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AN EMERGING, ENERGY-EFFICIENT CURE PROCESS FOR RAPID COMPOSITE MANUFACTURE

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ABSTRACT: Two types of bi-functional epoxy-amine matrix systems (one room temperature -RT cure type and one elevated temperature -ET cure type), used for aerospace composite applications, were cured at the sample mixture level, composite laminate level and representative component level using three different multimode, microwave (MW) cure equipment viz., a domestic MW oven, a laboratory scale MW set-up and conveyor-type MW facility respectively, all of them operating at a frequency of 2450 MHz.. MW cure schedules equivalent to supplier-specified thermal cure schedules were individually evolved, using the glass transition temperature (Tg) as the index of cure completion. The glass transition temperature (Tg) values of MW cured samples were equivalent or slightly superior to respective thermally cured samples. The cure status and cure uniformity were assessed using differential scanning calorimetry (DSC) and confirmed through thermal imaging techniques. For the MW cured glass-epoxy laminates, the mechanical properties determined, compared well with or were superior to those of the thermally cured counterparts. Further, the microwave curing process, per se, resulted in a significant reduction in the process cycle time and energy consumption. In order to investigate the versatility of microwave cure, preliminary cure trials were performed at representative component level viz., for a 15mm thick laminate, a sandwich panel and a curved radome profile.

1. INTRODUCTION

Microwave heating can reduce the processing times for a range of materials very substantially. Typical cure times for thermosetting resins are reduced from hours to tens of minutes or less. If MW processing of resins is to be applied for the production of components, the properties of the cured resins need to be understood. There are a few studies on MW curing of different epoxy resins and controversial results reported in the literature [1-5]. Nightingale and Day [6] cured carbon epoxy composites using a microwave oven in ~ 20% of the time required for thermal cure. Zainol et al [7] have shown that time for full cure of two bismaleimide resins could be reduced from 24 h to less than 20 minutes by opting for microwave cure. Thus, there is a lot of potential for cost-effective and rapid manufacturing of composite components through gradual upgradation of the processing techniques employing microwave heating [8,9]. Boey and Yap[1] studied the effect of microwave curing on a diglycidyl ether of bisphenol A (DGEBA) epoxy resin, with three different amine hardeners and observed that in each case, microwave heating led to faster crosslinking than conventional curing. Marand et al. [3] who carried out in situ dielectric property and infrared measurements during thermal and microwave cure of a DGEBA/DDS resin also concluded the same way. However, for the same resin, the results obtained by Mijovic & Wijaya [4] were contradictory. They reported that thermal curing was faster than microwave curing, attributing it to the possible difference in the mechanism of cure by thermal and MW energy, which needed to be further explored. The glass transition temperature (Tg) of cured thermosetting resins is normally maximized in order to get the highest possible working (service) temperature range. Some researchers [2,10,11,14] reported that, for a range of amine cured epoxy resins, Tg was higher for MW cured samples than for conventionally cured ones. The literature revealed that most studies reported reflect micro-level studies (very small sample sizes) and more so at neat resin level and a very few at composite laminate level. To date, there are few reports in open literature on MW curing of thermoset matrices at an engineering level that can clearly demonstrate the multi-faceted benefits of microwave cure processes.

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In the present work, two epoxy-amine systems and their glass fibre reinforced composites were cured, using both thermal and MW heating. Materials cured by these two routes have been compared using DSC, thermal imaging and mechanical property evaluation. Microwave cure trials from sample casting level through composite laminate level, upto preliminary cure trials at representative component level were successfully demonstrated.

2. EXPERIMENTAL

2.1. Materials used

System 1: LY556/HY951 (RT cure type): LY556 resin is a bifunctional epoxy resin ie., diglycidyl ether of bisphenol-A (DGEBA) and HY951 is an aliphatic primary amine, viz., triethylene tetramine – TETA. Mixing ratio is 100:11 w/w.

System 2: LY556/HT972 (ET cure type): HT972 is a solid, aromatic amine, viz., 4,4' – diamino diphenyl methane (DDM). Mixing ratio is 100:28 w/w. The resin systems were supplied by M/s. Huntsman Advanced Materials, Mumbai. The chemical formulae are shown in Fig.1. The glass fabric reinforcement used for fabrication of composites was E-glass woven fabric (twill weave) supplied by M/s. Arun Fabrics, Bangalore, having an an areal density of 280 g/m^2 (ie., 280 GSM). Further details are available in [14].

2.2. Methods

2.2.1. *Thermal Cure*: RT cure type samples were post cured and ET cure type samples were completely cured in an electrical heating oven as per the vendor supplied cure schedules (Table 1).

