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Optimization of infrared radiation cure process parameters for glass fiber reinforced polymer composites

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ABSTRACT

Elevated temperature post curing is one of the most critical step in the processing of polymer composites. It ensures that the complete cross-linking takes place to produce the targeted properties of composites. In this work infrared radiation (IR) post curing process for glass fiber reinforced polymer composite laminates is studied as an alternative to conventional thermal cure. Distance from the IR source, curing schedule and volume of the composite were selected as the IR cure parameters for optimization. Design of experiments (DOE) approach was adopted for conducting the experiments. Tensile strength and flexural strength of the composite laminate were the responses measured to select the final cure parameters. Analysis of variance (ANOVA), surface plots and contour plots clearly demonstrate that the distance from the IR source and volume of the composite contribute nearly 70% to the response functions. This establishes that polymer composites cured using IR technique can achieve the same properties using only 25% of the total time compared to that of conventional thermal curing.

1. Introduction

Fiber reinforced polymer composite finds application from head gear to aircraft due to its lightweight, higher strength to weight ratio and adaptability to customize the composite according to the required strength and functionality for which it is employed. In the processing of polymer composites curing plays a vital role. Curing is the process of conversion of liquid resin into hard solid structure and it takes place at molecular level. Curing is one of the significant and complex processes which requires considerable attention as it consumes lot of time and energy. Models developed by Loos and Springer for curing, stressed the importance of reducing the cure time and explained its importance on the final strength of the composite [1]. The process of polymerization is the joining of lower molecular weight reacting monomers to form a three dimensional network or polymer chain. The effect of cure cycle on the final quality of the composite studied by Naji and Hoa, has revealed the effect of different cure cycles on varying thickness and fiber volume fraction of the composite [2]. Zhang et al., have proposed three dimensional finite element models to analyze the temperature and degree of cure for epoxy resin. They explained that final hardness depends on degree of cure and the temperature variation during the cure for the epoxy resin [3]. Ramakrishnan et al., have suggested reducing the curing time by using internal resistive carbon mats without compromising with the composite quality [4]. Many researchers have worked on alternative methods of curing to overcome the disadvantages of conventional method, that is the hot air curing of composites, known as the thermal curing. Different methods of curing are in practice apart from thermal curing such as microwave, radiofrequency, ultraviolet and infrared radiation cure. Rao et al., have worked on the microwave curing of composites. There is drastic reduction in curing time with higher mechanical properties of the composite [5]. Sabit and Arumugham have successfully utilized ultraviolet radiation for curing of composites and stressed the need for better curing system [6]. Radio frequency (RF) was applied to cure the epoxy resin by Gourdenne and it was proved that the conventional heating and RF has no structural difference in the strength of the composite [7]. Ribeiro et al., have demonstrated the electron beam polymerization of epoxy resin. The cure rate increased and the kinetic model was developed based on duration of exposure and degree of cure [8]. It is understood from various works reported that improper curing leads to uncured resin patches inside the composite which leads to lower strength of the composite. Infrared radiation (IR) curing is one of the efficient methods of curing of polymer composites. Belhamra et al., have explained the technology and applications of infrared heating [9]. Chern et al., in their work reported about IR heating of Hoop wound cylinders, several process parameters were studied and

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the developed models agreed well with experimental results [10]. IR heating using lamps was proposed by Labeas et al., in their work for heating of thermoplastic parts. They studied the effect of various process parameters which influence the IR heating process, and concluded that as the thickness of the part increases the heating time also increases [11]. Direct electro-magnetic radiation is a very good source of energy for processing of polymer matrix composites. Since polymeric resins are polar in nature, they react to the electrical and magnetic field components of the EM radiation. Depending on the intensity and the frequency of the incident radiation the polymer molecules get polarized, in the process generating heat. Decker stated in his review article that the photo polymerization is one of the fastest means of generating three dimensional network [12]. Radiation curing finds lot of scope in curing of polymer composites and hence infrared radiation (IR) will be a suitable alternative to conventional curing. IR is part of electromagnetic (EM) spectrum, with wavelength in the range of 10^{-6}–10^{-4} m. Infrared is transmitted in three energy bands. Short wave (0.76–2.3 μm) used for complex part shapes. Medium wave (2.3–3.3 μm) used for curing coatings on objects. Broad wave (3–8 μm) used for laminates and packing machines. It is highly suited for FRP composites curing as the technology is proved for curing of paints, coatings, processing of food items and also for industrial heating applications. The main advantage of infrared curing is, it heats only the composite and not the air present in between the heater and the product. The radiation is directly absorbed by the composite and hence the losses are minimum. It is mainly suited for flat surfaces and the efficiency depends mainly on the infrared absorbance capacity of the material. Different materials have different absorbance capacity. Very few researchers have worked in the area of IR curing. In this context an attempt is made to develop optimum IR cure process parameters for glass fiber reinforced polymer composite laminates.

