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Determination of Optimum Cure Parameters of 977-2A Carbon/Epoxy Composites for Quickstep Processing

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ABSTRACT: In this study polymer-matrix composite has been processed using autoclave and Quickstep techniques. A phenomenological approach was adopted for the optimization of cure cycle for Quickstep of an aerospace grade epoxy composite (Cycom 977-2A carbon/epoxy prepregs). Though there are reports concerning the processing and properties of materials using Quickstep technique, little work has been reported on the design of optimized cure cycle for Quickstep processing based on rheology, ultrasonic testing, and mechanical characterization. A step by step account is provided in this study, describing the introduction, balancing and combination of isothermal dwell periods and temperature ramps in order to control the resin viscosity. All the panels manufactured were qualitatively assessed using ultrasonic C-scan and afterwards fiber and void content, panel thickness, flexural strength, and inter-laminar shear strength (ILSS) were assessed and compared with those obtained from traditional autoclave- and oven-cured laminates. The Quickstep panel properties produced from optimized cure cycle was superior to oven curing and was comparable with autoclave.
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INTRODUCTION

Fiber reinforced polymer composites have gained substantial popularity over the recent years mainly due to their ability to combine the properties of different materials which allows them to manufacture products having very effective and versatile properties. This has led to the use and application of composite materials in several high performance industries like aerospace and automotive. The major disadvantages of these materials, however, are high processing and equipment costs and difficulty of manufacturing. These parameters are even now gaining prominence in aerospace and automotive industries. Currently, high performance composites parts are predominantly manufactured by the use of prepreg materials and autoclave cure. This requires high manufacturing temperature, pressure and a cycle time of several hours. As the aerospace time scale requirements are shortening, an increase in production rates is required and this is difficult to achieve with traditional autoclave manufacturing. Also, high capital expenditure, infrastructure requirements and time to commission has made autoclave processing, increasingly undesirable.[1,2] Thus, the area of need has arised in terms of speedy manufacturing and low production cost without sacrificing part quality. Quickstep is a novel polymer composite processing technique designed and developed in Australia for out-of-autoclave processing of high quality, low cost components in comparatively short cure cycle times.[3–6] In contrast to autoclave processing, the Quickstep process is a low pressure process where approximately 10 kPa pressure is applied during processing as compared to 700 kPa external pressure, typically applied during autoclave processing.

Some research has been carried out regarding the processing and properties of materials using Quickstep technique.[7,8] Voids has been considered as the most common manufacturing induced defects in the composites and were found to have deleterious effects on most of the matrix dominated properties like inter-laminar shear strength (ILSS), compressive strength and bending strength. It has also been reported that voids may cause a significant decrease in fatigue life and cause a greater susceptibility to moisture and water absorption and environmental conditioning.[7,8] Voids usually formed in the composite structures due to many reasons; some of them are (i) due to dissolved volatiles within premix resins (ii) bubbles evolved from mixing process itself (iii) air pockets trapped during lay-up[9] (iv) due to

absorbed moisture during storage (v) due to resin rich areas in the cured part[10] (vi) due to volatile by-products during the cure reaction of the polymeric matrix (vii) due to use of high viscosity resin combined with closely packed fiber that are not completely wet out by resin (viii) due to fabrication mishaps such as leaking vacuum bag or a poor vacuum source.[11] However; it has been possible to control the void formation during the curing process. The processing of a void free laminate has found to be strongly dependent on the rheological behavior of resin system during manufacturing. "Rheology" refers to the flow process which the resin experiences, when subjected to temperature and/or pressure during cure. Several studies have been conducted[12,13] to investigate the cure cycle effect on the void content and then on ultimate properties. Most of the studies were conducted for autoclave processing and the supplier's recommended temperature schedule was used in order to avoid any change in the properties due to modification in the thermal profile.[14] In all of these studies, the maximum pressure and most suitable time of pressure application for minimum void content and optimum properties were reported. Defects or flaws in composite materials can be process-induced or service-related. One of the process-induced defects encountered in composites was due to variation in the degree of cure (under-cure or over-cure), which occur if proper temperature and/or time are not used in the moulding process.[15] If laminate was under-cured due to insufficient time in the mould, it caused the matrix to have lower properties compared with when fully cured and to be susceptible to creep.[16] Thus, the matrix-dominated properties like transverse tensile strength, in-plane shear strength and interlaminar shear strength would be reduced. Furthermore, the laminates may not be properly consolidated which may result in the development of fine inter-laminar cracks or delamination. Conversely, if the laminate has been over-cured, it results in the brittle matrix, which is then susceptible to crazing under stress.[15] Researchers[17] have been utilized and recommended differential scanning calorimetry (DSC) to determine the degree of cure and for developing cure cycles of thermosetting resin and prepregs. DSC can measure the heat flow required to maintain a sample at a given temperature, thus providing information on cure and reactions.[18]

The Quickstep technique functions by rapidly applying heat to the laminate, which is sandwiched between two floating rigid or semi rigid moulds, through heat transfer fluid (HTF) (Figure 1). Because of the flow of HTF, a ramp rate of up to 15 K min^{-1} can be achieved with Quickstep which is much faster than 2–3 K min^{-1} ramp rate usually employed for autoclaves. The HTF also acts as a large thermal sink to remove any excess heat generated by exothermic curing reaction, thus allowing a well maintained and controlled temperature throughout the cure cycle. The cure schedule provided by the material's manufacturer is usually designed for autoclave or other traditional processing techniques and the thermosetting resin systems are usually formulated for low ramp rate curing (typically 2-3 K min^{-1}). While in case of Quickstep, as mentioned above ramp rates up to 15 K min^{-1} can be achieved, thus change the chemorheology of the resin system. This has been the reason that when the cure cycle suggested by the material's manufacturer has employed for

Figure 1. Quickstep floating mould beds. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Quickstep processing, laminates having high void content and poor structural performances have been observed.[3] Thus, in order to produce a void free laminate of comparable physical and mechanical properties to autoclave, a complete understanding of the changes in the viscosity and reaction progress during the cure cycle was required.

An understanding of the formation and development of voids during autoclave processing has been sought by several researchers. Since, Dave et al.[19] proposed a model describing the dynamic processes occurring in water vapour voids and trapped air voids during the autoclave cure cycle of a composite prepregs system. The conclusions drawn from the modelling exercise suggested that the potential for void growth during the cure schedule could be eliminated if the resin pressure at any point satisfied the following equation:

$$P_{\min} \geq 4.4962 \times 10^3 \exp\left(\frac{-4892}{T}\right)(RH)_0$$

where $P_{\min}$ is the minimum resin pressure (atm) (in this case the external pressure), T is the temperature (K), and $(RH)_o$ is the relative humidity at room temperature. It signifies that in order to avoid the formation of voids during the autoclave cure cycle, the autoclave and vacuum bag pressure should be adjusted to follow the temperature-time history such that the resin pressure at every point in the laminate must be greater than $P_{\min}$.

The aim of this work was to determine the optimum processing parameters of an aerospace grade material (977-2A Carbon/epoxy prepregs) for Quickstep processing. A step by step account is provided, describing the introduction, balancing, and combination of isothermal dwell periods and temperature ramps to control key resin characteristics. All the panels manufactured were quantitatively assessed using ultrasonic C-scan and after that specimens were cut from the panels for physical and mechanical characterization. In the ensuing discussion, physical characterization refers to fiber content; void content and panel thickness along with mechanical characterization refers to flexural strength and inter-laminar shear strength (ILSS). The physical and mechanical properties of the autoclave-cured panel

were considered as a benchmark in this study for comparison with those cured in oven and Quickstep. It is pertinent to note that this paper only addresses the optimization process using rheological analysis of resin system, however, a comprehensive study was carried out which include cure kinetics and hygrothermal behavior of this specific resin system. Some aspects have also been addressed in the literature.[6]

The objectives of this research project, thus, have been accomplished and it was shown that QS-60 (Quickstep cure cycle with intermediate dwell time of 60 min) cure was the most appropriate cure cycle for Quickstep manufacturing to manufacture a well-consolidated composite laminates with comparable physical characteristics to its counterpart, autoclave curing. The basic knowledge of the laminate's response to QS-60 cure was obtained and compared with autoclave curing. However, several areas of research need to be uncovered to fully characterize and processing of composites using Quickstep, its response to adverse environmental exposure and the effect of dynamic loading on the physical and mechanical properties. This optimization is applicable to a wide range of thermoset resins. Normally, a resin may flow in the directions normal (z-direction) and parallel (x-y direction) to the plane of the composites. In actual environment, the flow in the x-y direction is often negligible because of width and length is very large as compared to thickness of the laminates and because the restraints placed around the composite. In this work, the panels manufactured were typically 2 mm and 3.5 mm thick and the cure schedule optimised were suitable for manufacturing components of this dimension. Double vacuum bag (DVB) technique was coupled with Quickstep processing and a slight improvement in the cured panel was observed, though, a variation in the properties of fabricated laminates were observed due to different bagging arrangements and thus, it is believed that by controlled application of vacuum pressure during Quickstep curing can be useful in reducing the time required to squeeze out excess resin. Consequently, care should be taken when manufacturing thick laminates as excess resin may not be squeezed out using this cure schedule. Some suggestions have been regarding the intermediate dwell time that may increase and/or the intermediate dwell temperature may be lower for thick composites in order to facilitate the excess resin to squeeze out from laminates. The cure cycle optimization presented in this work was specific to 977-2A carbon/epoxy prepregs. However, the same principle provided in this study (which is based on lower viscosity achieved through quickstep processing allow high quality laminates to be produced at low pressure) can be utilized for other epoxy systems. The exact replica of optimized cure cycle provided in this study may not be suitable for other thermoset resin systems; however, the steps of optimization process presented in this study could be performed to obtain cost-effective cure schedule for specific system. Thus, this study provided general guidelines for cure optimization of thermosets (especially aerospace grade epoxies) for Quickstep processing.

