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# The effect of a rapid curing process on the surface finish of a carbon fibre epoxy composite

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## article info

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#### ABSTRACT

This work investigated the effect of a rapid curing process, Quickstep™, in conjunction with different fibre architectures (unidirectional and  $2 \times 2$  twill) and surfacing film (SynSkin®), on the surface finish of a carbon fibre G83C epoxy composite. Different magnitudes of pressure, heating and cooling rate were used to cure the composite laminates and the surface finish was studied using surface profilometry. It was found that the surface roughness was the most sensitive to the heating rate, which increased in roughness with high heating rates as a result of surface porosity entrapment. The high heating rates increased the rate of molecular cross-linking prior to gelation, which reduced the processing window and the removal of surface porosity via resin transport. The surfaces cured using fast heating rates were also found to have low fibre volume fraction and high resin content, which also supported the hypothesis that the resin flow is restricted prior to gelation.

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together and is separated by a flexible silicone bladder that is de-

## 1. Introduction

Aerospace, automotive and other transportation industries favour carbon fibre reinforced composite materials for their lightweight, high strength and high stiffness properties [\[1\].](#page-8-0) These factors translate into improved performance, fuel savings and reduced emissions [\[2\].](#page-8-0) However, their inclusion on high volume production vehicles is somewhat limited, due to the high production and processing costs associated with conventional methods (i.e. autoclave) of manufacturing composite components. Components cured using autoclaves have shown to exhibit exceptional quality with high fibre volume fraction, low porosity and good surface finish [\[3\],](#page-8-0) which allows them to meet strict aerospace standards. The down side of producing ''aerospace grade'' components is the high capital and operating expenses and, in particular, the long cure cycle durations and expensive tooling that must withstand the high pressures of the process [\[2\].](#page-8-0)

lises the high thermal conductivity rates of a heat transfer fluid (HTF) to rapidly cure composite components. The HTF is preheated prior to the commencement of a cure in three separate tanks at three temperatures (cold, medium and hot) with the temperature of the tanks pre-determined according to the cure cycle required for the resin system. At the commencement of a cure, the preheated HTF is pumped into a clam-like shaped tool with an upper and lower cavity. When the tooling is closed, each cavity is mated







signed to conform to relatively flat composite tooling and permits fast heat transfer rates from the fluid to the composite part. Typical heating rates achieved by the process are  $10 °C/min$  [\[4\],](#page-8-0) as a result of pre-heating the HTF prior to laminate cure and the high heat transfer capacity of the fluid. Due to the silicone bladder design, the pressures imparted on the laminate are generally restricted from 20 kPa to 60 kPa (plus the vacuum applied in the vacuum bag) for current Quickstep machine designs. The HTF provides cure cycle time reductions of up to 90% and an 82% reduction in tooling expenses associated with the low pressure curing in comparison to the autoclave process [\[5,6\].](#page-8-0) Previous studies have shown that the mechanical properties of Quickstep cured laminates are comparable with those manufactured with an autoclave [\[4,7,8\]](#page-8-0). However, it has also been shown that the surface finishes of Quickstep cured Toray G83C laminates are of higher roughness in comparison to autoclave cured [\[9\],](#page-8-0) which reduces their appeal to the automotive industry who strive for Class A surfaces, especially for exterior

Typical defects that cause the surface roughness to increase on carbon fibre composite laminates are porosity and dry fibre voids [\[10\].](#page-8-0) Moisture has been found to be the major factor contributing to void formation within a laminate [\[11\],](#page-8-0) as a result of the resin's tendency to absorb moisture from exposure to relative humidity and incorrect handling procedures prior to cure. During the curing process, moisture and, thus, porosity can become trapped in the laminate due to inadequate consolidation pressure [\[11\]](#page-8-0) and/or rapid gelation of the matrix [\[12\].](#page-8-0) Wenger et al. [\[13\]](#page-8-0) investigated the effect of autoclave pressure on the laminate surface roughness and

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<span id="page-1-0"></span>reported that as the pressure increased, the surface roughness and scatter of the results decreased. However, the direct effect of rapidly heating and cooling the matrix on the surface roughness has previously not been investigated due to equipment limitations and further understanding is required for the future use and development of rapid curing processes.

