

CURING OF COMPOSITE MATERIALS USING THE RECENTLY DEVELOPED HEPHAISTOS MICROWAVE

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Abstract

Carbon-fibre reinforced epoxy prepreg laminates were cured using the VHM (Vötsch Hephaistos Microwave) microwave, which the manufacturer claims has tackled key issues associated with microwave processing of composites, such as non-homogeneous microwave distribution, the difficulty of using metallic tooling and vacuum bagging in a microwave environment, and arcing, thus providing a very significant difference to other systems in the past. The different, and sometimes relatively inconclusive, results obtained in past work could be attributed to the different systems employed.

The VHM system has allowed researchers to more accurately assess the real effects of microwave technology for processing polymer matrix composites (PMCs). The performance of the samples produced was evaluated using differential scanning calorimetry (DSC), optical microscopy and mechanical testing. The results have been compared to samples cured conventionally.

1 General Introduction

Electromagnetic energy has been employed for many years for industrial applications, including wood drying and bread processing. There are several ways in which electromagnetic energy can be applied, such as induction, radio-frequency (RF) or microwave. The main difference is the frequency at which these techniques operate. Depending on the material to be processed, the different frequencies can offer different levels of performance. Although RF and microwave operate in a similar way, microwaves can offer better uniformity. However, both RF and microwave are usually limited to dielectric materials with specific dipolar properties.

It is possible however to process non-polar materials using microwaves through the use of additives which can absorb microwave energy and subsequently convert this into heat [1]. These additives can be dipolar, magnetic or electrical conductors.

2 Background

2.1 Microwave Processing of Materials

Advantages of microwave technology (e.g. fast, volumetric, controllable heating, etc.) have been widely known. Significant work has been carried out in the past in microwave processing of materials, both with variable frequency and fixed frequency, with some work showing partially successful results in terms of performance. With fixed frequency microwaves, there has been limited progress in terms of microwave equipment, restricting its use in industry, thus microwave technology is considered to be inappropriate for most applications. The two main obstacles for microwaves being adopted in the composites processing industry are its inability to avoid 'hot-spots' (i.e. uneven microwave distribution), and the inability to process carbon fibre reinforced polymers (CFRPs) without arcing. Variable frequency microwave (VFM) devices have overcome these two difficulties, however their high cost remains a major barrier.

2.2 Brief Summary of Past Work in Microwave Processing of PMCs

Much work has been carried out in processing both thermoset and thermoplastic composites. Attention will be paid predominantly to thermoset composites due to their relevance to the current study.

In terms of mechanical performance, significantly varying levels of success have been reported. Nightingale [2] compared autoclave cured composites to microwave post-cured, autoclaved composites and full microwave cured composites. The flexure test results showed that microwave post-cured composites produced the lowest mechanical performance, with the conventional autoclave cured composites producing the highest. Lee and Springer [3] even reported microwave curing of multi-directional composites as unsuccessful where no cure was achieved.

Other studies have shown that microwave heating produced an improvement in the mechanical and interfacial properties. This is believed to be due to better adhesion between the fibre-matrix interface (in the case of carbon-epoxy composites) – as carbon fibres absorb most of the microwaves and therefore ‘locally’ heat the interface first, as opposed to relying on conduction/convection – as reported by Wei *et al.* [4] and also possibly due to a reduction in thermal residual stress as microwaves heat through the material.

Certain studies, such as those carried out by Boey and Yue [5, 6], reported an increase in elastic modulus but a decrease in tensile strength for microwave cured composites compared with thermally cured ones.

The inconsistency in the results obtained by past research makes it difficult to draw a clear conclusion. It is believed this can be attributed to three reasons. First is due to the different equipment employed by the researchers – this is critical as in many cases microwave field homogeneity is not achieved and therefore the measured performance will depend on the location of the sample within the chamber. Secondly by the different experimental techniques employed, e.g. some used time and power as reference, whereas others used temperature; some employed pressure, others did not, etc. And finally, due to the different materials used – the curing mechanism and its interaction with microwaves differ between materials of different chemical structures.

3 Details of Equipment, Materials and Processing

3.1 HEPHAISTOS Microwave System

The VHM microwave has 12 magnetrons each with 0.85 kW of power at a fixed frequency of 2.45 GHz. The power ranges from 5% (i.e. 510 W) to 100%, with a minimum resolution of 0.1%. The system has an internal hexagonal chamber, with a diameter of 1 m and a depth of 1 m. The manufacturer claims that the microwave’s unique hexagonal chamber and waveguides allow an even microwave distribution.