2. Materials

The polymer matrix used in this investigation is bifunctional diglycidyl ether of bisphenol A (DGEBA) type epoxy resin (LY556). The curing agent used is 4–4′aminophenylmethylaniline (HT972). The resin and the curing agents were procured from M/s. Huntsman Advanced Materials, USA The reinforcement used is bidirectional, E-glass fabric with a fiber orientation of 0°/90° and a fabric weight of 200 g/m^2. Table 1 presents the properties of the materials used for the preparation of composite laminates. The fabric layers were pre-heated at 120 °C in an oven so as to remove adsorbed moisture, prior to lay-up. Composite laminates with the weight ratio of 65:35 that is glass fibers to resin ratio and the hand-layup method was adopted for preparing the laminates. The laminates were then allowed to cure at room temperature (RT) for 24 h. The fiber weight fraction of all the laminates was maintained at 65 ± 2%. The post curing process was carried out in a custom designed infrared curing system having 400 × 400 stainless steel chamber mounted with IR heater of 3–8 μm wavelength of 2 kW capacity. The chamber is insulated by ceramic board of 50 mm thick to avoid the heat loss to surroundings.

Distance from IR source to laminate maintaining constant power of 2 kW, IR cure schedule and the volume of composite were selected as the main factors in order to optimize the cure process. Preliminary trials confirmed the effect of main factors selected on the overall quality and strength of the composite laminate. Composition of all the laminates and power was maintained constant for all the trials during the experimentation process.

Table 1
Properties of materials.

Materials	Density (g cm^{-3})	Tensile strength (MPa)	Young's modulus (GPa)
E-glass fibers	2.55	1750	70
Epoxy	1.25	55	3.5

3. Experiments

The experiments were conducted based on central composite design (CCD) approach of design of experiments (DOE). CCD is a powerful diagnosing experimental tool to study the large number of factors. Two or more factors with three or more levels require higher experimentation time and cost in terms of materials, power and labor. In order to save the same without sacrificing the required quality CCD approach is best suited. CCD is used extensively for second order response surface models. Several papers have reported the application of CCD to optimize the process and properties of the composites. Suresha and Sridhara [13], Ruijun and Kokta [14] and Onal and Adanur [15] have successfully employed CCD approach to determine the optimum properties and process of composites. The factors, their levels and the range selected are presented in Table 2. The total number of experimental runs presented by both coded and actual values of factors along with the central runs as per CCD approach [17–19] is indicated in Table 3.

The thermal cure schedule is based on conventional thermal curing as indicated in Table 4. The curing time schedule has different cure schedule of ramping and soaking. Ramping and soaking at different temperatures is beneficial in achieving improved properties compared to room temperature and single high temperature cure. Optimum properties were achieved by curing in steps [16]. Curing process is more uniform and the fiber matrix bonding is improved. Although at room temperature the epoxy matrix appears to be solidified still there is still large amount of un-reacted resin

Table 2
Factors and their levels as per CCD.

Factors	Levels				
	(−α)	(−1)	(0)	(1)	(+α)
Distance from heater (mm)	150	180	225	270	300
Curing time (min)	56	93	148	203	239
Volume of the laminate (mm^3)	81,675	120,000	175,692	231,852	270,000

Table 3
Coded and actual values of factors.

tc	A	B	C	Distance in mm (A)	Curing time in min (B)	Volume in mm^3 (C)
1	−1	−1	−1	180	93	120,000
2	+1	−1	−1	270	93	120,000
3	−1	+1	−1	180	203	120,000
4	+1	+1	−1	270	203	120,000
5	−1	−1	+1	180	93	231,852
6	+1	−1	+1	270	93	231,852
7	−1	+1	+1	180	203	231,852
8	+1	+1	+1	270	203	231,852
9	−1.682	0	0	150	148	175,692
10	+1.682	0	0	300	148	175,692
11	0	−1.682	0	225	56	175,692
12	0	+1.682	0	225	239	175,692
13	0	0	−1.682	225	148	81,675
14	0	0	+1.682	225	148	270,000
15	0	0	0	225	148	175,692
16	0	0	0	225	148	175,692
17	0	0	0	225	148	175,692