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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.

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 500× 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 h 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.

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- 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. 125 mm), 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 (50 mm+) 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 $76 \times 76 \times 38$ mm – 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_{1C} indicated an apparent linear increase with fibre–matrix interfacial shear strength (IFSS), where the MW cured G_{1C} was greater than the oven cured G_{1C} 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. 600 g/m^2 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 255 GPa, tensile strength of 4.3 GPa, fibre density of 1.8 g/cm^3, and cured ply thickness (CPT) of 0.6 mm [27]. Four plies were laminated to produce 2.4 mm thick laminates for the tension, compression and in-plane shear samples. Two plies were first debulked in a vacuum table for 30 min, 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 $[0°]_4$, whereas the laminates produced for the in-plane shear tests had a stacking sequence of $[\pm 45°]_S$.

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)