A Short Review On Microwave Curing of CFRP Composites

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Abstract - Carbon fibre reinforced polymer (CFRP) composites have found extensive applications in aerospace, automotive industries, sports equipment, etc. due to their excellent mechanical properties and high strength-to-weight ratio. These composites have been traditionally cured with autoclave for years. In recent times, microwave curing has gained considerable attention among the researchers and some organizations due to their advantages like energy and space saving, reduced processing time, uniform heating and increased process control. This paper presents the review of microwave curing of CFRP composites, their cure properties and change in mechanical properties in comparison to the autoclave curing. Challenges associated with the microwave curing of CFRP composites are also addressed.

Keywords: CFRP composites, microwave curing, mechanical properties, challenges.

I. INTRODUCTION

CFRP composites have been increasingly used in aerospace, automobile and other industries due to their excellent mechanical properties and high strength-to-weight ratio [1]. These composites are fabricated using Hand layup, compression molding, filament winding and resin transfer molding, and then cured in autoclave traditionally till date. Factors like increased energy consumption, cost and high processing time involved with the autoclave curing makes the researchers and organizations to look for an alternative curing method or out of autoclave curing (CC) process to cure the CFRP composite parts. Alternative processing methods include UV cure, thermal oven cure, radiation curing, EB curing and microwave curing (MW) [2]. Among these technologies, microwave curing has gained considerable attention among the researchers and organizations due to the increased process control, uniform heating, reduced curing cycle time and energy saving [3]. A typical microwave oven consists of a magnetron which produces the electromagnetic waves, when the power is supplied to it. The produced electromagnetic waves are distributed by the waveguide system into the applicator. Temperature sensors present in the microwave oven prevents the system from overheating. Reflected electromagnetic waves from the applicator are deflected to the circulator or cooling chamber which contains absorber to absorb the microwaves [4].

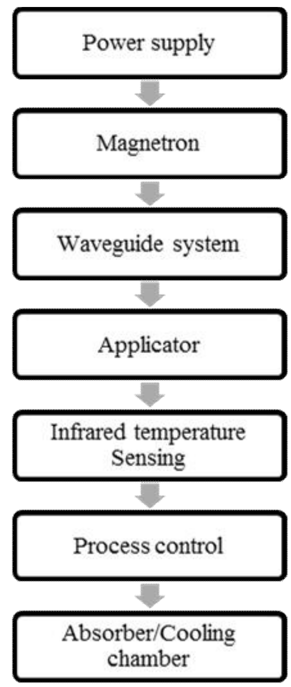

Fig 1 Components of a typical microwave oven [4]

Microwave curing (MW) relies on volumetric heating and involves the use of electromagnetic radiation to cure the composite parts. Microwave curing in the industries has been accomplished at fixed frequencies like 915MHz, 2.45GHz, 5.8GHz and 24.124GHz. Frequency of 2.45GHz has been reported by various researchers as a favorable frequency for processing of the composite parts due to its homogeneity in MW distribution [1]. According to Xu et al. and Tianchun et al, the microwave curing process is an inside-out solidification which involves the absorption of microwaves by the carbon fibres which gets heated first and then the heat is transferred to the resin matrix. In contrast, thermal curing takes place by an outside-in solidification [5,6]. Experimental analysis by Nanya Li et al. revealed that carbon/epoxy ply orientation does not affect the temperature distribution during microwave curing whereas thickness influences the temperature distribution [1]. Research work on the microwave curing of CFRP composites is limited mainly due to the following challenges: arcing, tooling design, void content and uneven energy distribution. In this paper, microwave curing of CFRP composites, their cure properties and mechanical properties and challenges associated with the technology are discussed.

II. CURE PROPERTIES

The cure properties of the MW processed samples such as degree of cure, void volume (V_v), fiber volume fraction (V_f) and glass transition temperature T_g have been evaluated by the various researchers. Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA), Fourier Transform Infrared spectroscopy (FTIR), IR and miniature gas thermometer, Optical Microscope and Scanning Electron Microscope (SEM) were utilized to determine the cure properties of both autoclave processed and MW processed CFRP samples.

a) Degree of Cure

Degree of cure is defined as the extent of curing rate obtained in terms of %, i.e. 0% corresponds to uncured and 100% corresponds to completely cured [6].

