Curing Methods for Advanced Polymer Composites - A Review

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SUMMARY

Advanced polymer composites have obtained great application interest in a number of demanding aerospace, wind energy, automotive, infrastructure, and consumer applications. Great varieties of curing methods are investigated to develop low-cost and high-efficient fabrication of advanced polymer composites, which still remains as a great challenge and thorny issue. Especially, the autoclave curing process, which is widely used for curing of high performance advanced polymer composites, is labor- and capital-intensive, with costs increasing exponentially with part size and limiting increased use of advanced polymer composites. Researchers and industries have long desired to explore and develop low-cost and high-efficient curing methods for fabrication of advanced polymer composites and investigated different radiation and thermal curing alternatives. In this paper, current development status of the radiation curing (gamma ray, x-ray, ultraviolet, accelerated electron beams) and thermal curing (radiation heating (infrared, laser and microwave), convection and conduction heating (hot gas, flame, oven and hot shoe), induction heating, ultrasonic heating, resistance heating and thermal additives (magnetic particles, NIR absorbent particles) based heating methods applied for the curing of advanced polymer composites are reviewed. The curing mechanism and current application status of the different curing processes for fabrication of advanced polymer composites is discussed, and main advantages and disadvantages of these methods are comparatively analysed and evaluated according to the material, cost, feasibility and power criteria for successful curing application of advanced polymer composites.

Keywords: Advanced polymer composites; Thermal curing; Radiation curing; Out-of-autoclave curing

1. INTRODUCTION

Due to the high stiffness-to-weight and strength-to-weight ratio, excellent fatigue and corrosion performance, fiber-reinforced polymer composite materials, also known as advanced polymer composites, have demonstrated clear-cut advantages with advanced performance at decreased weight over more conventional metallic materials, in a number of demanding aerospace, wind energy, automotive, infrastructure, and consumer applications. The latest Boeing-787 airplane is fabricated with up to 50% composites by weight[1], and the Airbus-A350 XWB is also planning to increase the composite usage to about 53%[2]. In recent years, applications of advanced polymer composites for a variety of large and complex structural parts and components are becoming reality: e.g. airplane wings and bodies, wind turbine blades of more than 70 m, rail cars and truck bodies, etc.

The dominant curing process today for advanced polymer composites is still based on thermal curing using a range of thermal heating processes, in which the oven or autoclave is widely used. Because polymers and their composites have low thermal conductivity (epoxy resin thermal conductivity at 25 °C: 0.19 W/m K), conventional thermal curing processes for polymer composites are time and cost intensive. In order to keep the temperature profile in range without degrading the composite parts, very low heat up rates (1-2 °C/min) are used during the curing process, which leads to long curing cycles with high energy consumption. In certain applications where thick laminates with thickness of up to hundreds of millimetres are needed, the non-homogenous curing of parts with large thermal stresses due to the big temperature gradient is a thorny issue. Especially, in the case of autoclave curing for high-performance applications, critical challenges arise with the increasing of size and thickness of composite parts[3,4]:

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- long curing cycles with high energy consumption.
- non-uniform curing of parts with large residual stresses due to the unavoidable temperature gradients
- large capital investment and operating expense, with costs increasing exponentially with part size.

Moreover, in many cases, an oven or an autoclave big enough for the part is not available or has limited availability. "ASC Process Systems" company (USA) developed a world's largest autoclave (working area: ϕ 9 m × 25 m) to satisfy the curing demands from the Boeing-787's epoxy/carbon-fibre composite parts[3], but the size limitation is still a great challenge which is limiting increased use of polymer composites in the wind and aerospace industry.

Therefore, researchers and industries have long desired to explore and develop low-cost and highly efficient composite curing methods and investigated different curing processes. According to the curing mechanism, the curing technologies can be categorized as radiation curing and thermal curing. In this article, the curing mechanism and current application status of the different curing processes for fabrication of advanced polymer composites is reviewed, and existing challenges are summarized.