EXPERIMENTAL

Material System and Lay-Up Procedure

The material being used for manufacturing all the panels (autoclave/oven/Quickstep) was Cytec Cycom®977-2A based prepregs containing epoxy resin with woven 6KHTA carbon fiber with cured ply thickness of 0.362 mm. For the viscosity measurement, uncured 977-2A epoxy resin was used. Since a five harness satin single ply is asymmetric about its mid plane and either the warp or weft tows run predominantly in one direction on either face, so plies need to be laid-up in such a manner to ensure that the laminate has balanced fiber direction through its thickness, to avoid any fiber distortion in the part after cure. For this purpose, it was assumed that side dominated by warp fibers is on the top while if it were on the bottom, it would be considered to be flipped. In this study, the plies were stacked in the warp direction back to back like flipped pairs so that the whole stacking sequence was semi-symmetric about the mid plane $\{0/0_f\}$.[20] All of the laminates used in this study consisted of six plies $\{0/0_f\}_3$, 200 mm wide and 160 mm long resulting in the nominal thickness of 2 mm with the fibers aligned in the warp directions.

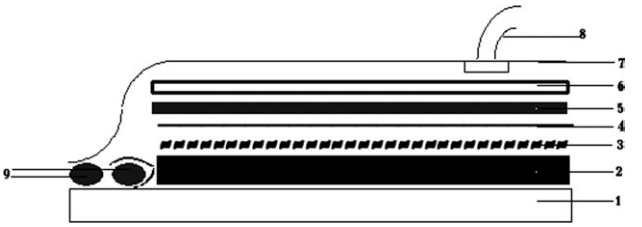

Figure 2. Vacuum bag assembly for autoclave, Quickstep and oven curing (1) Tool plate and release film (2) Laminate (3) Peel Ply (4) Solid release film (5) Caul Sheet (6) Breather (7) Bagging film (8) Vacuum Line (9) Sealant tape.

Vacuum Bagging Lay-Up

Composites for oven, autoclave and Quickstep curing were fabricated using a conventional vacuum bag lay-up as illustrated in Figure 2. The laminates were debulked three times for 15 min after two, four, and six plies had been laid-up. The specifications and application of the bagging material used in this study are provided in Table I.

Autoclave and Oven Curing

The manufacturer recommended cure cycle was employed for oven and autoclave curing and steps of the cure cycle are provided in Table II. A consolidation pressure of 7 Bars was applied and vacuum consolidation of at least 96 kPa was achieved throughout the cure cycle for autoclave curing. Three panels were manufactured and analysed for comparison. A conventional thermal oven (Townson and Mercer) was employed to cure 977-2A carbon/epoxy prepregs in order to simulate the autoclave cure cycle without any over- consolidation pressure. A vacuum consolidation of at least 96 kPa was achieved throughout the cure cycle for oven curing. Three panels were manufactured and analysed for comparative study.

Quickstep Cure

A number of cure cycles were being employed for Quickstep processing and can be categorised as QS_{Direct}, QS_{ID}, and QS_{AD} (ID referred to intermediate dwell and AD for additional dwell). The curing steps involved in these cycles are listed in Table II. Four different cure cycles were employed for QS_{AD} cycle and

Table I. Specification and Application of Bagging Materials

Material	Specification/application
Tool Plate	An Aluminum tool plate of size 900mm x 900mm x 6mm was being used as tool plate
Release Film	Glass reinforced PTFE supplied by Aerovac System is used as release film. This is placed between laminate and tool plate to prevent the adhesion
Caul Plate	An aluminium Caul plate of 250mm x 200mm x 3mm is used on top of the laminate and inside the bag to define the second surface
Bleeder/Breather Cloth	The bleeder / breather fabric used was an ultra weave 1032 nylon material provided by Tygavac Ltd. This fabric is used between the Caul plate and the bagging film. The functions of this cloth are to soak up the excess resin and reactants bled from the laminates and provide an air passage to laminate so that uniform pressure may be applied on the laminate
Bagging Film	An Elastomax 224 nylon supplied by Aerovac Systems Ltd was used as bagging film. This is a flexible membrane, which permits vacuum to be drawn within the bag
Tacky tape	Tacky tape provided by Schnee Morehead Inc. are used as a sealant tape and used to bond completely the bag to the tool
Breach unit	It is a connector passed through the bagging film to permit vacuum to be drawn
Vacuum Pump	A high-volume pump was being used in the study which was capable of providing a consolidation pressure of up to 102 kPa

distinguished by the intermediate dwell time at 130°C. The intermediate dwell times were 35, 45, 60, and 75 min for QS_{AD} cycle. Three panels for each cycle were fabricated under the conditions stated above.

Ultrasonic C Scan

Ultrasonic C-scan inspection is a common nondestructive testing method used for high-performance composites. In this method, the relative attenuation of ultrasonic waves across the surface of the composite part is measured. An ultrasonic transducer is used to scan the surface of a material using a computer generated raster scanning pattern. C-scan units digitize the collected signals, which subsequently can be used to produce a visual representation of the subsurface. There is always an energy loss through the thickness of composite parts and magnitude of this loss is altered when there is a defect in the part like resin rich or starved areas, voids, thickness variation, distortion in the fiber orientation, etc. To evaluate the exact nature of defect, a thorough microscopic examination is required. All the panels manufactured in this study were qualitatively assessed using a Midas NDT ultrasonic C-scan system. A 5 MHz frequency probe, dynamic range and the scan speed of (0–74) db and 200 mm sec^{-1} respectively were used for all the scans. From these images one can easily observe, if there is a defect that would cause the part to be discarded. Nevertheless, to exactly quantify the analysis process of the C-scan images for comparison purposes, an averaging technique was used[21] in which a (25 × 25) mm square was employed to define a small region on the image and both mean and standard deviation using a histogram tool in the software (Zeus 3.0) was determined. Since, there was not

Table II. Steps of Cure Cycles Employed in This Study

Autoclave/oven cure cycle	QS_{Direct} Cycle
a. Heat from room temperature to 130°C at 2 K min^{-1}	a. Heat from Room Temperature to 180°C at 10-12 K min^{-1}
b. Dwell at 130°C for 60 min	b. Dwell at 180°C for 180 min
c. Heat from 130°C to 180°C at 2 K min^{-1}	c. Cool from 180°C to room temperature at 8-10 K min^{-1}.
d. Dwell at 180°C for 120 min	
e. Cool from 180°C to room temperature at 2 K min^{-1}	
QS_{ID} cycle	**QS_{AD} Cycle**
a. Heat from room temperature to 130°C at 10-12 K min^{-1}	a. Heat from Room Temperature to 175°C at 10-12 K min^{-1}
b. Dwell at 130°C for 60 min	b. Dwell at 175°C for 10 min
c. Heat from 130°C to 180°C at 10-12 K min^{-1}	c. Cool from 175°C to 130°C at 10-12 K min^{-1}
d. Dwell at 180°C for 120 min	d. Dwell at 130°C for 35,45,60 and 75 Min
e. Cool from 180°C to room temperature at 8-10 K min^{-1}.	e. Heat from 130°C to 180 Dwell at 180°C for 120 min at 10-12 K min^{-1}
	f. Dwell at 180°C for 120 min
	g. Cool from 180°C to room temperature at 8-10 K min^{-1}.

Table III. Porosity Level Depicted from C-Scan Images

Description	Ultrasound attenuation
Porosity is negligible – panel is considered good. Attenuation (dB) levels are low.	dB<=8
Medium levels of porosity – micrographs required to confirm actual part quality. OR Higher levels of attenuation but low standard deviation – doesn't usually suggest high porosity	8< db with low standard deviation
Porosity is high – panel is considered bad quality.	8< db with high standard deviation

any provision in the software to store these histograms; the values are shown in the Table format. The step was repeated 15 times on different locations of the image and the maxima and minima were determined. After that the part quality can be categorised as shown in Table III. Different locations of the panel at which these (25 × 25) mm squares were selected are shown in Figure 3.