Using the formula (Eqn (1)) below, it is possible to calculate the degree of cure of sample 'x', where the delta H value is measured from sample 'x' to the delta H value of the reference sample 'H' [5,8].

$$Degree\ of\ cure\ (x) - \left(1 - \frac{\Delta H(x)}{\Delta Hreference(x)}\right) \times 100 \quad (1)$$

Differential Scanning Calorimetry (DSC) studies were carried out by many researchers to identify whether the composites are fully cured or partially cured at a given time. Xu et al. utilized DSC to identify the degree of cure. Based on their experiments, MW cured CFRP samples and CC sample showed similar degree of cure i.e. approximately 95% [5]. Experiments by Papargyris et al have shown that CFRP samples were fully cured. In their research work, the samples were heated from 20^{O}C to 250^{O}C at 10^{O}C/min in an inert nitrogen atmosphere [7]. Kwak et al also achieved nearly 97% of degree of cure with the microwave under conditions similar to that of Papargyris et al [8]. Initial results of DSC analysis on CFRP composites performed by GKN shows that microwave curing could achieve the parts quality of that of the autoclave curing [9].

Another method of determining degree of cure involves the use of Fourier Transform Infrared spectroscopy (FTIR). Sato and Hotta assessed the degree of cure through the absorbance ratio vs wave number obtained from FTIR. According to their analysis, CFRP composites remained partially cured even after the thermal curing (CC) followed by the post microwave curing [10].

b) Transition glass temperature (Tg)

Transition glass temperature (Tg) of both the autoclave and microwave cured CFRP composite is measured from thermo-gravimetric analysis (TGA) as well as DSC. Accordi.ng to Papargyris et al, Nightingale and Day, and Kwak et al, no significant changes in Tg were identified between MW and autoclave cured CFRP specimens [3,7,8]. Fang and Scola reported higher Tg for MW cure CFRP samples than the autoclave cured samples [11]. Similar results were obtained by Xu et al [6]. In contrast to the results of Fang and Scola, Paulaskas, reported lower Tg values for MW cured CFRP samples in comparison to the autoclave cured CFRP samples [12].

c) Void volume (V_v) and Fibre volume fraction (V_f)

SEM and optical microscopes are quite useful to visualize the voids that occurs in the microwave cured CFRP sample, to calculate the fiber volume fraction V_f and to study their interficial bonding properties of the failed specimen. From the microsopic results of SEM and optical microscope of CFRP samples, it is observed that small voids join together to form large voids caused due to lower resin viscosity. These large voids can have a detrimental impact on the mechanical and physical properties of the composite [5]. Despite of the higher void content, microwave cured samples have better interfacial adhesion than the autoclave cured samples. This may be due to lower resin viscosity and better resin flow resulting from higher heating rate and interface temperature during the microwave curing process, thus enhancing the interfacial bonding [5]. Similarly, Sato and Hotta studied the interfacial properties through SEM and reported better adhesion properties between carbon fibers and matrix with the microwave cured CFRP specimens [10]. V_v and V_f were measured by some researchers through SEM images and optical images. The Table 1 below shows the V_v and V_f values as reported by the researchers.

d) Cure cycle time

One of the main advantages of using microwave curing for CFRP parts is that it leads to cutback of curing cycle time. Nanya Li et al achieved 25% reduction in curing cycle time [1]. Xu at al reported 39% reduction in cure cycle time [5]. Papargyris et al. achieved 50% reduction in cure cycle time with microwave processing in comparison to the autoclave curing to acquire similar mechanical properties [7].