The objective of this work was to investigate the effect of processing parameters on the surface finish of cured composite laminates, which are typical of the low pressure, rapid curing Quickstep

#### Table 1

Level combinations for the laminate cure cycles.

process. This was accomplished by curing a series of laminates with different magnitudes of pressure, heating and cooling rates, and the tool-side laminate surface finish was evaluated using surface profilometry techniques. A sensitivity analysis was conducted to identify which parameters had the most effect on the surface roughness. To identify what caused the increase in surface roughness, the porosity and fibre volume fraction at the laminate surface was also evaluated together with the resin viscosity at various heating rates.





Fig. 1. Effect of pressure on the  $Ra$  (a),  $Rt$  (b),  $Rsk$  (c) and  $Rku$  (d) of the composite laminates.

## <span id="page-2-0"></span>2. Experimental details

#### 2.1. Laminate manufacture

The laminates in this study were manufactured using unidirectional (T600S 190 g/m $^2$ , laminate lay-up [0/90/0] $_{\rm S}$ , referred to as UD) and 2  $\times$  2 twill (12 k T700S 380 g/m<sup>2</sup>, laminate lay-up [0/90/  $0<sub>T</sub>$ , referred to as TW) carbon fibre pre-impregnated with G83C epoxy resin as supplied by TorayCA. An epoxy-based surfacing film, Henkel SynSkin® HC 9837.1 (150 g/m<sup>2</sup>, referred to as SYN), was cocured with the composite. SynSkin is used in the aerospace industry to minimise surface porosity and print through of core materials and to, therefore, reduce secondary surface preparation prior to painting that would be otherwise required. SynSkin HC 9837.1 contains silica (quartz and crystabolite) embedded in an epoxy resin film. The Syn-Skin technical data sheet [\[14\]](#page-8-0) specified for the resin-rich side of the film to be placed on the tool. However, to improve the laminate's surface quality using the Quickstep process, it was necessary to place the SynSkin film's fibrous side adjacent to the tool. The improved surface finish was accomplished by the fibres increasing the surface's breathability before gelation and, thus, allowing the vacuum to increase the efficiency of porosity removal.

The laminate stack was laid on Alanod Miro 4/4400 GP aluminium sheeting ( $Ra = 0.011 \mu m$ ,  $Rt = 0.12 \mu m$ ,  $Rsk = 0.09$ ,  $Rku = 3.45$ ). Following the completion of each cure the Miro sheeting was replaced with a new sheet to ensure processing conditions were consistent and resin build-up on the mould plate was not a factor influencing the surface finish. The Miro sheet was coated with a semi-permanent release and placed on a rigid tool. A perforated release film was placed on the edge of the laminate stack, with a solid release film on the centre. A peel ply and breather layer was placed over the lay-up and the vacuum bag was sealed with bagging film and sealant tape. To ensure the best possible surface finish was achieved, laminates were debulked for 15 h at 90 kPa in order to remove any trapped air within the laminate.

To investigate the effect of curing pressure, heating and cooling rate on the surface finish, laminates were cured with a Quickstep QS5 machine. Seven different cure cycles were used, as shown in [Table 1](#page-1-0), by changing the level of one parameter at a time to either the low or high setting while keeping the remaining parameters at the medium level. The pressure within the vacuum bag was held at 95 kPa during all cure cycles.

## 2.2. Surface roughness

The surface profiles of the cured laminates were measured using a Taylor–Hobson Form Talysurf Intra surface measurement instrument with a standard  $2 \mu m$  radius conisphere stylus. Profile



Fig. 2. Effect of heating rate on the  $Ra$  (a),  $Rt$  (b),  $Rsk$  (c) and  $Rku$  (d) of the composite laminates.