Rheological Aspects

The viscosity of neat 977-2A resin was determined as a function of time and temperature, using 50 mm diameter parallel plates in a Rheometrics RMS 800 System. The gap between the plates was set at 1 mm for all the measurements. The method for determination of gel point (GP) is noticeably explained in results and discussion section. Three samples were tested for each viscosity profile presented in this study.

Hot Acid Digestion

The fiber volume fraction and void content were determined by using hot acid digestion method according to BS 2564.[22] Five specimens were selected from different location of the panel and tested from each manufactured panel.

Mechanical Testing

Flexural and inter-laminar shear strengths (ILSS) testing were carried on using an Instron 4411 test apparatus using three point bend jig according to ASTM D790[23] and ASTM D2344,[24] respectively. The dimensions of the flexural and ILSS specimens were 60 × 15 × 2 mm^3 and 20 × 10 × 2 mm^3 with support spans of 40 mm and 10 mm, respectively. Five specimens were considered from each manufactured panel for each (flexure and ILSS) tests.

Thickness Measurement

The thickness variation of the fabricated composite panels and specimens were assessed using Digimatic thickness gauge. The thickness was measured at different points equally distributed across the panel or specimen surface according to ASTM standard.

RESULTS AND DISCUSSION

Rheological Study of 977-2A Neat Resin

The viscosity of a thermosetting resin undergoing a curing reaction is a function of time, temperature and degree of cure.[25] For polymeric molecules, motion can occur on a variety of length scales, from the relaxation of molecular segments to diffusion of entire chains. There will be a range of characteristic times at a given temperature corresponding to motion at different length scales, with longer times characterizing motion on larger length scales. If a sample is cooled at a certain rate, the mobility modes corresponding to longer relaxation times will freeze out before the mobility modes corresponding to shorter relaxation times. Despite the large difference in the length scale and character of the different motions of a molecule, the temperature dependence of the viscosity of a thermosetting resin, characterizing whole chain diffusion, is typically very similar to that for the characteristic times corresponding to the glass transition (α-relaxation). Specifically, the temperature dependence of the viscosity and the α-relaxation times is well-described by a Vogel-Fulcher function of the form as:

$$A \exp[T_A/(T - T_0)]$$

Where T_A is activation temperature; T_0 is the temperature at which exponential factor diverges to infinity; A is a temperature independent prefactor. The similarity of the temperature dependence of dynamic processes that occur on different length scales is a phenomenon known as *thermorheological* simplicity, and it is the basis for time-temperature superposition which is used extensively in the study of the mechanical properties of bulk polymers.[26] Figure 4(a) shows the viscosity profile of 977-2A resin as a function of the heating rate. The resin was heated at heating rates of 2, 5, 10 and 15 K min^{-1} to 180°C, where the temperature was held until the curing of the resin occurred. The minimum resin viscosity, time and temperature at minimum resin viscosity are presented in Table IV. Two concurrent phenomena governed the rheological behavior of the reacting system [Figure 4(a)]. First one was associated with the decrease of viscosity with increase of temperature due to intensification of the molecular mobility (as the temperature was increased from 70°C to 180°C) and the viscosity reaches its minimum value. Secondly, the molecular mobility started to decrease, thus increase the viscosity owing to growing of molecular network (polymerization and cross-linking) during the cure.[25] Minimum viscosity is one of the property-determining factors in the processing of polymeric composites. It has been reported that lower

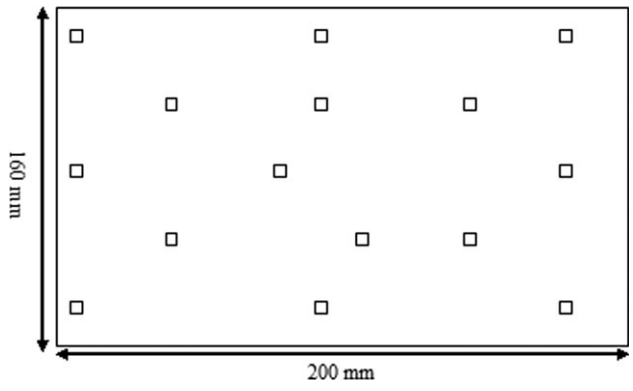

Figure 3. Location of selected squares (25 x 25) mm^2 for C-scan analysis in all cured panels.

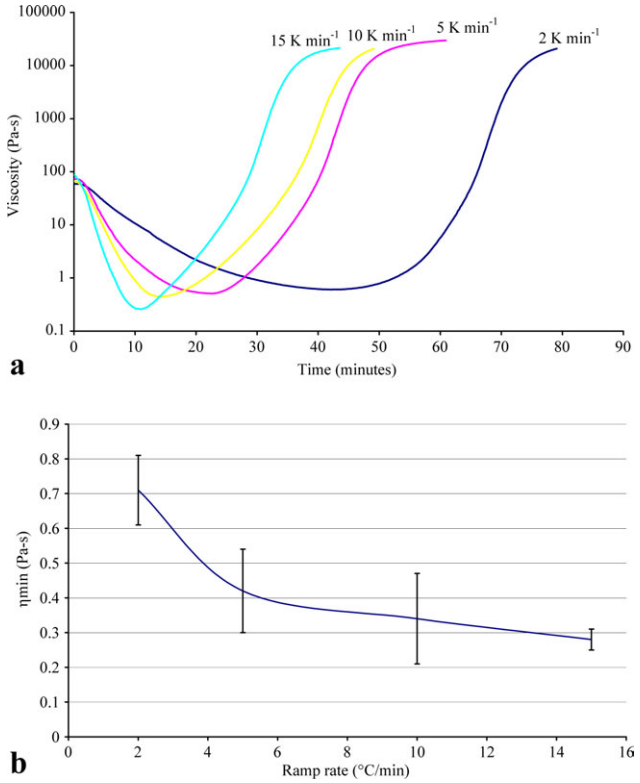

Figure 4. (a) Viscosity Profile of 977-2A epoxy resin as a function of heating rate. (b) Relationship between ramp rates and minimum viscosity. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

viscosity during the cure cycle accounts for lower void content and improve the adhesion properties due to enhance tendency for fiber wetting.[27] Figure 4(a) shows that time required to reach the minimum viscosity of the resin decreased with increasing heating rates. However, the values of minimum viscosity remained statistically insignificant at ramp rates higher than 2°C min^{-1} as evident from Figure 4(b). The relationships among heating rate, time and minimum viscosity lead to an interesting observation that minimum viscosity can be achieved at slightly higher ramp rate, however, the processing time can greatly be reduced by selecting suitable ramp rate. From Table IV, it can be perceived that time to reach the minimum viscosity was significantly reduced from 43 to 10 min as the ramp rate increased from 2 to 15 K min^{-1}. As mentioned above that the parameters affecting the viscosity of thermoset resins are the

Table IV. Viscosity, Time, and Temperature at Minimum Viscosity for Different Heating Rates

Ramp rate (°C/min)	η_{min} (Pa s)	Time (min)	Temperature (°C)
2	0.71 ± 0.1	43.17 ± 0.21	154.7 ± 1
5	0.42 ± 0.12	22.60 ± 0.42	178.5 ± 2
10	0.34 ± 0.13	13.35 ± 0.96	179 ± 1
15	0.28 ± 0.03	10.30 ± 0.85	180 ± 1

temperature and time, so, the molecular mobility were found to be strongly dependent on the heating rates. Similar trend has been reported by Davies et al for 6376 neat epoxy resin.[3] These factors are also important in the present work, as a ramp rate of 15 K min^{-1} can be achieved through Quickstep as compared to processing of composites in autoclave which usually worked on 2–3 K min^{-1}. Accordingly, the lower minimum viscosity reached facilitated improved resin wet-out of the laminates during Quickstep processing, and was advantageous. Apart from the viscosity execution, another important factor determined from rheological analysis is the gel point (GP). It has been known that if the minimum viscosity is extremely high or the GP is attained rapidly during processing, the consequences may include improper fiber wetting and poor adhesion between fiber and matrix.[28] The determination of GP is, therefore, very important as it plays a vital role in the prediction of mechanical property increase during the cure of thermosets resin.