Author	Void volume V_v (%)		Fibre Volume Fraction V_f (%)		Measuring method
	CC	MW	CC	MW	
Kwak et al [8]	0.8	2	41	46	Optical microscopic image analysis
Xu et al [5]	6.3	7.5	---	---	SEM analysis
Papargyris et al [7]	<2	<2	---	---	Hot acid digestion method
Nightingale and Day [3]	0.1	1.5 to 19.9	---	---	Optical microscopic image analysis

Table 1 V_v, V_f as reported by the authors

e) Penetration depth

Penetration depth is defined as the depth at which the MW magnitude decreases to e^{-1} of its original magnitude. Theoretical formula for the calculation of penetration depth is as follows [8]:

$$D_p = \sqrt{\frac{1}{\Pi . f . k . \mu}} \quad (2)$$

Kwak et al reported a maximum penetration depth of 2mm [8] whereas a non-contact infrared thermometer and FBG sensor was used by Nanya Li et al to measure the surface temperature of the CFRP composites as well as the temperature distribution. Nearly 10 degree Celsius temperature difference was identified in a laminate thickness of 22.5mm [1].

III. MECHANICAL PROPERTIES

A) Tensile Properties

Based on the experimental results from Kwak et al. it was observed that there was only a little variation in longitudinal and transverse tensile modulus for both the autoclave cured and microwave cured CFRP samples whereas transverse ultimate tensile strength of autoclave cured samples were higher than the microwave cured samples [8]. Sunil and Somen studied the tensile behavior of microwave-post autoclave curing technique. They found that microwave - post autoclave achieved superior tensile strength than the individual microwave and autoclave cured specimens [13]. Brian and Jeff investigated the tensile properties of carbon fiber/epoxy composite as per ASTM D 5083-02 and found that the tensile strength was higher for autoclave cured samples than the microwave cured samples [14]. The lower strength for microwave cured samples may be due to the presence of large number of voids, since vacuum bagging was removed due to arcing. According to Tianchun et al, only little variation in longitudinal and elastic modulus was observed for both the CC and MW cured CFRP samples [6].

B) Compressive Properties

Compressive strength of the CFRP composites were found to be significantly higher than the autoclave cured samples but lower than the tensile strength of microwave cured samples. Compressive strength of the CFRP composites decreases as the porosity or void increases. This is mainly due to the lower resin viscosity and a higher interface temperature that occurs during the microwave curing. 2-step microwave curing process yielded higher compressive strength than the 1-step curing and 3 step curing process [5]. Similarly, higher compressive strength for all microwave cured CFRP samples was also reported by Tianchun et al and Kwak et al [6,8]. According to Kwak et al's findings, microwave cured matrix is comprised of a 2-phase system with brittle region close to the fibers and a ductile region away from the fibres which causes the increment in compressive strength [8].

C) Flexural properties

A flexural test creates tensile stress in the convex side of the specimen and compressive stress in the concave side [7].

Sato and Hotta reported remarkable increase in flexural strain for microwave curing than the autoclave cured CFRP specimens [10]. But the flexural modulus calculated from the 3 point bending tests showed inferior properties for microwave specimens in comparison to the autoclave cured samples [7].

4-point bend test have been carried out by Nightingale and Day in accordance with ASTM standard D 790-95a (Refer to Fig 2) [3].

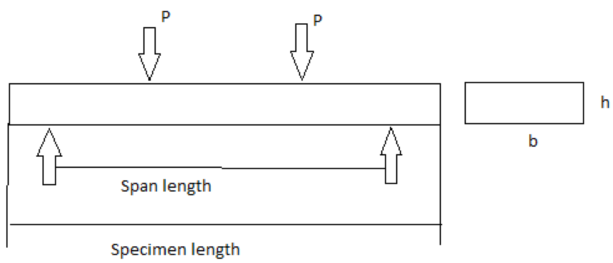

Fig 2 Schematic of 3-point short beam bending test

D) Interlaminar Shear Strength (ILSS)

3 point short beam bending tests were performed by various researchers based on the ASTM standard D2344 to determine the ILSS behaviour of composites [14]. The fig 3 below gives the schematic of 3 point bending test or ILSS method.