<span id="page-3-0"></span>

Fig. 3. Effect of cooling rate on the Ra (a), Rt (b), Rsk (c) and Rku (d) of the composite laminates.

data was analysed using the Taylor–Hobson Ultra software (V5.5.4.20). Instrument parameters were selected according to ISO 4288-1996, ISO 3274-1996 and ISO 4287-1997. A long evaluation length, l, of 25 mm was chosen to minimise the standard deviation of the measurements. Three samples were tested from each sample set, each with three random measurements taken at a  $45^\circ$  angle to the laminate's 0 $^\circ$  surface ply to obtain a representative surface roughness value. The raw profile was filtered with a cut-off of 0.25 mm and Gaussian filter, using a bandwidth ratio of 100:1. The roughness parameters that were measured in this study to analyse the profile's amplitude information were the arithmetic mean (Ra), maximum peak to valley height (Rt), skewness (Rsk) and kurtosis (Rku). A comprehensive description of the roughness parameters and how they are calculated has been provided in a previous publication by the authors [\[9\]](#page-8-0).

#### 2.3. Surface porosity

A method to measure the percentage surface area containing surface porosity was developed, so that a quantitative comparison can be made between laminates. The surfaces of laminates manufactured using the different processing levels were scanned using a Canon CanoScan 8400F at 300 dpi and analysed using ImageJ software (V.1.37s). Images were converted to 8-bit grey scale and the porosity was determined by using a threshold function to convert the image into black and white pixels only. The threshold function can be used to separate distinct physical characteristics within an image, as long as they have a contrasting colour when compared to the majority of the image. The grey scale histogram relating to the threshold information of the image shows a distinct separation between the matrix/fibres and porosity. This level selected was kept constant for all laminates analysed. The percentage of black pixels remaining in the analysis area is the percentage surface porosity of the laminate. This technique is similar to that used to determine fibre volume fraction via areal method [\[15\].](#page-8-0) It is a quick and easy procedure that can be used routinely to analyse the surface porosity of laminates. Although the ImageJ surface porosity analysis technique is limited to flat laminates, it can be used during process/cure optimisation and development or new materials and curing techniques.

#### 2.4. Fibre volume fraction

The fibre volume fraction was analysed for one unidirectional and twill panel from each of the cure cycles using optical microscopy. Three samples were taken across the centre of each laminate,

<span id="page-4-0"></span>

Fig. 4. Relative sensitivity of curing pressure, heating rate and cooling rate on the Ra (a), Rt (b), Rsk (c) and Rku (d) of the composite laminates.

placed in metallographic mounts and set in an epoxy casting resin. Once cured, samples were demounted, rough ground and finally fine polished using a Struers Force-5 and TegraPol-21 automatic polishing unit. Optical images of the laminate's surface morphology were taken at 1000 $\times$  magnification, making sure that minimal casting resin was in the image to maximise the analysis of the surface. The image was cropped to remove the casting resin from the analysis and the resultant image was analysed using a similar procedure as described in the previous section using the ImageJ software. The threshold level used to convert the image into black and white pixels was selected with the guidance of the grey scale histogram, which clearly showed the separation between the matrix and fibres pixel colour within the image. Micrographs of the twill surfaces were taken in the middle of the carbon fibre tow bundles, so that the effects of the resin rich regions at adjoining warp and weft tows would not influence the results.

## 3. Results and discussion

## 3.1. Surface roughness

The surface roughness results of the laminates cured using different levels of pressure are shown in [Fig. 1.](#page-1-0) It can be seen that the amount of pressure applied during the cure cycle has some affect on the laminate surface roughness. As the curing pressure is reduced, it can be seen that the Ra and Rt is increased. The reduction in curing pressure has also reduced the negative skewness and kurtosis of the roughness profile. High  $R^2$  correlation has been shown for the linear fit between the unidirectional laminate roughness results for all the roughness parameters measured. A high  $R^2$  correlation indicates that the linear fit of the line to data is a true representation, where an  $R^2$  of 1.00 represents a perfect fit. Apart from the pressure affects on the surface roughness, the twill laminates are affected by the resin rich regions at the carbon fibre tow intersections, where surface porosity tends to become trapped, which may affect the clarity in results. This is also supported by the increased roughness value for the twill and SynSkin laminates.

The effect of heating rate on the surface roughness results is shown in [Fig. 2](#page-2-0). A clear increase in roughness is shown for almost all roughness parameters with increasing heating rates. The unidirectional laminates maintain low roughness regardless of the heating rate used. A clear distinction between the unidirectional



Fig. 5. Effects of pressure and heating rate on the twill laminate surface porosity. Error bars represent one standard deviation.