In the literature, different methods have been proposed to determine the GP from rheological measurements.[29–32] In this investigation, the GP is determined by Fourier Transformed mechanical spectroscopy (FTMS) that effectively decouples the frequency dependence and time dependence of the fluid properties. In this method, a multi-wave oscillatory strain is applied and the resulting stress is measured. The GP is determined by taking several simultaneous multi-wave frequency sweeps to measure tan δ in the gelation time-scale. Seeing as, tan δ is independent of the frequency at GP, the curves pass through a single point and define an instant where the GP forms. The method is much appropriate for transient structures that require speedy testing, because the structure may change during the test. A typical diagram of tan δ *vs.* time in the gelation region for 10 K min^{-1} viscosity profile is shown in Figure 5. Different data points converge at a single point where the GP appeared. As noted in Figure 5, the rheological data was scattered and somewhat obscured by the noise in low frequencies which may be induced due to enhanced thermal fluctuations. Nevertheless, the gel points could be obtained from the convergence of data as evident from this Figure. 'Processing window' usually provides a guideline to minimize internal stress levels and to prevent void formation during the cure of thermoset composites.[33] This study explored the advancement of the viscosity and the

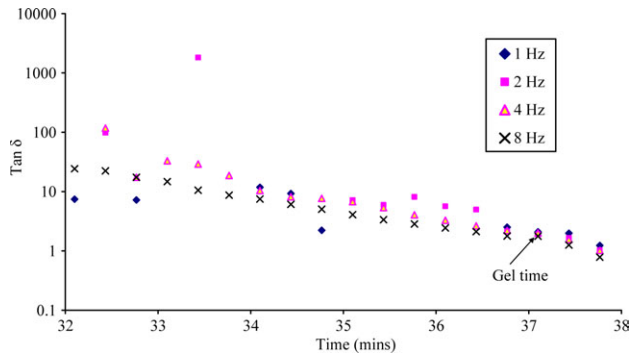

Figure 5. Typical tan δ versus time diagram in the gelation region for 10 K min^{-1} viscosity profile. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table V. Mechanical Properties of Autoclave/Oven and QS Panels

Panel	Flexural Strength (MPa)	ILSS (MPa)
Autoclave	1389 ± 30	81 ± 4
Oven	1093 ± 31	64 ± 4
QS_{Direct}	1019 ± 18	54 ± 5
QS_{ID}	1129 ± 28	65 ± 3
QS_{AD} (QS-60)	1258 ± 46	84 ± 2
QS-35	1228 ± 86	70 ± 10
QS-45	1301 ± 30	76 ± 4
QS-75	1118 ± 51	71 ± 2

point of gelation as a function of resin flow and void formation/removal for Quickstep processing. The boundaries of the processing window were restricted to GP in this study.

Autoclave/Oven Cure

The cure schedule employed for oven and autoclave curing is the one which was recommended by the material's manufacturer.[34] Several studies were conducted for autoclave processing and the supplier's recommended temperature schedule was used in order to avoid any change in the properties due to modification in the thermal profile,[3,35] however, reservation of using materials supplier's cure cycle for autoclave processing is also exist.[36] Our primary focus was to investigate the optimum processing parameters for Quickstep processing and it was found that the supplier's cure cycle-cured autoclaved panels were of superior quality with high fiber volume fraction and low void content (less than 1%). Table V presents the average values of flexural strength and interlaminar shear strength (ILSS) obtained for the manufactured composite panels. Whereas, Table VI listed the fiber volume fraction, void content, and average panel thicknesses. From both the Table, it can be seen that autoclave-cured panel exhibited superior physical and mechanical properties as compared to oven-cured panel. The ultrasonic C- scan and representative micrographs (20x) of oven and autoclave-cured panels are shown in Figure 6. It was experiential that significant dB loss occurred in the oven-cured panel while negligibly small dB loss observed in the autoclave-cured panel (Figure 6 and Table VII). The microstructures confirmed the presence of porosity in the oven-cured panel as the major defect. A uniform distribution of fiber/resin over the entire panel was, so, obvious for autoclave-cured panel and the void content in the autoclave-cured panel was typical for high performance application (< 1%). Since the oven and autoclave panels were manufactured using a similar thermal profile, the comparison of these processes indicated the significance of a high pressure process on the properties. The higher flexural and ILSS and lower panel thickness values of autoclave-cured panels in Tables V and VI were attributed to higher fiber volume fraction and lower void content achieved through high consolidation pressure. The fiber volume fraction and void content of the oven-cured panel showed (Table VI) higher standard deviation values, which can be attributed to different sizes and nature of voids present and nonuniformity of fiber/resin distribution in the panel.

Quickstep Cure

The cure cycle profiles and vacuum pressure achieved for all the Quickstep cure cycles are shown in Figure 7. Total cycle time for autoclave/oven processing was recorded as 340 min (Table II), although owing to the increased ramp rate, most of the cycle time for Quickstep cure was in the range of 220-260 min. Two thermocouples were attached to the part for the real time monitoring of the temperature as shown in all thermal profiles and the cure cycle was based on these thermocouple readings. A pressure sensor available in the plant fitting also continuously monitored the vacuum pressure achieved throughout the process. Quickstep processing provided greater flexibility to control the cure cycle temperature in order to manipulate the key resin characteristics specially viscosity as compared to autoclave. The processing window obtained for autoclave/oven and Quickstep curing is shown in Figure 8. The change in viscosity of neat 977-2A resin over the heating profiles (steps in Table II for autoclave/oven and Quickstep curing) was provided. The starting point of the development of Quickstep cure cycle was the two cure cycles recommended by the material supplier[34] but with higher ramp rates achievable through Quickstep. The two cure cycles were termed as QS_{Direct} and QS_{ID} and the steps of these cycles are provided in Table II. Figure 9 presents the ultrasonic C scan and representative micrographs (20x) of QS_{Direct} and QS_{ID}-cured panels. Table VI and Figure 9(a) showed that QS_{Direct}-cured panel exhibited high void content resulting in the poor mechanical performance as compared to autoclave. Although the resin viscosity quickly reached a minimum value but at high curing temperature (180°C), the resin quickly gelled, thus the time to exploit this for consolidation significantly reduced causing resin rich areas in the panel (Figure 8). It has been reported previously that excess resin must be squeezed out before the gelation,[37] the lower fiber volume fraction values in Table VI confirmed that inadequate flow of resin occurred during the processing. The higher void content and low fiber volume fraction was thought to be responsible to increased panel thickness. Table V revealed that 33 % reduction in the mean ILSS values was observed for QS_{Direct}-cured specimens as compared with autoclave-cured specimens, which was believed to be due to high void content in the specimens. Low fiber volume fraction due to resin rich areas also affected the flexural strength of the specimens and 28 % reduction in the flexural strength was observed for QS_{Direct}-cured specimens. As QS_{Direct}

Table VI. Physical Properties of Quickstep and Autoclave/Oven-Cured Panels

Panel	Fiber volume fraction (%)	Void content (%)	Avg. panel thickness (mm)
Autoclave	63 ± 0.5	0.8 ± 0.1	1.95
Oven	56 ± 3.4	4.6 ± 2.4	2.09
QS_{Direct}	54.6 ± 3	6.1 ± 1.4	2.23
QS_{ID}	57 ± 3	4.8 ± 1.1	2.14
QS_{AD} (QS-60)	61 ± 1	1.7 ± 0.2	2.0
QS-35	58 ± 3	4.25 ± 1.1	2.10
QS-45	60 ± 2	1.98 ± 1.12	2.04
QS-75	64 ± 3	1.5 ± 0.4	1.98

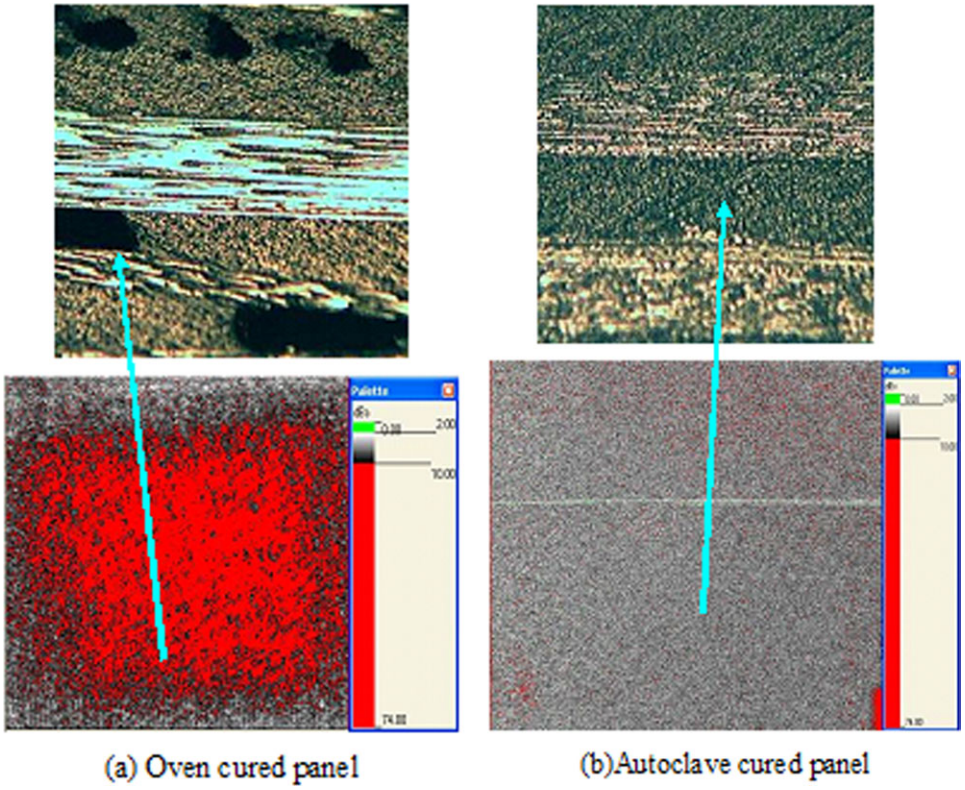

Figure 6. C-Scan images of panels cured in (a) oven (b) autoclave and their representative micrographs. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