3.2 Materials

The material used for this study is Gurit’s 600g uni-directional (UD) low modulus (LM) carbon fibre reinforced epoxy WE91-2 composite, typically employed for the production of wind turbine blades. The manufacturer suggests various out of autoclave (OOA) curing profiles (Table 1).

Table 1 WE91-2 cure cycles recommended by Gurit [7]

Cure Temp. (°C)	Time to 95% Cure (Minutes) (Incl. ramp-up @ 2°C/min from 20°C)
85	753
90	335
100	140
110	105
120	85

3.3 Curing Procedures

3.3.1 Conventional oven curing

The reference laminate, with dimensions of 300x300 mm, was produced using Gurit’s suggested cure cycle of 35 min at 120°C to ensure complete curing – the achieved ramp-up rate was of 1.5°C/min. Four UD 0° plies were laminated, achieving a thickness of approximately 2.4 mm. Typical consumables employed for oven curing composites were used, such as breather cloth, release ply and vacuum bag, with an aluminum base plate.

3.3.2 Microwave oven curing

The same laminate lay-up sequence and geometry was employed, i.e. four 300x300 mm 0° plies, and heated at different cure cycles (Table 2).

Table 2 Attempted microwave cure cycles

Ramp Rate (°C/min)	Cure Cycles	
	Dwell Temp. (°C)	Time at Dwell (Minutes)
10	90	170
		200
	100	60
		85
	110	40
		60
	120	20
		30
		35

The remaining setup, i.e. vacuum bagging and use of consumables, were in line with those employed for the production of the reference laminate. The most distinctive aspect of the setup is the shielding of the tips of the carbon fibres – this is imperative as without this shielding the vacuum bag may be punctured (i.e. not allowing full consolidation), and the material may be damaged due to arcing. The health and safety implications of arcing – potentially causing burns/fires in the chamber – should also not be forgotten.

The author has identified two different ways of avoiding arcing; through the use of aluminum tape or epoxy resin.

Aluminum tape is used to earth the sample and to reduce electrical stress. Any excess current at the tips of the carbon fibres are transferred to the aluminum tool and ultimately to the walls of the microwave chamber, thus avoiding charge build up and arcing. The main drawback of using aluminum tape as the shielding mechanism is the difficulty in ensuring even current distribution. The overlap area between the tape and the laminate will need to be (almost) identical on all four sides to ensure even (and consistent) heating of the laminate. An additional disadvantage is that excessive current may be lost, leading to an increase in microwave power and therefore making the process less energy/cost efficient or even unsuccessful (particularly with UD laminates). It is also believed that aluminum tape restricts the consolidation process, increasing the laminate void content.

On the other hand, the use of epoxy resin provides more consistent results, with significantly lower energy usage. The role of the epoxy is to reduce the electric charge density (at the tips of the carbon fibres) and retain the heat within the laminate. One major advantage of this is the possibility of removing a microwave reflecting material (i.e. aluminum) as a tool and replacing this with a microwave transparent material (e.g. thermoplastics, ceramics, etc.), thus further increasing the process efficiency as the aluminum tool inevitably stops some microwaves from the lower half of the chamber from reaching the material, allowing the processing of thicker parts. A material with high thermal conduction properties (e.g. aluminum) is also disadvantageous as it acts as a heat sink. This is not an issue in conventional heating processes as the entire chamber is at the same temperature. However in a microwave environment, where only the component is heated, losing heat to the tool and the rest of the chamber can be a considerable drawback, especially when taking into account that the volume of the sample to be processed will always be much smaller than the volume of the chamber.

4 Void Content Measurement

Optical microscopy and resin burn-off (ASTM D3171 [8]) was used to assess the consolidation quality. As it can be seen in Fig. 1 and Fig. 2, the difference in consolidation is similar between oven cured and microwave cured laminates.

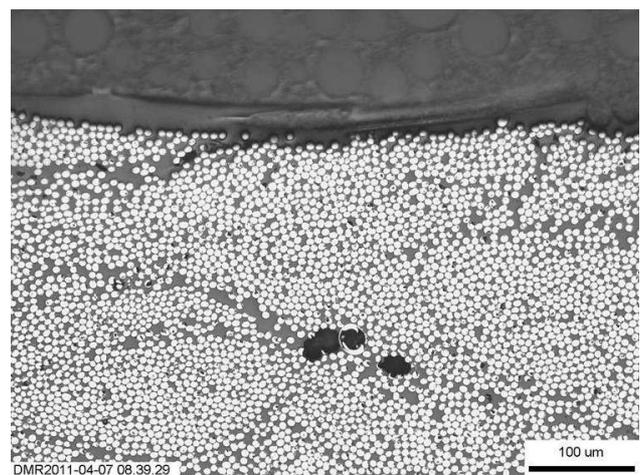


Fig. 1 An optical image of an oven cured WE91-2 laminate.