2. RADIATION CURING

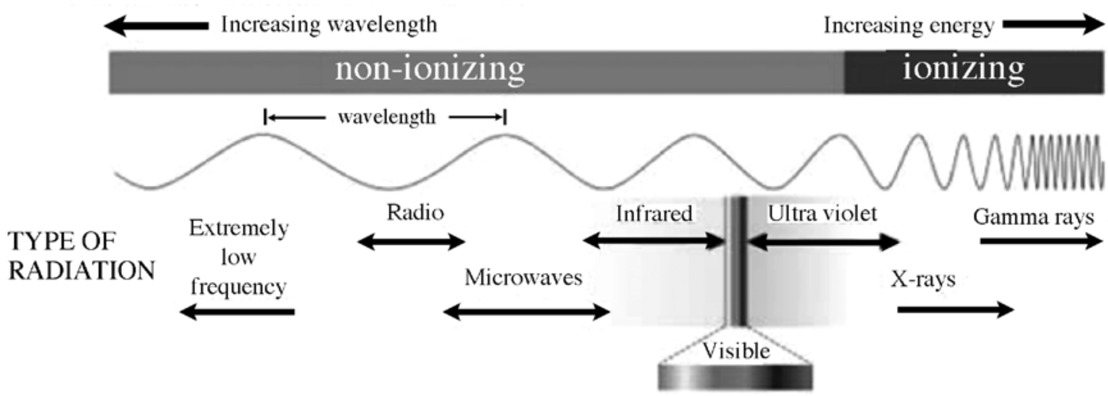
Radiation curing is based on the ionization (bond breakage) of radiation sensitive polymers by the use of high-energy electromagnetic radiation such as gamma ray, x-ray, ultraviolet or accelerated electron beams. Unlike the thermal curing of a two-component resin system, which needs a hardener or catalyst combination with the primary resin to induce crosslinking or curing, radiation curing is initiated by the ionic or free radical intermediates decomposed by the radiation sensitive resin on irradiation[5,6]. Due to the special curing mechanism, radiation curing provides some unique technological superiority compared to thermal curing, including improved resin stability, handling flexibility, fast curing speed, energy efficiency, etc. Additionally, it offers considerable control and some process latitudes to manufacture complex and large structures in contrast to thermal curing techniques.

2.1 Electron Beam

Electron beam curing is based on accelerated electrons that can provide energy for the initiation of the curing process by the decomposition of the radiation-sensitive polymer. Since AEROSPATIALE company of France firstly investigated the electron curing of advanced composites for aerospace applications from the early 1970s and manufactured a missile fuel tank by electron beam curing[7], the application of electron beam curing for advanced composites has obtained great development and several airplane parts, including bulkhead and wing skin, were successfully manufactured[8,9].

Electron beam curing of polymers was explored as a very attractive alternative for Out-of-Autoclave (OOA) production of high-performance composite materials. However, applying high-energy electrons (5-15MeV) to penetrate the whole thickness of the part requires large capital costs for the high-energy emitters and their operation, because of the concrete walls and ceiling to shield x-rays which are produced as a by-product of the electron beam process. Aiming at these issues, Cirri *et al.*[10] put forward low-energy electron beam layer-wise curing fabrication process to reduce the high operating, maintaining and shielding costs in high-energy electron beam processing. Afterwards, Guasti *et al.*[11] investigated filament winding process by *in-situ* electron beam curing and successfully fabricated thick composites by this process. Goodman[12, 13] and Bykanov *et al.*[14] carried out research on the *in-situ* electron beam curing fibre placement process. However, because of several critical deficiency in the mechanical properties of the low-energy electron beam cured composites[15], including

Figure 1. Electromagnetic wave spectrum



poor interface bonding between matrix and fibre, low transverse mechanical properties and higher brittleness of the matrix compared to the state-of-the-art thermally cured composites, electron beam curing has not gained large acceptability in a production environment until now.