cure schedule failed to produce a void free laminate and the primary reason of the bad quality of QS_{Direct} laminate was the lack of resin bleed because of short processing window, an intermediate dwell period of 60 min is introduced at 130°C in order to facilitate to squeeze out the excess resin from the laminates. To improve the mechanical performance, an intermediate dwell has been often used and is documented.[38] The dwell temperature of 130°C and dwell time of 60 min were chosen for quickstep processing (Figure 8), the resin viscosity remained low at this temperature and time period for autoclave cure. Table VII and Figure 9(b) depicted that relatively less spread in the dB loss occur for QS_{ID}-cured panel as compared to the QS_{Direct}-cured panels. However, the microstructures confirmed the presence of porosity in the specimens. Also, an extended processing window resulted in an increase in fiber volume fraction and decrease in void content for QS_{ID}-cured panels. An improvement in flexural strength, void content and average panel thickness (Tables V and VI) was observed for QS_{ID}-cured panels. The physical and mechanical properties remained significantly inferior compared with autoclave-cured panel. An additional dwell was introduced in the Quickstep cycle in an attempt to further improve the panel quality. In this cure cycle, the prepreg was quickly heated to 175°C and isothermal heating was applied for 10 min in order to allow the resin for proper fiber wetting. Although, the resin viscosity was still at minimum value, the temperature was rapidly reduced to 130°C where isothermal heating was applied for 60 min suitable for panel consolidation and so increased viscosity was solely a function of polymer cross-linking. The viscosity profile of 977-2A resin during the initial stages of the cure cycle employed for QS_{AD} cure is exposed in Figure 8. Upon heating, the viscosity of the resin decreased rapidly due to high heating rate (10 K min^{-1}), showing a minimum value of (0.4 ± 0.1) Pa s at 175 °C. The viscosity then steadily increased during the hold time at 175 °C owing to progressing polymerisation reaction and reaches a maximum value of (4 ± 2) Pa s at the end of dwell. When the resin was cooled down from 175 to 130°C, the viscosity increase was more rapid due to the combined effects of polymerisation and cooling effect and reached a value of (76 ± 8) Pa-s. Similar behavior of epoxy resin under cooling effect during cure has been observed by the researchers in the past.[3,39] The resin was subsequently allowed to flow for 60 min at intermediate dwell

Table VII. Maxima and Minima of Ultrasound Attenuation in the Quickstep and Autoclave/Oven Cured Panels

Panel	Maxima	Minima
Autoclave	6.00	5.50
Oven	12.10	7.06
QS_{Direct}	22.96	10.04
QS_{ID}	19.00	16.00
QS_{AD} (QS-60)	6.00	4.50
QS-35	20.04	10.30
QS-45	7.20	4.60
QS-75	11.00	8.20

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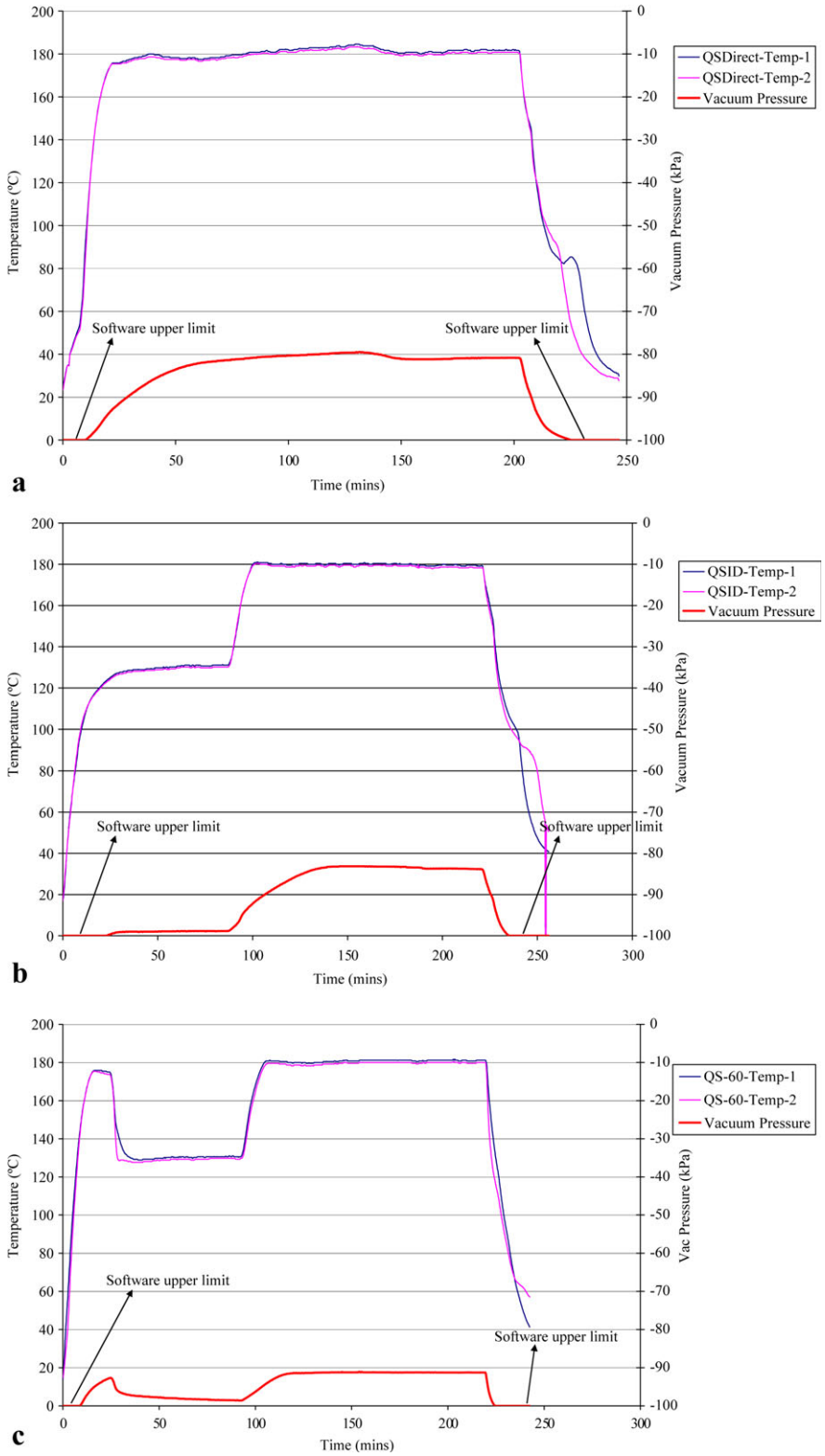

Figure 7. (a) Cure Cycle profiles for $QS_{Direct.}$ (b) Cure Cycle profiles for $QS_{ID.}$ (c) Cure Cycle profiles for QS 60. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

temperature of 130°C, where sufficient flow of resin occurred and the viscosity of the resin attained a maximum value of (1780 ± 180) Pa s. At second ramp, the viscosity of the resin decreased for certain time period and then increased again due to polymerisation. Eventually, the GP reached at the curing temperature of 180°C.

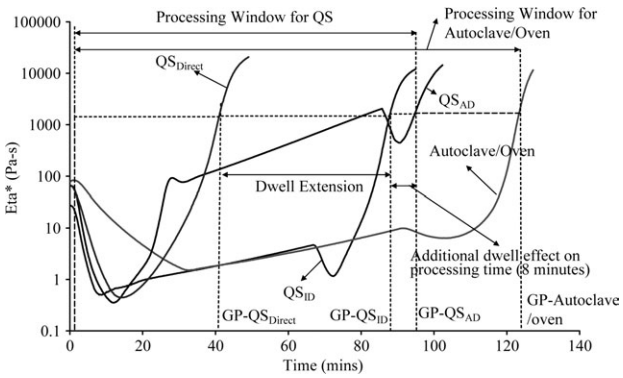

Figure 8. Viscosity profiles of autoclave and Quickstep cure cycles.