<span id="page-5-0"></span>

Fig. 6. Grey scale images of the laminate surfaces showing the differences in surface porosity with the twill laminates cured using 5.2 °C/min (0.3% surface porosity) and 14.4 °C/min (1.3% surface porosity) heating rates.



Fig. 7. The viscosity profile of the neat G83C resin as a function of time and heating rate. Data received from Quickstep Technologies Pty Ltd [\[17\]](#page-8-0).

and twill laminates with SynSkin was not as obvious with increasing heating rate as previously shown with the curing pressure effects charts in [Fig. 1](#page-1-0). The increase in negative Rsk with the faster heating rates for all laminates suggests an increase in trapped porosity on the tool face. This is also reflected in the Rku results, with the high spikiness of the surface profile with faster heating rates relating to the increase in porosity. The twill and SynSkin laminates also showed a dramatic increase in roughness with increasing heating rate. The high linear correlation ( $R^2$ ) for the twill and SynSkin results also supports that the data fits the linear model indicated in [Fig. 2](#page-2-0).



Fig. 8. The effect of heating rate on the minimum resin viscosity and processing window. Data manipulated from Quickstep Technologies Pty Ltd [\[17\]](#page-8-0).

The effect of the cooling rate on the laminate surface roughness is shown in [Fig. 3](#page-3-0). It can be seen that there are no significant changes in roughness as the cooling rate increases for the unidirectional laminates. However, there is an increase in Rsk and Rku for the twill laminate combinations with the faster cooling rate.

As shown in [Figs. 1–3](#page-1-0), the twill laminates had the highest surface roughness when compared to the roughness of the unidirectional laminates. Also, the additional layer of SynSkin surfacing film increased the surface roughness when combined with both the unidirectional and twill fabric.

#### <span id="page-6-0"></span>3.2. Sensitivity analysis

The results presented in [Figs. 1–3](#page-1-0) have indicated general surface roughness trends with respect to the pressure, heating and cooling rates used to cure laminates. However, a sensitivity analysis was conducted to better represent the results so that direct comparisons can be made between the manufacturing parameters. A sensitivity analysis can provide quantitative and comparable changes in the output from different input sources. The relative sensitivity, S, was calculated by

$$
\left| S_A^F \right| = \frac{\partial F/F}{\partial A_n / A_n} \Big|_{n}
$$
\n<sup>(1)</sup>

where S is the ratio of the percentage change of the input ( $\partial F/F$ ), over the percentage change of the output ( $\partial A_n/A_n$ ). The percentage change of the input was calculated as the change in magnitude for each of the processing parameter values from low to high level. The percentage change in the output was calculated by the application of a line of best fit to each data series first and then calculated the difference in the roughness value using the line of best fit equation at the low and high input values for each of the process parameters. The relative sensitivity calculations assume a linear relationship for the data in the effect plots. Therefore, to evaluate how well the data fits to a linear trend in the effect plots, the  $R^2$  was calculated.  $R^2$  values range from 0.0 to 1.00, where 0.0 represents no linear model fits the data and 1.0 represents a perfect linear trend.  $R^2$  is calculated by

$$
R^2 = \frac{(SST - SSE)}{SST} \tag{2}
$$

where SST is equal to the sum of squares total and SSE is the sum of squared errors [\[16\].](#page-8-0)

The relative sensitivity compares how a 1% change in the input will affect the output. For example, as shown in [Fig. 4\(](#page-4-0)a), a 1% change in heating rate for the twill laminate with SynSkin resulted in a 2.5% change in Ra. As seen in [Fig. 4](#page-4-0), the surface roughness of the laminates was most sensitive to the heating rate parameter during the curing process. The surface roughness of the SynSkin laminates also appeared to have the highest sensitivity to the heating rate. It is interesting to note that the Rt and Rsk parameters have the highest sensitivity to changes in the surface roughness (maximum of 26% and  $-17%$  respectively) in comparison to the Ra (2.5%) and Rku (4.6%) results. The twill laminate showed the highest sensitivity to the heating rate for the laminates without a surfacing film. Again, the sensitivity of the twill laminate is possibly due to the surface porosity that could become entrapped between the weft and warp fibre tow intersections. The results of the relative sensitivity analysis suggest that with increasing heating rate, the average roughness increases, with the frequency and depth of the surface valleys increasing also. Pressure did have a slight affect on the surface roughness. However, the effect of the cooling rate on the laminate surface roughness appeared to be insignificant when compared to the effect of the heating rate.