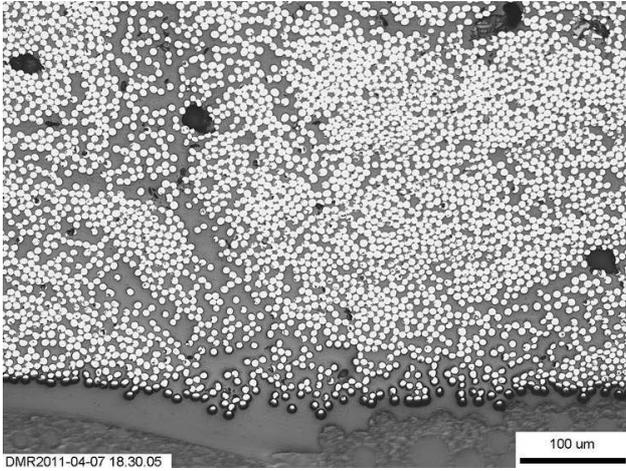


Fig. 2 An optical image of a 35 min at 120°C microwave cured WE91-2 laminate.

A resin burn-off technique was used in order to quantitatively assess the void content of the conventional oven and microwave cured composites. The process was carried out for 6 hours at 600°C under nitrogen. The sample was removed at regular intervals and weighed, until the sample weight reached a plateau. The burn-off test was carried out for the conventional oven cured sample and the 30 min at 120°C microwave cured sample.

The results show that void contents of 2.0% and 2.5% were obtained for the conventional oven cured and microwave cured samples, respectively.

5 Thermal Analysis and Mechanical Testing

5.1 DSC

A Perkin Elmer DSC 6000 was used to identify the material's glass transition temperature (T_g). DSC was carried out across a diverse range of laminates (at three different locations per laminate) processed in a conventional oven as well as in a microwave, under different cure cycles on the latter, in order to identify the degree of cure and consequently obtain the ideal cure cycle. An effort was made to cut samples as consistently as possible as DSC can be significantly influenced by sample preparation. Factors to take into account include things such as thermal damage and grease.

The samples were heated from 30°C to 200°C at 10°C/min.

5.2 Tensile Testing of Conventional Oven and Microwave Cured Specimens

The tensile strength properties of the material were determined based on ASTM D3039M [8]. An Instron 5567 B723 test machine with a 30 kN load cell for the 90° direction tests, and an Instron 8500 B530 with a 100 kN load cell was used for 0° direction tests. The test specimens had a geometry of 230x25x2.4mm. The cross-head speed was 2 mm/min. and the tensile strength was calculated from Eq. (2) [8].

$$\sigma_{max} = \frac{P_{max}}{A} \quad (1)$$

where σ_{max} is the maximum tensile strength (MPa), P_{max} is the maximum load at failure (N) and A is the cross-sectional area (mm²).

6 Results and Discussion

6.1 DSC

Although T_g values were obtained for every sample, there was little difference in T_g values from one cure cycle to another. The T_g of the reference sample cured in an oven was 119°C – Gurit suggests a T_g value of 110-125°C [7] – with a standard deviation of 4°C. The results can be seen in Table 3.

Table 3 DSC Results for microwave cured samples (T_g obtained using mid-point).

Curing cycle	Average T_g (°C)	Standard Deviation (°C)
170mins @ 90°C	119	3
200mins @ 90°C	119	3
60mins @ 100°C	119	1
85mins @ 100°C	114	6
40mins @ 110°C	120	1
60mins @ 110°C	120	1
20mins @ 120°C	120	2
30mins @ 120°C	121	2
35mins @ 120°C	121	1

Although samples cured at 120°C show slightly higher and more consistent results, it is unclear why the difference in T_g values is not greater.

6.2 Tensile Tests

The results of the 90° and 0° tension tests can be seen in Fig. 3 and Fig. 4, respectively.

