samples more prone to crack jumps than MW-cured

Table 1 (cont)

Cure Method-Dwell Time	Tension 0°	Tension 90°	Compression 0°	IPS (±45°)	Dimension (mm)
MW-30	S11	S12	S1	-	300x300x2.4
MW-35	S4	S5	S13	-	
MW-40	S7	S6	S8	S28	
MW-45	S15	S16	S18	S29	
O-45	S21	S22	S20	S27	

Table 2

	ΔT_3 , Average (°C)	ΔT_3 , Deviation (°C)	ΔT_4 , Average (°C)	ΔT_4 , Deviation (°C)
0 Plies (R1)	22.6	2.6	12.9	1.2
0 Plies (R2)	0.3	0.0	0.1	0.0
1 Ply (~0.5mm)	4.9	0.8	2.1	0.1
2 Plies (~1.0mm)	3.9	1.2	1.3	0.2
3 Plies (~1.5mm)	3.7	1.6	1.3	0.6
4 Plies (~2.0mm)	2.2	0.9	0.7	0.1

Table 3