2.2 Gamma Ray and X-ray

Gamma ray or x-ray curing of advanced composites was investigated since 1970s and has been proved to be of potential industrial interest, especially for thick composites (up to 300 mm) due to the extra-high electromagnetic energy[16,17]. However, the dose rates of x-rays and gamma rays are low compared with those of electron beams, resulting in curing times ca. 60 times longer. Most importantly, curing by gamma ray and x-ray techniques involves great potential risk considering the high level of radioactivity and the produced hardly disposable matter, which is not practical and takes many years and with high accompanying risk, to apply these technologies successfully for industry.

2.3 Ultraviolet

Compared to electron beam, gamma and x-ray curing, UV-curing is more cost efficient, energy-saving, environment and user friendly, so widely used in curing of coating and thin films[6,18]. In recent years, some researches on UV curing advanced polymer composites have been conducted to develop low cost manufacturing process[19,20]. However, due to the absorption behaviour of UV radiation passing through matter, the use of UV curing has so far been limited to open mould processes and transparent composites (mostly glass fibre reinforced) with limited thickness[21,22]. Several scholars have combined UV curing with filament winding and tape placement processes, and investigated layer-wise curing fabrication of transparent UV curing advanced polymer composites[23]. A critical limitation in application of UV curing for the high performance carbon fibre-reinforced polymer composites is the opacity of the carbon fibre, so Gupta *et al.*[24] investigated UV irradiated photo-thermal dual-curing of carbon fibre reinforced composites by combined photo- and thermal-initiators: the radiation curing is initiated by the decomposed photo-initiators by UV irradiation at the surface, and exothermic heat generated by the radiation curing process accelerates the thermal curing of resin at deeper places where UV cannot penetrate. However, due to the limited amount of heat can be generated by the radiation curing, the final properties of the cured composites were not satisfactory. Therefore, the process could only be used for rapid prototyping.

3. THERMAL CURING

Thermal curing is by far the most popular curing method for polymer composites, in view of the mature thermal curing material systems and curing processes. Nowadays, great variety of thermal heating processes for thermal curing are in application, including infrared, laser, microwave, hot shoe, hot gas, flame, oven, induction, ultrasonic, resistance heating and etc. According to the heating mechanism, they can be categorized into radiation heating (infrared, laser and microwave), convection & conduction heating (hot gas, flame, oven, and hot shoe), induction heating, ultrasonic heating, resistance heating and thermal additive-based heating.

3.1 Radiation Heating (Infrared, Laser and Microwave)

According to the radiation heating theory[25], radiation heating is based on conversion of the electromagnetic radiation to heat due to the resonance vibration of molecules in a certain radiation wavelength. According to the wavelength range of the incident irradiation energy, the mechanism for electromagnetic energy absorption differs from resonance vibration of molecules in the infrared (1.5 to 1000 μm) wavelength range to molecular rotation in microwave (above 1000 μm). It is important here to point out that the infrared and microwave radiation cannot produce ionization in the resin material due to the low energy, and the curing is still driven by the heat converted by the radiation energy. Therefore, the basic curing mechanism by the infrared and microwave radiation is still thermal curing, even if electromagnetic radiation is used for the curing. Because of the different frequency of infrared and microwave, the heating process by infrared radiation differs from microwave radiation due to the different energy absorption mechanism.

3.1.1 Infrared and Laser

Infrared radiation heating is based on conversion of electromagnetic energy to heat by resonance vibration of molecules. Polymers are containing many couplings such as CH, CH_2, CH_3 and CC and these molecules are vibrating at specific frequencies. The large majority of the vibration frequencies of these molecules correspond to the short wave and medium infrared region above 1.5 μm[26]. When irradiated by resonance energy, the vibrations will intensify and produce thermal energy. Infrared and laser sources are advantageous considering their high flexibility and controllability, so they are rather attractive for heating, welding of thermoplastics and similar applications[27-29]. However, because of the strong resonance vibration of molecules at infrared wavelengths, most of the electromagnetic energy in the infrared wavelength is consumed at the surface of the material. Therefore, most of the electromagnetic waves in the infrared range cannot penetrate deep into the polymers, so the curing of the bottom of the part is based on heat conduction from the surface. Because of the "surface heating" mechanism of infrared, if too high a radiation intensity is applied, e.g. by laser, the surface of the polymer will be degraded before the heat transfers to the bottom. Therefore, the emitting spectrum and power density of infrared heating source for heating of advanced polymer composites are critical parameters, which need to be carefully tuned to the specific material type and application.