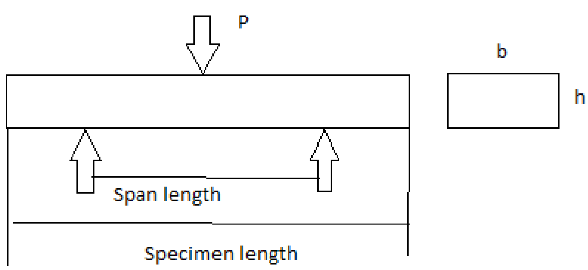

Fig 3 Schematic of 3-point bend test

According to Sunil and Somen, microwave - post autoclave curing exhibited higher ILSS followed by the autoclave and microwave curing [13]. Day and Nightingale studied the ILSS behaviour of autoclave - post microwave cured composites. They found that ILSS of autoclave cured specimens were higher followed by microwave and autoclave-post microwave curing technique [3]. From the results of Sunil and Somen, it can be decided that microwave-post autoclave curing is beneficial than the autoclave-post microwave post curing [13]. Little variation in ILSS and ILS modulus was observed for both the microwave cured and autoclave cured CFRP samples from Kwak et al's experimental study [8]. Paprgyris et al. reported 9% increase in ILSS for the microwave cured CFRP parts [7]. Wei and Kai found that temperature and curing agent can cause significant impact on the ILSS of CFRP composites. ILSS for MW cured CFRP samples. This is due to the fact that void content was low in the MW cured CFRP composite [14, **15**].

E) Impact properties

Kwak et al studied the impact behaviour of MW cured and CC samples through nano indentation. From their results, it was observed that only little variation in indentation modulus regardless of the heating method (CC or MW curing) [5].

IV. CHALLENGES

The challenges associated with the microwave curing technology include arcing, tooling design and void content and uneven distribution of microwave energy.

a) Arcing

In the early days of research in microwave curing of CFRP composites, Lee and springer were not able to achieve fully cured multi-directional samples due to arcing [15]. Recently, Nightingale and Day also encountered arcing problem as soon as the oven was switched on with the sample inside [3]. Exposed carbon fibers on the edges of the testing sample leads to arcing inside the microwave chamber, thereby damaging the vacuum bag and the composites [2,5]. Arcing is also caused due to geometric discontinuities like edges and sharp corners of the rectangular shaped samples subjected to microwave curing (Refer to Fig 4) [7].

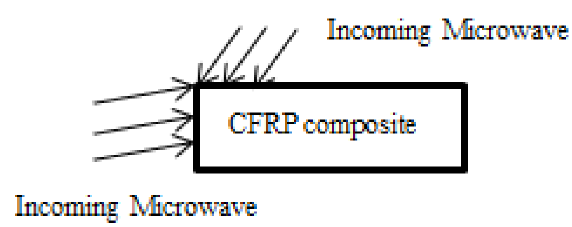

Fig 4 Geometic discontinuity & Electromagnetic waves [5]

Various effective measures have been used by researchers to weaken this coupling effect in recent times. , Nanya Li et al used sealant tapes on the edges of the composite to avoid arcing [1]. Xu et al. found that power less than 180W and vacuum pressure of 0.1Mpa could cure the composites without arcing [5]. Aluminum tapes were used to seal the edges of the composite where the carbon fibers are exposed to arcing [13].

b) Tooling Design

Wave transparent material such as glass or ceramic tooling, etc. could be suitable for microwave curing whereas metal material cannot be used for tooling due to their reflecting properties [1]. Nanya Li et al and Papargyris et al utilized ceramic tooling for the microwave curing [1, 7]. Sunil and Somen used tools made of polypropylene (PP) to cure the sample along with the application of pressure by tightening the screws in the PP fixture. Disadvantage of PP fixture is that, it melted at 610W power [13]. Day and Nightingale utilized a molding tool made from PTFE plates and sleeves. This arrangement protected the vacuum bag and carbon fibers from arcing [3]. Yingguang Li et al developed an anisotropic compressive tooling based on the simulation results to achieve high quality and geometrically accurate parts [16].