The ultrasonic C-scan for the panel cured using QS_{AD} schedule is given in Figure 10. It was observed that a uniform fiber/resin distribution was obtained for the entire panel cured using QS_{AD} cure schedule. Table VII depicted that dB loss value of only 6dB observed with negligible variation (4.5-6.0). The microstructures confirmed the production of void free laminate using QS_{AD} cure schedule. It is worth noticeable that the dB attenuation for QS_{AD}-cured panel was similar to autoclave cured panel, confirming that excess resin was squeezed out just by manipulating the resin viscosity during the processing which was a cheaper option relative to autoclave curing where high pressure application is required for the same. QS_{AD}-cured panel exhibited a slightly higher void content and lower fiber volume fraction than autoclave-cured panel, but the void content was relatively very low compared with $QS_{Direct-}$ and QS_{ID}-cured panels (Table VI). The slightly higher void content and lower fiber volume fraction can be attributed to the absence of high pressure application during Quickstep processing. QS_{AD}-cured specimens exhibited ILSS values comparable to the autoclave-cured specimens. The shear failure generally occurs due to shear stress and strain concentration in the matrix region between the fibers; promoting the interfacial failure. Hence, the ILSS of any material is a matrix dominant property and have been used for the determination and comparison of the adhesion strength between fiber and matrix. Here, void content played an important role because it greatly influenced the composite properties specially ILSS. It is, therefore, obvious that the lower void content of QS_{AD}-cured specimens (lower than other cured specimens except autoclave-cured specimens) account for the increase of interlaminar shear strength. In addition, this is also thought to be due to better fiber/matrix adhesion achieved through mechanical interlocking of the resin with the fibers. Good fiber/matrix adhesion was due to the improved wetting of fiber by resin as a result of lower viscosity. The lower viscosity during cure cycle was also accountable for lower void contents and higher strength. In case of flexural loading, the stress distribution is a function of location of fiber segment, thus flexural strength of any sample indicates that how well the load is transferred from one ply to the neighbouring ones. The mean value of flexural strength of QS_{AD}-cured samples although, was lower than autoclave-cured samples and thought to be due to the reason of the lower fiber volume fraction achieved through QS_{AD}

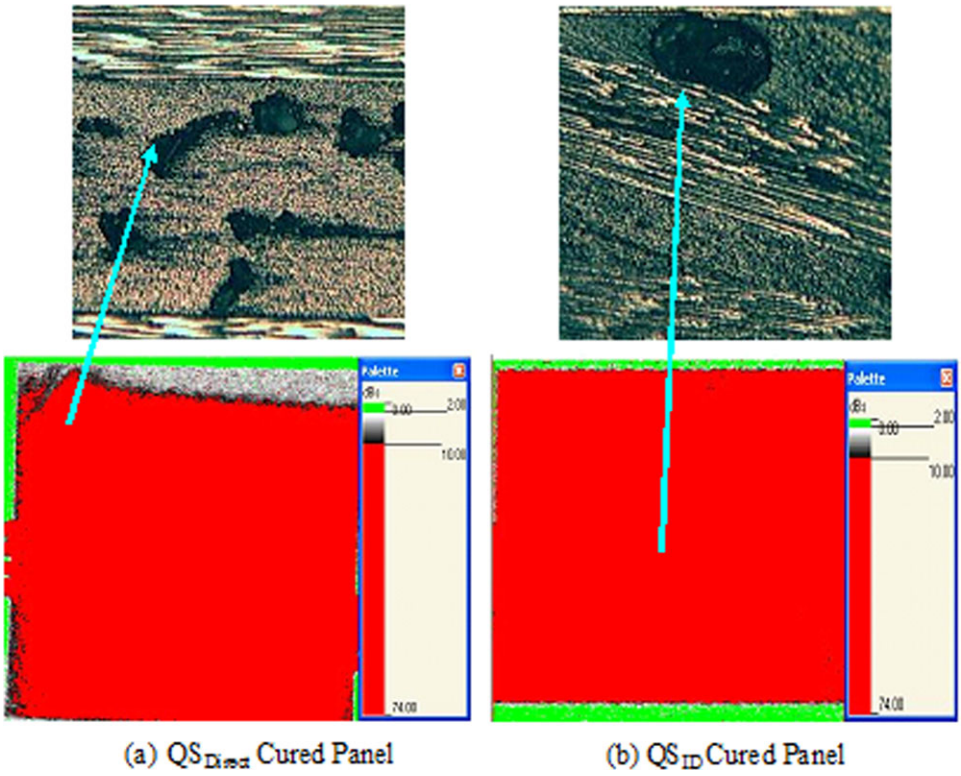

Figure 9. C- Scan images of panels cured using (a) QS_{Direct} (b) QS_{ID} cure schedules and their representative micrographs. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

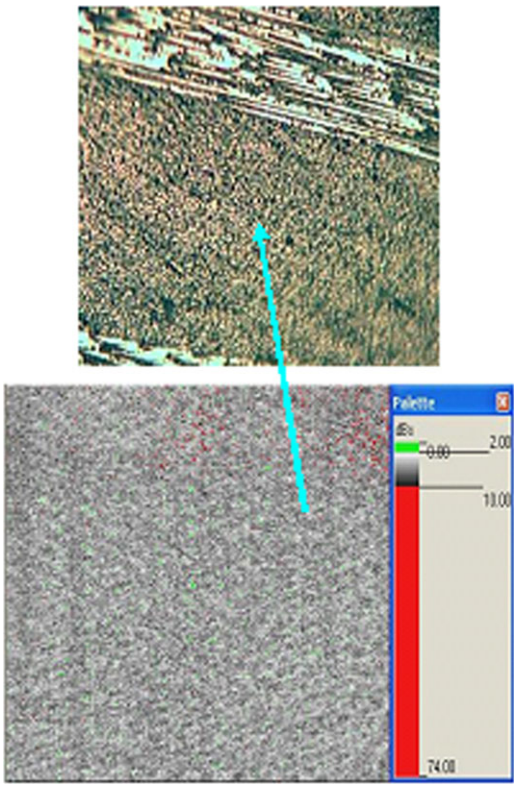

Figure 10. C- Scan images of panel cured using QS_{AD} cure schedule and associated micrographs. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

cure. Under flexural loading, the transition of the matrix material from elastic to plastic state is not uniform. It has been reported that the average bending strength of composite laminate depends upon fiber volume fraction and matrix yield strength

$$\sigma_{\text{bend}} = V_f * \sigma_f - (1 - v_f) * 3/2 * \sigma_y$$

where, σ_{bend} ,V_f, σ_f, and σ_y are the average bending strength, fiber volume fraction, fiber bundle strength, and matrix yield strength, respectively. Owing to lower fiber volume fraction and higher crosslink density,[6] quickstep-cured samples showed lower flexural strength as compared to autoclave-cured samples. The average panel thickness of QS_{AD}-cured panel was greatly reduced because of additional dwell effect in the cure schedule. The higher fiber volume fraction and lower panel thickness confirmed that excess resin has squeeze out during the processing. Although the average panel thickness was slightly higher than autoclave-cured panel which was again believed to be caused by high pressure application during autoclave processing.

Manipulation of Intermediate Dwell Time at 130°C (QS_{AD} Processing)