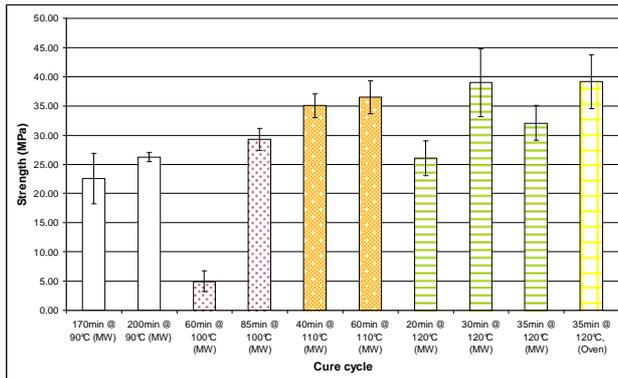


Fig. 3 Results of 90° tensile strength tests with standard deviation (MW represents microwave cured samples)

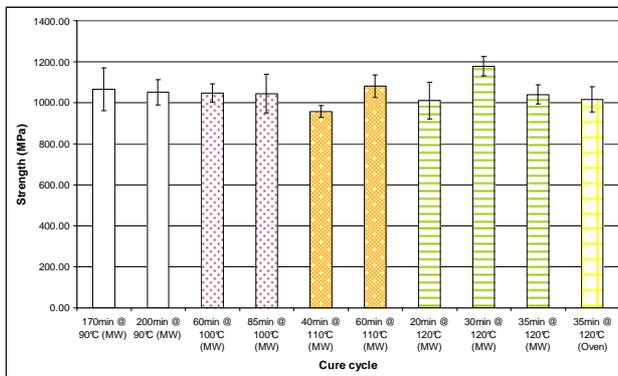


Fig. 4 Results of 0° tensile strength tests with standard deviation (MW represents microwave cured samples)

As it is only the matrix which is ‘physically modified’ (i.e. cured) – carbon fibres absorb microwaves but are unaffected by microwaves at this processing temperature (i.e. 120°C) – the difference between microwave and oven cured samples is more evident in the tensile results of the samples tested perpendicular to the fibres than the parallel to the fibres. In terms of maximum strength

in the 90° direction, the samples which were cured for 30 min at 120°C had a comparable performance to the reference oven cured samples, albeit with a slightly higher degree of variation. The rest of the samples had a significant level of difference compared to the reference samples. For the 0° tests, the variation was similar across all samples (which is to be expected as most of the load is carried by the fibres), however the samples cured for 30 min at 120°C showed a noticeable improvement compared to the reference oven cured samples. This is possibly attributed to a better load transfer (i.e. better adhesion properties between matrix and reinforcement as explained earlier) from the matrix to the fibres (through the matrix-fibre interface) as reported by Wei *et al* [4].

7 Conclusions

The VHM microwave system was used to process prepreg composite laminates. The laminates produced using this equipment has been compared with conventionally processed laminates using DSC, optical microscopy and mechanical testing.

In terms of equipment, it is believed that the setup employed in the current study for processing CFRPs in the VHM microwave can be further improved. The use of epoxy resin instead of aluminum tape has improved the consistency and efficiency of the process, however a replacement for the aluminum base still needs to be further investigated. However bearing in mind the difficulties that past researchers have had with arcing, the current setup finally allows the microwave processing of composites without arcing. Due to the application of vacuum bagging for consolidation, similar void contents were observed between the conventionally cured and microwave cured composites.

The effect of the different cure cycles was relatively clear when the samples were bent manually, where samples with shorter dwell periods had a significantly lower stiffness compared to the samples which underwent a longer dwell period. However this was not translated to a significantly different T_g value – it is still unclear as to the cause of this.

Nightingale [10] reported comparable mechanical properties (i.e. flexural strength and modulus) between conventional autoclave cured and microwave cured composites, despite the lack of vacuum and pressure on the microwave processed laminate. Therefore it is no surprise to see that the mechanical performance of the microwave processed samples investigated in the current study show slightly improved (approximately 10%) tensile strength compared to conventionally cured samples. This possibly re-affirms once again the prediction that microwave heating improves the adhesion properties between the reinforcement and matrix [4, 10]. When taking both 0° and 90° test results into consideration, it is clear that the ideal microwave cure cycle has yet to be determined. The improved performance in the 0° direction is not directly reflected on the performance along the 90° direction, which could mean that the matrix in the composites cured for 30 min at 120°C may have been embrittled due to over-curing. Based on these results, it is the authors prediction that the ideal dwell time at 120°C is approximately 30 min ± 3 min.

The current study has demonstrated the feasibility of producing composite laminates under similar conditions to those currently used in industry, obtaining similar (and in certain cases slightly better) mechanical performance. The microwave cured sample which showed highest mechanical performance, i.e. 30 min at 120°C was processed in approximately half the time of that recommended in a conventional oven, thus demonstrating that microwaves, when used appropriately, can offer significant advantages in terms of time and cost reduction (with no penalty on performance). In practice, the actual reduction in processing time is greater than this, as with microwave processing, the samples can be removed from the chamber and the process repeated straight away (during these tests the chamber temperature did not go beyond 35°C), whereas in a conventional oven, the sample can only be removed once the temperature of the chamber has reached a safe temperature.

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