c) Void content

A major disadvantage of using microwave curing technology is that it leads to higher void content in comparison to the autoclave curing. Formation of voids is mainly due to the lack of application of pressure. Void volume V_v is calculated by researchers from the optical microscope and SEM images. For thermoset composites like CFRP composite, their production requires maximum glass transition temperature and void content to be minimum. Removal of voids is critical for high performance applications like aerospace structures [3]. Experiments by Xu et al. revealed that compressive strength of the composites is sensitive to the void content [5]. Efforts taken by the researchers to minimize the void content have been effective to some extent. Measures include the use of vacuum bagging and application of pressure [3]. Vacuum-pressure assisted microwave curing helps in reduction of thermal stress and non-uniform cross-link reaction; thereby enabling the MW cured CFRP samples to achieve void free, geometrically accurate and good quality parts [16].

d) Uneven energy distribution

Xu et al reported higher degree of variation in composite strength of MW cured CFRP samples among various samples under the same conditions [5]. It could be due to poor or uneven distribution of microwaves inside the applicator resulting in localised hotspots on the composites and inferior mechanical properties [7,13,16]. This problem of uneven energy distribution and hot spots can be remedied to some extent by the use of variable frequency microwave (VFM) which produces non-homogenous MW and non-uniform heating inside the applicator [12].

V. CONCLUSION

CFRP composites have found extensive applications in aircraft, sports equipment's, automobile, etc. due to their excellent mechanical properties and high strength to weight ratio. These composites are fabricated and cured traditionally using autoclave or thermal curing. However, increased energy consumption, cost and higher processing time associated with autoclave curing makes the researchers to look for alternative curing method. Among the various curing methods, microwave curing could overcome the above mentioned limitations and is emerging as a viable alternative for autoclave curing in recent times.

Microwave curing involves the use of electromagnetic waves to cure the CFRP composite by an inside-out solidification process i.e. carbon fibres get heated first and then the heat is transferred to the resin matrix. Research work on microwave curing of CFRP composites is limited mainly due to the challenges like arcing, uneven distribution of microwaves, tooling design and high void content. In this paper, literature review on microwave curing of CFRP, their cure properties, mechanical and physical properties and challenges associated with the microwave curing technology is addressed. Arcing can be avoided by sealing the edges with aluminum tape or sealant tape or epoxy tape. Uneven energy distribution can be eliminated to some extent by using VFM technology. For tooling design, it is better to use a wave transparent material which has low co-efficient of thermal expansion (CTE) such as ceramic tooling, PTFE plates, PP, etc. Void content can be minimized by using vacuum bagging and through application of pressure to the microwave applicator.

Compressive strength of the MW cured CFRP samples were found to be higher than that of the autoclave CFRP cured samples. A general trend cannot be observed for transition temperature T_g, flexural properties and tensile properties between CC and MW cured CFRP samples. It has been found that compressive strength of the CFRP composite is sensitive to void content and it decreases as the porosity or voids increases.

Microscopic results of MW cured CFRP samples show that, better interfacial adhesion can be achieved by MW curing between carbon fibres and resin matrix. DSC and FTIR on results were found to be helpful in determining the degree of cure. Ply thickness was an influencing factor in temperature distribution whereas ply orientation was found to be insensitive to temperature distribution.

MW curing has the ability to produce required quality of parts in a shorter curing time as observed from the results of various researchers. MW curing of CFRP samples can be beneficial when used with vacuum bagging and pressure under proper tooling conditions.

VI. FUTURE SCOPES

Fossil fuel consumption, environmental impacts and the increased demand for the synthetic fibers like carbon has turned the focus of researchers toward environmentally sustainable and eco-friendly natural fiber reinforced (NFRP) composites in recent times. Moreover, lignocellulosic fibers like sisal, hemp, flax, etc were found to be suitable for microwave curing by researchers. Also, these lignocellulosic fibers are cultivated to a significant percentage and readily available for use at present. So, the future work will involve the `microwave curing of NFRP composites or hybrid composite comprising of synthetic fibers and natural fibers.

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