Table 1: Thermal cure schedules for the polymer matrices/composites

Sl. No.	Matrix/GFRP System	Cure conditions	Post-cure conditions	Tg after full cure (°C)
1	LY556/HY951	RT cure - 24 h	50°C/0.5h, 70°C/1h, 85°C/2h	101
2	LY556/HT972	120°C/1 h	150°C/2 h	160

2.2.2. Microwave Cure: Both RT and ET Cure type samples were cured at three different levels in three different microwave equipment as shown in Table 2.

Table 2: Different levels of microwave curing

Sl. No.	Curing sample level	Microwave set-up used	Total Power	Mode of MW heating
1	Sample mixture	Domestic Oven	800W	Continuous & Pulsed
2	Casting/Laminate	Lab-scale equipment	2400W	Pulsed
3	Representative component	Conveyor-type facility	3200W	Pulsed

The sample preparation methodology for cure trials in the domestic and lab-scale microwave set-up are described elsewhere [14].Cure schedules were optimized taking Tg attained as the basis. The pulsed heating schedules that involved a combination of alternating heating and soaking periods (ON and OFF) [12] was found to yield desired results. Temperature was measured using a digital, non-contact type infrared pyrometer (M/s. Raytek, USA).

2.2.3. Cure uniformity: Cure uniformity of the MW cured samples was assessed in a novel way, by using a thermal imaging camera [15], followed by measuring the Tgs of the samples chipped out of the cured-castings. The thermal mapping and the Tg mapping techniques together characterized the cure uniformity.

Glass transition temperature (Tg): The Tg of thermally cured and MW cured samples was determined using a DSC 2910 (M/s. Waters Inc.,) module as per ASTM D 3418 procedure.

2.2.4. Mechanical Properties: The cured laminates were cut into mechanical test specimens as per ASTM standard specifications for generation of tensile, compression, flexural and shear properties and tested as per ASTM standard procedures using a universal testing machine (INSTRON 6025).

3. RESULTS & DISCUSSION

Table 1 shows the standard thermal cure schedules adopted for the two resin systems used in the present studies and the Tg values obtained after full cure, which were taken as reference to confirm completion of cure in the microwave cured systems.

3.1. Cure in a Domestic MW oven: The optimized cure schedules, in continuous and pulsed heating modes for systems 1 & 2 are shown in Figs. 1&2 respectively. The cured samples of both the systems, with Tgs labeled at different positions are presented in Fig.3.

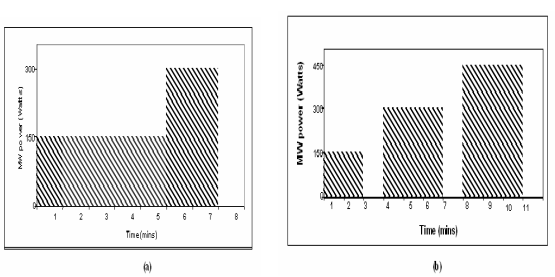


Fig.1: MW Cure schedule for RT cure type System 1 (a) Continuous (b) Pulsed heating mode

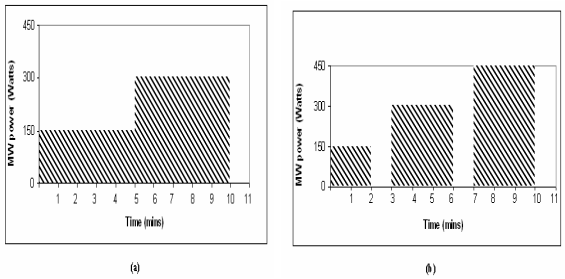
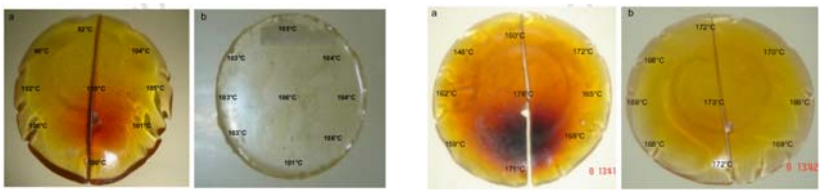


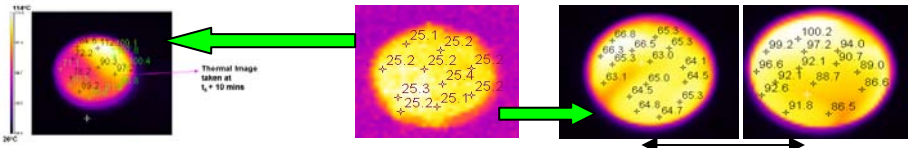
Fig.2: MW Cure schedule for ET cure type System 2 (a) Continuous (b) Pulsed heating mode