3.1.2 Microwave

Microwave heating is based on the dipolar molecular rotational interaction with the electromagnetic field. It has been considered as a highly efficient volume-heating radiation process for polymeric materials, due to the deeper penetration capability. The heating efficiency of the microwave depends greatly on the dielectric properties of the material, so it shows good potential for curing of pure polymer or glass/ aramid fibre reinforced composites that have lower dielectric loss properties[30-32]. Research results of Boey *et al.*[33,34] showed that the stiffness and strength properties of the glass fibre composites cured by microwave are equivalent to those by autoclave curing. However, great challenges arise when microwave heating for carbon fibre composites using the traditional microwave ovens[35,36]: due to the high dielectric loss of the carbon fibre, the reflectance of the first few layers was too high to achieve efficient heating of thick laminates. Besides, the incorporation of high conductivity carbon fibres results in the formation of local hot spots and electrical arcing. Aiming at these issues in microwave heating of carbon fibre-reinforced polymer composites, recently Feher and Meyer *et al.*[37-39] developed microwave autoclaves by tuning the electromagnetic frequency and heating field and successfully achieved accelerated heating of high performance carbon fibre polymer composites without material degradation. The microwave autoclave provides a good alternative for the conventional autoclave processing of high-performance composites curing, which can remarkably decrease the cycle time. However, the microwave autoclave is limited by the energy efficiency and heating depth considering the high reflection by the carbon fibre material. Besides, this microwave heating device needs high development cost and precise process control to prevent local hot spots. In addition, lack of flexibility due to the harmful radioactive nature of the microwave to human body is still a critical issue to be widely applied in industry.

3.2 Convection and Conduction Heating (Hot Gas, Flame, Oven and Hot Shoe)

Convection heating is based on the transfer of heat by the movement of gases or liquids between surfaces. Although often discussed as a distinct method of heat transfer, convective heat transfer also involves processes of conduction (heat diffusion). In a heating process by hot gas, flame and oven, heat is transferred by convection of the heated liquids and gases to the surface of material to be heated[40]. Pitchumani and Tierney *et al.*[41,42] applied hot gas for layer-wise fabrication of thermoplastic composites. However, as the convection and conduction heating is still dominated by conduction of heat from the surface to the bottom of the material, which is based on the thermal conductivity of the material, they are challenged in the speed of heating by the very low thermal conductivity of the polymer matrix[43], which needs long cure cycles and makes it difficult to achieve efficient uniform heating of thick polymer parts.

3.3 Induction Heating

Induction heating is based on the principle that when an electrically conductive, non-magnetic material is exposed to an alternating magnetic field, eddy currents are induced and the material is heated due to resistive losses of the eddy currents. In magnetic materials, hysteretic losses occur which lead to an additional heat generation[44]. The past two decades have seen the emergence of induction heating as a suitable and effective technology for heating and welding of thermoplastic composites. Glass-fibre reinforced polymer composites can be induction heated by means of an additional electrically conductive susceptor material which is placed at the place to be heated and joined. Carbon fibre-reinforced composites on the other hand, are considered to be induction heated without any additional material since the carbon fibres are electrically conductive. However, the heat generation mechanisms by induction in the carbon fibre-reinforced composites are not clear yet: Miller[45] and Fink[46] investigated the principle of induction heating of carbon-fiber reinforced thermoplastics, and both found contrary reasons for the heat generation. Miller found that eddy currents are induced in the carbon fibers and electrical current transfers between the fibers and fiber layers since they are in contact or close proximity. The heat generation is caused by Joule losses in the fibers. However, the existence of conductive loops is required, so that the unidirectional carbon-fiber laminates cannot be induction heated efficiently. On the other hand, Fink claimed that the heat generation is caused by dielectric losses in the polymer between the fibers, so even laminates with fiber volume fraction of 60% where there is not contact or close proximity between the fibers, can be efficiently heated by induction. Despite the contrary approaches for the heat generation mechanisms both Fink and Miller reported good agreement between the experiment and the theory. Moreover, there are several issues, most notably the edge effect and the local heating effect, that prevent embracing induction welding on a large scale[47].