#### 3.3. Effect of pressure and heating rate on surface porosity

The results discussed thus far have indicated that the change in roughness is due to the amount of surface porosity at the surface. Therefore, the percentage surface area of porosity on the laminates surface was measured to identify if the surface porosity was influenced by the curing parameters. Significant contrast between surface porosity and the laminate surface could only be identified with the twill laminates using the technique described in Section [2.3](#page-3-0). Porosity on the unidirectional laminate surfaces were significantly smaller in size than those on the twill laminates, which could not be detected with this technique. As the cooling rate

was found to have an insignificant affect on the surface finish, it has been disregarded from the remainder of this study.

It is shown in [Fig. 5](#page-4-0) that both the pressure and heating rate had a linear effect on the surface porosity. Although not identified in the surface roughness results, the 15.2 kPa change in curing pressure between the high and low levels did have an effect on the surface porosity. The percentage surface porosity of laminates cured using 3.2 kPa and 18.4 kPa, showed a decrease in surface porosity from 0.98% to 0.53%, a drop of 47%. However, a greater reduction in surface porosity was observed with the slower heating rates. The difference in the percentage surface porosity of laminates cured using  $14.4 \text{ }^{\circ}$ C/min and  $5.2 \text{ }^{\circ}$ C/min heating rates showed a 73% decrease in surface porosity from 1.28% to 0.35%. An example of the scanned laminate surfaces manufactured using the two extreme heating rates and the resultant surface porosity maps are shown in [Fig. 6.](#page-5-0) A clear differentiation can be seen between the two porosity maps, with large cylindrical shaped volatiles occurring at the tow intersections. Subsequent to these observations, a series of samples were manufactured using a combination of high curing pressure (19 kPa) and slow heating rates (3  $°C/min$ ) and it was found that the surface porosity was significantly reduced to 0.2%. Although a considerable improvement in the surface finish was observed, not all of the porosity could be eliminated from the surface suggesting that slower heating rates than  $3^{\circ}C/m$ in are required to remove all of the porosity. However, this was found to be the minimum achievable heating rate with the Quickstep, as the process was primarily designed for rapid heating and curing.

#### 3.4. Effect of heating rate on resin viscosity

To understand how the heating rate affected the removal of porosity at the surface during cure, the resin viscosity as a function of the heating rate was investigated. The rheology data presented in [Fig. 7](#page-5-0) was obtained and used with permission from Quickstep Technologies Pty Ltd [\[17\]](#page-8-0). [Fig. 7](#page-5-0) showed that as the heating rate increased, the minimum resin viscosity decreased during cure. In addition, as a result of the rapid cross-linking associated with the increased heating rate, it was also shown in [Fig. 7](#page-5-0) that the length of time in which the resin was maintained at a low viscosity prior to gelation was significantly reduced. The effect of heating rate on the minimum resin viscosity and the length of time the resin viscosity was below  $10^2$  P, labelled as the processing window is shown in [Fig. 8.](#page-5-0) Comparing the length of time the resin is below  $10^2$  P when heated at rates 2.8 °C/min and 11.1 °C/min, the



Fig. 9. Fibre volume fraction results as a function of the heating rate for the unidirectional and twill laminate surfaces. Error bars represent one standard deviation.



Fig. 10. Optical micrographs of the unidirectional (top) and twill (bottom) laminate surfaces, with and without a SynSkin layer, cured with 5.2 °C/min (left) and 14.4 °C/min (right) heating rates.

processing window is reduced from 9.5 min to 4.6 min, respectively. Further, it is shown in [Fig. 8](#page-5-0) that when a heating rate of 22.2  $\degree$ C/min is used, the processing window is reduced further to only 2.9 min. The reduction in the resin processing window with fast heating rates suggested that insufficient time is given for the porosity to be removed from the laminate surface by the flow of resin. The low curing pressure used by the Quickstep process implies that the primary method of volatile removal is via resin transport. Therefore, maximising the pre-gelation window is vital in producing a surface with low roughness. Laminate surfaces manufactured using slow heating rates experienced a longer duration at minimal viscosity, allowing sufficient time for the resin to flow and conform to the tool surface. The slow heating rates also decreased the quantity of surface volatiles as they are able to diffuse prior to gelation and, thereby, further improve the laminate surface finish.