The isothermal hold at 130°C was an important factor, as this step has been used to allow the resins to flow and volatiles to escape. The dwell time at 130°C was also a significant aspect signifying the sufficient or insufficient flow of resin before the ramp to the final curing temperature. The second ramp (130–180°C) and hold (dwell at 180°C) was the cross linking portion of the cure cycle. During these stages, the viscosity rose dramatically to the gel point and additional cross-linking occurs. Thermal integrity and structural strength have been developed during this portion of the cycle.[40] The second ramp portion of the cure cycle has been critical from a void nucleation and growth standpoint as during this ramp the temperature was high, the resin pressure was at minimum and volatile vapour pressure was high and rising with temperature, these conditions have been ideal for void formation and growth.[40] Hence, it was important to minimize the void content by squeezing out excess resin in the stage (175–130°C) and intermediate hold time at 130°C of the cure cycle. In order to determine the optimum hold time at 130°C, QS_{AD} cure cycle was manipulated with intermediate dwell times of 35 (QS-35), 45 (QS-45), and 75 (QS-75) min. The actual cure cycle profiles of QS-35, QS-45 and QS-75 cure are shown in Figure 11. The average and standard deviation values of corresponding viscosities at GP were determined for all rheological measurements, which are shown in Table VIII. The maximum and minimum values of these corresponding viscosities were taken as critical viscosity range where GP can occur. It is noteworthy that in all cases, the GP was reached during the isothermal hold at 180°C (Figure 8). It was thus assumed that for 977-2A neat resin, a wide range of dwell time options may be possible for resin flow at 130°C. An attempt was made to correlate the resin viscosity with the intermediate dwell time at 130°C to determine the optimum hold time at this temperature. It is also imperative that this rheological study was specific to the 977-2A resin system. Figures 12 and 13 showed that the viscosity at the end of isothermal dwell of QS-60 cure was 1780 ± 180 Pa s and due to lower cure temperature the resin was not gelled, however, because of sufficient dwell time (60 min), the viscosity reached a value which lies between the critical viscosity range (1250-2050) Pa s. The corresponding viscosity at the end of isothermal dwell of QS-35 cure was (462 ± 102) Pa s, which was much lower than the critical range. As the main objective of intermediate dwell time was to allow time to squeeze out excess resin from the component, the effect of resin viscosity was assessed along with dwell time on the panel properties. Table VII and Figure 14 displayed a very high spread in the ultrasound attenuation for the QS-35-cured panel, which is also depicted in Tables VI and VII. The microstructures confirmed the poor quality of the panels cured, suggesting that poor squeeze out of excess resin occurred due to insufficient dwell time at 130°C. In an attempt to further remove the resin rich areas from the panel, the intermediate dwell time was increased from 35 to 45 min. Figure 12 depicts that the corresponding viscosity at the end of isothermal dwell of QS-45 cure was (667 ± 160) Pa s, which was slightly greater than that for the QS-35 cure, though, it was still well below the critical viscosity range. The improvement in the quality of QS-45 panel as compared to the QS-35 panel is evident from the C-scan image (Figure 14) and Table VII. There was low attenuation and very low spread in the attenuation of ultrasound for the QS-45-cured panel. Small patches of resin rich areas were observed on some parts of the panel. In all of the cure cycles, the GP occurred at 180°C, so this was possible to

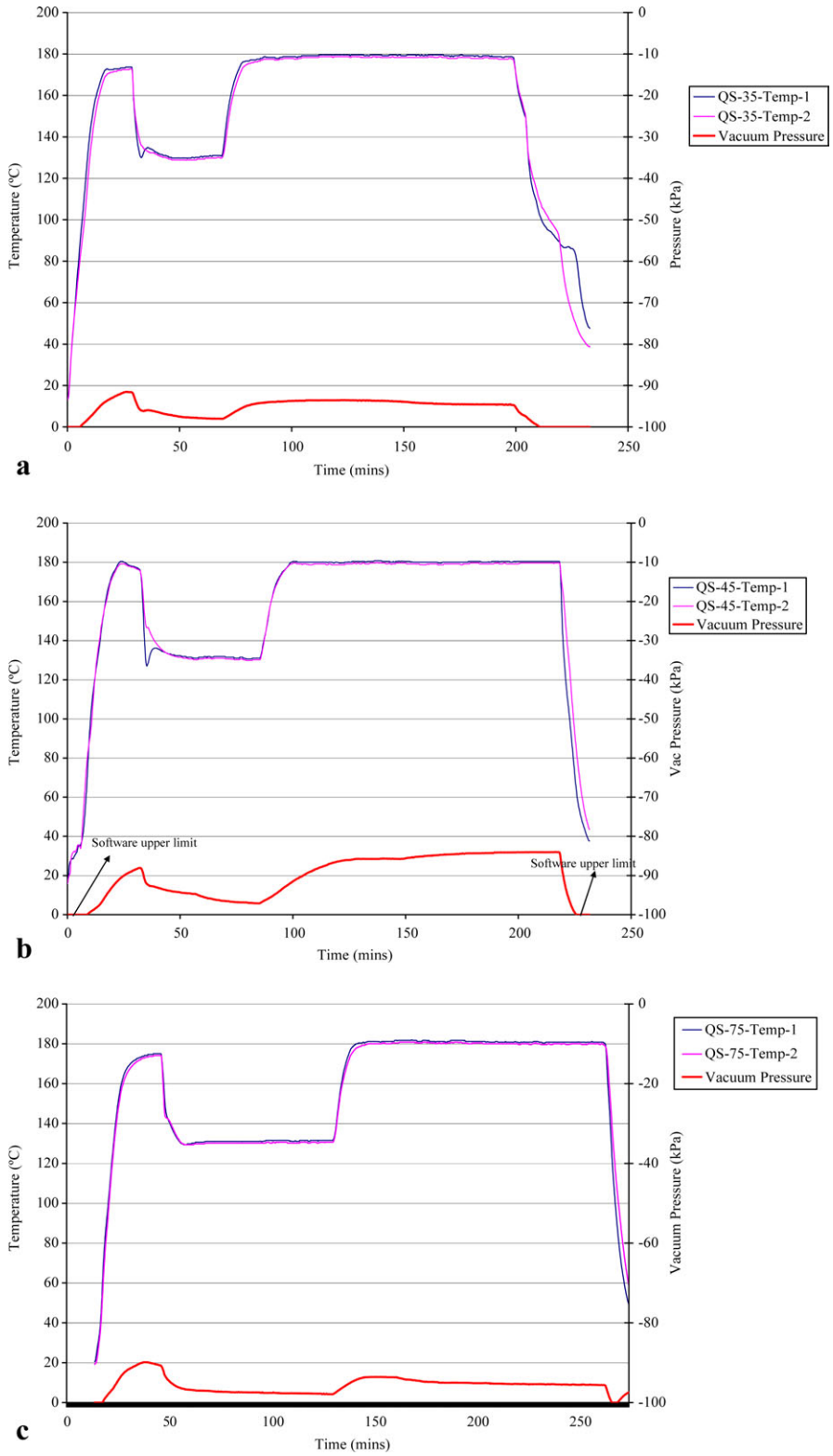

Figure 11. (a) Cure Cycle Profiles for QS-35. (b) Cure Cycle Profiles for QS-45. (c) Cure Cycle Profiles for QS-75. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

increase the intermediate dwell time further (75 min) in an attempt to improve the panel quality, especially to try to obtain a reduction in porosity. The viscosity profile obtained using the QS-75 cure cycle is shown in Figure 13. For ease of comparison, the viscosity profile for the QS-60 cure cycle is also provided. Figure 13 also shows that the corresponding viscosity at the end of

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Table VIII. Corresponding Viscosities at the GP for Various Curing Cycles

Ramp rate/Cure cycles	Corresponding viscosity at gel point (Pa s)
2 K min^{-1}	1497 ± 275
5 K min^{-1}	1555 ± 135
10 K min^{-1}	1632 ± 140
15 K min^{-1}	1492 ± 46
Autoclave/Oven	1733 ± 110
QS_{Direct}	1555 ± 135
QS_{ID}	1305 ± 97
QS-60	1939 ± 80
QS-35	1857 ± 132
QS-45	1385 ± 118
QS-75	1585 ± 187
Minimum	1250
Maximum	2050

isothermal dwell (75 min) was (3050 ± 240) Pa s which was far above the critical viscosity range (1250-2050) Pa s (Table IX). Figure 14 and Table VII revealed that higher ultrasound attenuation was observed for the QS-75-cured panel relative to the panel cured using the QS-60 cure schedule. The microstructures confirmed that the resin starved regions were formed due to excessive bleed of resin. Owing to resin starved areas, unacceptable physical and mechanical property variations were introduced in the panel (Tables V and VI). The QS-75-cured panels exhibited inferior mechanical properties to the QS-60 and autoclave-cured panels.

It has been stated that resin starved regions were less capable of transferring inter or intra-laminar shear forces, despite the QS-75 panel having similar void content to the QS-60-cured panel, the ILSS values were reduced by 15% as compared with QS-60-cured specimens. The resin starved regions also affected the fiber volume fraction and large standard deviations in the values were observed due to uneven fiber/resin distribution over the entire panel. This uneven fiber/resin distribution also influenced the flexural strength which was also reduced by 11%. The effect

Table IX. Comparison of Total Processing Time of Different Quickstep Cure Schedule with Autoclave Cure

Cure cycle	Total processing time (min)	Reduction in processing time against autoclave curing (%)
Autoclave	340	-
QS_{Direct}	246	28
QS_{ID}	255	25
QS_{AD} (QS-60)	249	27
QS-35	232	32
QS-45	236	31
QS-75	263	23

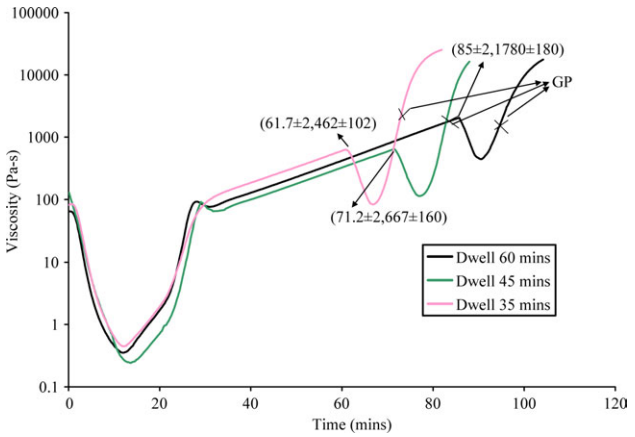

Figure 12. Viscosity profile for the QS-35, QS-45, and QS-60 cure cycles. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

was attributed to the fact that fibers were not held together and were not well supported caused by resin starved regions. The average panel thickness of QS-75-cured panel was pragmatic to be slightly lower than that of QS-60-cured panel which was again attributed to the excessive resin bleed.