3.4 Ultrasonic Heating

The principle of ultrasonic heat generation is based on the conversion of high-frequency mechanical vibrations into heat through frictional and viscoelastic effects. It is already applied for thermoplastic welding due to the extremely short welding times, ease of automation, excellent quality, etc.[48,49] Curing of pure thermoset epoxy resin by ultrasound was also investigated by Sharma *et al.*[50]. However, the use of ultrasound for the consolidation of polymer matrix composites containing more than 35 to 40% by volume of reinforcing fibre was viewed as very challenging due to severe fibre disruption and damage[51]. Besides, as ultrasonic vibration is a mechanical wave and needs a solid medium to propagate efficiently, great attenuation occurs in air, solid contact between the ultrasonic transducer and material is needed to apply the energy efficiently; this remarkably reduces the handling flexibility for the curing process, especially for parts of large size.

3.5 Resistance Heating

Resistance heating is based on the electrical-resistance "Joule heating" effect of the material after application of electric current. Resistance heating is most attractive for composite bonding or welding processes using a heating element (steel mesh, copper electrical connectors) to heat up the interface and pressure to create bonds between materials[52-54]. The electric conductivity of carbon fibre provides a potential to use the carbon fibres as a heating element by directly applying electricity to them[53,55]. However, due to the specific electric application nature of the heating process, it is mostly applicable for welding, but it is not quite flexible to be combined with typical fabrication process for high-performance composites, so no research work relating curing of composite structures by this process is noticed.

3.6 Thermal Additives Based Heating

Adding nano-additives/fillers to polymers to improve thermal, mechanical and other specific properties to achieve functional nano-composites are being widely investigated and proved to be a potential development interest in plenty of fields[56-60]. Heating based on additives means generation of heating due to the coupling of the corresponding additives in the polymer matrix with the applied exterior energy, e.g. alternating magnetic field, near infrared (NIR), etc.

3.6.1 Magnetic Additives Heating

Magnetic particles (iron oxides, nickel, ferrite particles etc.) heat inductively due to magnetic losses associated with the magnetization/demagnetization cycling under an alternating (AC) magnetic field, so addition of magnetic additives to the material to be heated will generate heat after exposure to an alternating magnetic field. This method is mostly used in medical application for tumour thermal ablation[61,62], and induction brazing of metallic parts[63,64]. Zhang *et al.* summarized the magnetic induction heating of nano-sized ferrite particles within different solutions in a book chapter. For detailed information relating this process, readers are redirected to this paper[65].

3.6.2 NIR Additives Heating

Most polymers are nearly transparent to NIR from 0.75-1.5 μm wavelength due to the decreased coupling of the dipoles in polymers with the NIR electromagnetic wave[26]. By applying NIR additives, including dyes and pigments (dyes are organic molecules which dissolve in the medium of application, while pigments can be of organic or inorganic structure and are insoluble in polymers.) which absorb NIR and generate heat, efficient heating can be obtained through the thickness of the polymer material. This method is already in application for through-transmission-welding of thermoplastics and fast drying in coating industry[66,67]. Some NIR absorbing dyes and pigments including carbon black, Lumogen IR765, indocyanine green, lanthanum hexaboride (LaB_6), etc. are applied in the welding, coating and drying industries. Dosser *et al.*[68] investigated the joining of plastics by carbon nano-particles (including nanotubes, Bucky balls, etc.). Recently, Kubota *et al.*[69] investigated the OOA curing of thermoplastic composites by specially developed NIR absorbing nano-particles. The same heating process by much stronger NIR absorption Si-Au nanoshells[70,71] and Au/Ag nano-particles[72] were investigated in medical applications for non-invasive photo-thermal ablation of inner-body tumours.