#### 3.5. Effect of heating rate on the fibre volume fraction at the surface

It was shown that composite laminates cured with high heating rates reduced the processing window and resulted in the entrapment of porosity at the surface and, therefore, increased roughness. To assess if the entrapment of porosity was due to reduced resin flow at the surface, the fibre volume fraction at the laminate surface was assessed. As shown in [Figs. 9 and 10,](#page-6-0) a reduction in the fibre volume fraction at the surface was observed with increasing heating rates for the unidirectional laminates. The fibre volume fraction was reduced from 55.0% to 50.1% when the heating rate was increased from 5.2  $\degree$ C/min to 17  $\degree$ C/min. A linear correlation between the heating rate and surface fibre volume fraction was also shown, with an  $R^2$  of 0.97. Furthermore, a similar effect was shown for the twill laminates in [Figs. 9 and 10.](#page-6-0) The fibre volume

<span id="page-8-0"></span>fraction reduced more noticeably with the twill laminates from 61.3% to 47.5% when the heating rate was increased from 5.2  $°C/$ min to 17 °C/min. Again, a high linear correlation was observed for the data, with an  $R^2$  of 0.85. However, large variation in results was observed for the fibre volume fraction of the twill laminate surfaces as opposed to the unidirectional, possibly caused by the inconsistent surface morphology due to the carbon fibre tows. The high fibre volume content at the surface manufactured with low heating rates has indicated that the laminate has more time to consolidate prior to gelation and, therefore, an improvement in the surface finish can be seen. Curing with high heating rates "locks-in" the resin and porosity within the laminate, as shown by the high resin and porosity content.

Further inspection of the SynSkin layer found that the surfacing film contained large, irregular shaped silica particles within the matrix. The silica particles could be obscuring the resin flow at the surface that has already been restricted with the fast heating rates and reduced pre-gelation window and, thus, resulting in a higher surface roughness and relative sensitivity on the SynSkin co-cured laminates.

#### 4. Conclusions

This work has investigated the effects of using a rapid composite manufacturing process, Quickstep, to cure G83C epoxy carbon fibre composite laminates with respect to the laminates surface finish. The high heating rates typical of the Quickstep process ( $>10$  °C/min) were found to increase the surface roughness of the laminates. In addition, it was shown that the laminate surface roughness was most sensitive to changes in the heating rate, rather than the applied pressure or cooling rate during the cure cycle. The rough surface resulting from fast heating rates was further emphasised when SynSkin surfacing film was used on both unidirectional and twill laminates, possibly due to the large, irregular silica particles contained within the film. Interestingly, Rt, Rsk and Rku parameters had a higher sensitivity to the heating rate and pressure than Ra, which suggests the importance of considering roughness parameters other than Ra in surface roughness studies.

Analysis of the surface porosity on the twill laminates revealed that the surface porosity was directly related to the pressure and heating rate and as a result, caused the increase in the surface roughness. Laminates cured with fast heating rates were shown to have a higher quantity of surface porosity. Analysis of the G83C resin viscosity profile showed that as the heating rate increased, the rate of molecular cross-linking occurred at a faster rate which decreased the processing window and restricted the resin flow within the laminate prior to cure. The reduced resin flow at the surface was also observed with the optical cross-sections of the laminate which showed a resin-rich surface. Therefore, it is essential that the pre-gelation window of the resin is maximised in order to produce a surface with low surface roughness and minimal porosity. The maximum curing pressure that is obtainable by the process should also be utilised when curing laminates to further improve the surface finish. As the cooling rate was found to have minimal impact on the surface finish, rapid cooling rates can be used to assist with the reduction of cure cycle durations without compromising the surface.

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