SYSTEM 1 **SYSTEM 2**

Fig.3: Tg mapped samples of Systems 1&2: (a)Continuous heating (b) Pulsed heating

These showed that the continuous mode led to undesirable hot-spots (as reddish brown zones as shown above), which were avoided by opting for the pulsed heating mode. Fig 4 shows the thermal images during MW cure of System 1. It was observed that continuous heating caused temperature non-uniformity, even within a small sample. However, the thermal images during pulsed heating, showed a uniform temperature distribution for a similar sample size. Hence for the subsequent cure trials using lab-scale and conveyorised set-up, pulsed mode of heating was chosen.



During Continuous heating **Start of Cure** **During Pulsed heating**

Fig. 4: Thermal imaging of temperature distribution during MW Cure of Epoxy Matrix System 1

3.2. Cure in a Lab-scale MW Equipment: The optimized pulsed mode cure schedules for the laminates made of matrix systems 1&2 (Fig.5) resulted in the desired Tg values [12,13] and mechanical properties, that either compared well with or were superior to those of thermally cured composite laminates (Table 3)

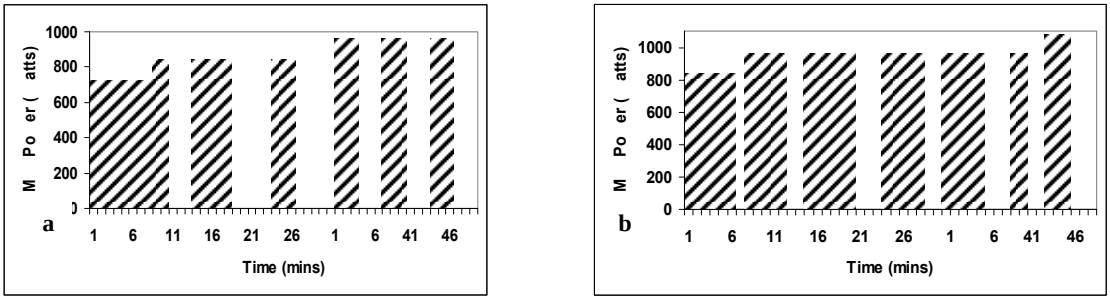


Fig. 5: Optimised, Pulsed MW Cure Schedules of laminates made from (a) System 1& (b) System 2

Table 3: Mechanical properties of Composite systems 1&2: Thermal & Microwave cure

GFRP Laminate	Tensile Strength (Kg/mm^2)		Compression Strength (Kg/mm^2)		Flexural Strength (Kg/mm^2)		ILSS (Kg/mm^2)	
	Thermal	MW	Thermal	MW	Thermal	MW	Thermal	MW
LY556/HY951	38.57	36.76	35.55	37.36	58.64	58.81	3.40	5.4
LY556/HT972	40.14	42.24	38.42	44.24	64.68	63.44	3.85	5.08

MW curing also resulted in enormous reduction in the process cycle time and electrical energy consumption vis-à-vis thermal curing (Figs. 6&7). This is attributed to the instantaneous and volumetric mode of MW heating.

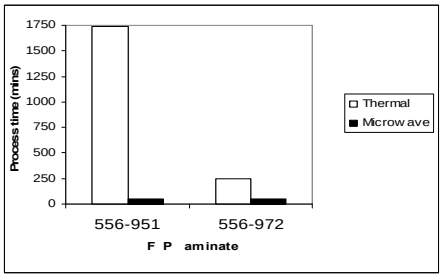


Fig.6: Process cycle time: Thermal vs MW

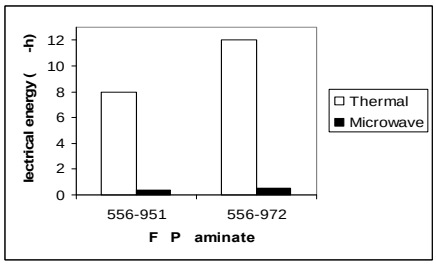


Fig.7: Electrical energy consumed: Thermal vs MW

3.3. Cure in a Conveyor type MW Equipment: After successful trials at laminate level in a lab-scale MW set-up, the cure process was further scaled-up to a 15 mm thick laminate and representative component shapes, eg., scaled down radome profiles (Fig.8(a),(b)&(c)) through preliminary cure trials. The respective cure schedules and temperature profiles are presented in [14].