4. CONCLUSION

Low-cost and high-efficient curing method for fabrication of advanced polymer composites is of great academic and industrial interest, and it still remains as a great challenge and thorny issue. To select a proper curing method, the following criteria, including material (availability, universality and potential), applicability (curing/heating speed, know-how, handling complexity and controllability), penetration capability (polymer matrix, carbon fiber, and glass fiber/natural fiber), and cost (investment, running and maintenance) needs to be fully considered. **Table 1** shows the evaluation of the curing methods described according to the application criteria and requirements. This table also provides a good selection basis of current heating and curing processes for different application areas of polymer and polymer composites according to the specific technical requirements.

Due to the special energy absorption mechanism in radiation curing, radiation provides unique technological superiority compared to thermal curing, including fast curing speed, energy efficiency, improved resin stability and etc. Besides, it offers considerable control and some process latitude to manufacture complex and large structures, in contrast to thermal curing techniques. However, the application of radiation curing needs specially prepared radiation sensitive material, which is currently not mature as thermal curing material. Besides, though high energy radiation sources, including gamma ray, x-ray and electron beam, can provide deep penetration into

Table 1. Evaluation of different curing methods for advanced polymer composites

Curing methods \ Criterions			Material			Applicability				Power (Penetration)			Cost		
			Availability	Universality	Potential	Curing/ heating speed	Know-how	Handling flexibility	Controllability	Polymer matrix	Carbon fiber	Glass/ natural fiber	Investment cost	Running cost	Maintenance cost
Radiation curing	Gamma ray, x-ray		−	+	+/−	+	+/−	−−	−−	++	++	++	−−	−−	−−
	Electron beam		−	+	+	++	+/−	−	−	+	+	+	−	−	+/−
	Ultraviolet		+/−	+/−	+	++	+/−	++	+	+/−	×	+/−	+	++	++
Thermal curing	Radiation heating	Infrared	++	+	+	+/−	+	++	++	+/−	−	+/−	+	++	++
		Laser	++	+	+	+	+	+	++	+/−	−	+/−	+/−	+	+
		Microwave	++	+/−	+/−	+	+/−	−	+/−	++	−	++	+/−	+	+
	Convection & Conduction heating	Hot gas	++	+	+/−	+/−	+	+	+	−	−	−	+	+/−	+/−
		Flame	++	+/−	+/−	+/−	+	+	+/−	−	−	−	+	+	+
		Oven	++	++	+/−	+/−	+	+	++	−	−	−	+	+	++
		Hot shoe	++	+	+/−	+	+	+/−	+	−	−	−	+/−	+	+
	Induction		++	+/−	+	+	+/−	+	+/−	×	+/−	×	+	++	+
	Ultrasonic		++	+	+	−	+/−	+/−	+/−	++	++	++	+/−	++	+
	Resistance		++	+/−	+	+	+/−	+/−	−	×	+	×	+/−	+/−	+
	Particle-aided heating	Magnetic particles heating	++	+	+	+	+/−	+	+	+	×	+	+	+	++
		NIR particles heating	+	+	+	+	+/−	+	+	+	−	+	+	++	++

(++) very good; (+) good; (+/−) moderate; (−) bad; (−−) very bad; (×) not applicable

carbon fibre-reinforced polymer composites, they lack cost efficiency and handling flexibility considering the highly radioactive nature, while lower energy UV cannot provide efficient curing considering the very limited penetration in semi- and non-transparent polymer composites.

Thermal curing is by far most popular curing method for polymer composites, considering the mature thermal curing material system and curing processes, but still challenged by the curing efficiency. Conventional infrared and laser radiation heating methods face the issue of "surface heating" mechanism, while microwave radiation is limited by the coupling nature with carbon fibre-reinforced composites. Convection and conduction heating methods in which heat transfer by temperature gradient lead to long cure cycles because of the low heat conductivity of polymers. Other newly emerged heating methods including induction, ultrasonic and resistance and thermal additives heating methods provide new potential alternatives advanced polymer composites fabrication, but they still need further investigation relating the curing mechanism and efficiency, flexibility and etc.

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