CONCLUSIONS

Carbon/epoxy composite (aerospace grade prepreg) was processed using traditional oven/ autoclave and relatively new technique, Quickstep. A manufacturer's recommended cure cycle was employed for oven/autoclave processing. Because of the greater flexibility to control the cure cycle temperature (to manipulate the key resin viscosity) several cure cycles were employed for Quickstep processing.

Rheological Study

The optimum processing parameters for Quickstep processing was initially determined by proper fiber wetting when the resin was at minimum viscosity. The lower viscosity during the cure cycle accounted for lower void content by improving the

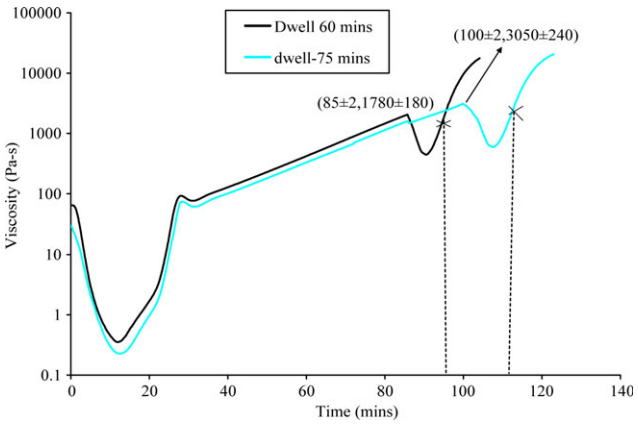

Figure 13. Viscosity Profile of the resin using the QS-60 and QS-75 Cure cycles. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

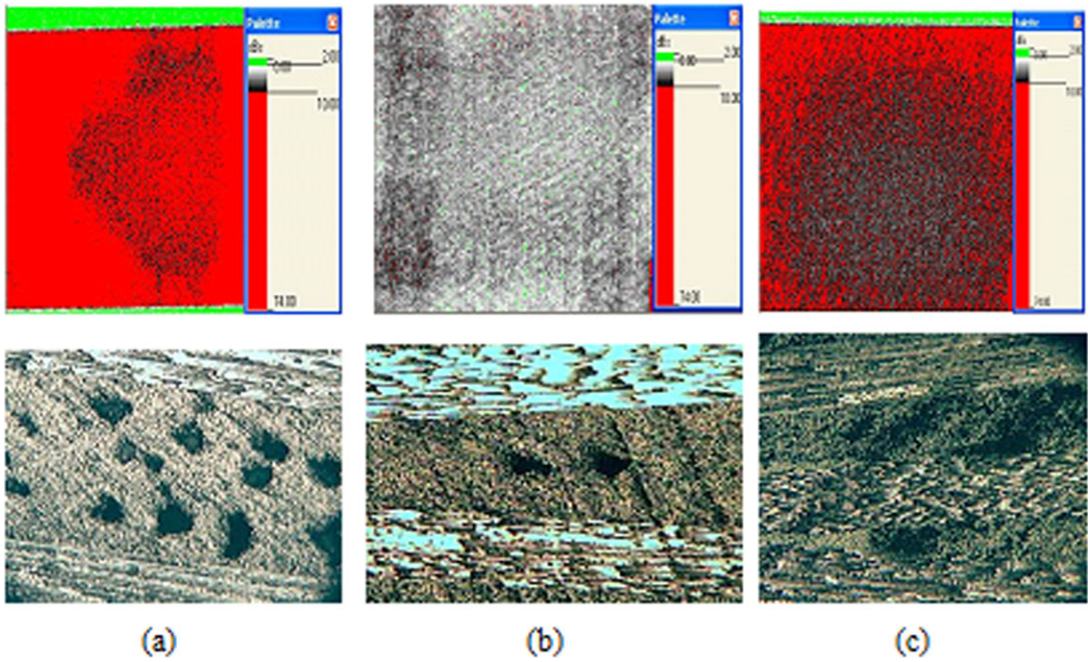

Figure 14. C- Scan images and associated micrographs of (a) QS-35, (b) QS-45, and (QS-75) cured panels. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

adhesion properties and fiber wetting. It was observed that the time required reaching the minimum viscosity of the resin decreased with increasing heating rates. The minimum viscosity was significantly reduced from 43 to 10 min, as the ramp rate increased from 2 to 15 K min^{-1}. A ramp rate of 15 K min^{-1} was achieved through Quickstep as compared to processing of composites in autoclave which usually worked on 2–3 K min^{-1}. Gel point, an important aspect of rheological measurements, was also determined by taking several simultaneous multi-wave frequency sweeps to measure tan δ in the gelation time-scale

Mechanical Strength Measurement of QS_{AD}-Cured Panel

The QS-60 (QS-60) cured panel showed a significant improvement in physical and mechanical properties compared with the other Quickstep-cured cycles. Flexural strength of the laminates made using the QS-60 cure cycle was sufficiently higher than the oven-cured panel (slightly lower than those made using the traditional autoclave route). Better flexural strength of autoclave panel was believed to be due to a higher fiber volume fraction achievable through application of high pressure. The ILSS values of QS-60 panels were found to be comparable to those made in the autoclave. The important factors responsible for fine interlaminar shear strength were low void content, better fiber/matrix adhesion, and lower viscosity during initial stages of cure, etc. Lower void content determines higher ILSS values of QS-60 panels. The improved fiber/matrix adhesion was possible due to improved mechanical interlocking of fiber with resin as a result of lower viscosity during initial stages of cure). Good fiber/matrix adhesion was also due to the improved wetting of fiber by resin as a result of lower viscosity. The lower viscosity during cure cycle also accounts for lower void contents, thus higher strength. Consequently, QS-60- and QS-45-cured specimens had comparable mechanical properties to the autoclave-cured specimens, relatively very low void content (less than 2) was observed for QS-60 cure schedule with improved ILSS values.

Ultrasonic C-Scan Imaging

A homogeneous distribution of fiber/resin was acquired via ultrasonic C-scans for the entire panel cured using QS_{AD} schedule. The microstructures confirmed the production of void free laminate using QS_{AD}. QS_{AD}-cured panel showed lower void content and fiber volume fraction as compared to $QS_{Direct-}$ and QS_{ID}-cured panels, however, slightly higher than autoclave-cured panel.

Intermediate Dwell Time Manipulation

Another important concept to determine optimum processing parameters for Quickstep processing was the suitable intermediate dwell time in order to allow the resin to flow and volatiles to escape of volatiles. The intermediate dwell time was manipulated in an attempt to further reduce the total processing time. It was observed that when the intermediate dwell time was reduced from 60 min to 35 min and 45 min, resin rich areas were formed as depicted from ultrasonic C-scan images and associated micrographs. The rheological analysis however, showed that sufficient resin flow with minimum void content is possible only with intermediate dwell time of 60 min.

Double Vacuum Bagging Lay-Up

In order to reduce the total cycle time of QS_{AD} (QS-60) cure schedule, an attempt was made to facilitate the excess resin to squeeze out during QS-35 and QS-45 cure. This attempt was based on the incorporation of double vacuum bagging (DVB) technique into Quickstep processing. A slight but not sufficient improvement in QS-DVB-35 panel was observed; however, it is believed that small resin rich patches of QS-45-cured panels can

be removed with controlled application of external pressure using DVB.

Total Processing Time of Quickstep Cure Cycles

Total processing time for all the Quickstep cure cycles was compared with autoclave curing. A 23-50% reduction in total processing time using Quickstep is possibly relative to autoclave cure. Without compromising panel quality, the overall cure cycle time was hence reduced considerably using the QS-60 cure schedule compared with equivalent panels fabricated using the autoclave process. The total processing time for autoclave/oven curing and QS_{AD} (QS-60) curing was 340 min and 248 min, respectively, thus without compromising panel quality, the overall cure cycle time was reduced by ~27% using Quickstep relative to autoclave.

On the basis of all data, the QS-60 cure schedule was considered to be the "Optimised cure cycle for Quickstep processing for 977-2A carbon/epoxy composites" based on the chosen laminate dimensions and processing conditions.

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