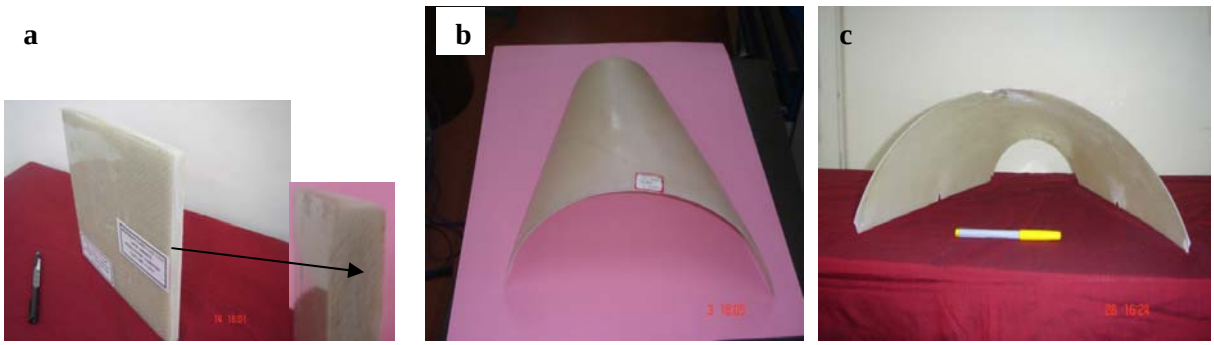


Fig. 8: Composites cured in the conveyor-type MW facility: (a) 15mm thick GFRP laminate;
(b)&(c) Scaled-down radome profile

The microwave cure schedule followed for the 15 mm thick GFRP laminate of RT cure type System 1 required a cure time of 65 minutes and an energy consumption of 0.44kW-h, to yield a Tg of 101±4°C. For the radome shaped profile, the total cure time was 51 minutes, at an energy input of 0.281kW-h, yielding a Tg of 100±4°C. The Tg values measured across the part were in the same range, confirming cure uniformity. More trials are still in progress to standardize the microwave cure schedules at the level of representative component shapes of larger size.

4. CONCLUSIONS

The present studies have clearly shown that the cure trials in the domestic oven, though yielded non-uniformly cured samples attributed to its inherent constructional, established the proof of principle of microwave cure, for polymers, and the savings accomplished in terms of cure cycle time and energy. The microwave facility, when suitably designed & upgraded to composite laminate level cure studies, yielded results that demonstrated cure uniformity, reduced cure cycle times, significant energy savings with comparable or better thermal and mechanical properties as compared to those of thermally cured laminates. The versatility & scalability from laboratory level to industrial level have been satisfactorily proved through scaled-up cure studies for representative components. More intensive studies are in progress to perfect this technique for full-scale component development by microwave curing.

5. ACKNOWLEDGEMENTS

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REFERENCES

1. Boey F Y C, Yap B H (2001), Polym. Test. 20:837
2. Wei, J., Hawley, M.C. and DeMeuse M T. Polym Eng Sci, (1995), 35(5):461
3. Eva Marand, Baker H R, Graybeal J D, (1992) Macromolecules; 25; 2243-52.
4. Mijovic J, Wijaya J, (1990), Macromolecules, 23, 3671-3674
5. A Nesbitt, P Navabpour, B Degamber, C Nightingale, T Mann, G Fernando and R J Day, (2004), Meas. Sci. Technol. 15, 2313-2324
6. C.Nightingale and R.J.Day, (2002) Composites A 33; 1021-1030
7. I. Zainol, R J Day, F Heatley (2003), J Appl Polym Sci 90 (10):2764 –2774
8. M S Johnson, C D Rudd, Hill D J (1998), Composites A 29(1-2): 71-86
9. Boey F Y C, Gosling I, Lye S W, (1992) J Mater Process Technol 29: 311-319
10. Yarlagadda KDVP, Hsu S-H (2004) J Mater Process Technol 1532:155
11. Boey FYC (1995) Polym Test;14:837–45
12. P S Mooteri, Sandhya Rao, M. R. Prakash, R M V G K Rao, B K Sridhara, (2006)Journal of Reinforced Plastics and Composites, 25, 5; 503-512
13. R.M.V.G.K. Rao, Sandhya Rao, B.K. Sridhara, (2006) Journal of Reinforced Plastics and Composites 25, 7; 783-795
14. Sandhya Rao, Ph.D. thesis entitled “Studies on Radiation cured thermosetting polymer matrices and composites”, (Feb 2008)
15. Sandhya Rao, RMVGK Rao, (2008), Polym Test 27: 645– 652