

OUT OF AUTOCLAVE MANUFACTURE OF STRUCTURAL AEROSPACE COMPOSITE MATERIALS

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SUMMARY

This paper presents an investigation of the mechanical and physical-chemical properties of an epoxy/carbon fibre prepreg (Hexply M18-1-43%-G939) cured using a new out-of-autoclave low pressure manufacturing method called Quickstep™.

The purpose of the investigation was to explore the effect of the new curing technique on the carbon fibre prepreg material properties and the process potential to reduce the manufacturing costs is envisaged.

The cure cycle time was reduced by 43% compared to an equivalent autoclave process, due to the rapid heat-up and cooling. Test results indicated that the degree of cure, glass transition temperature and ultimate tensile strength were in the range of the autoclave values. Due to higher heating rates with the new process, the initial resin viscosity is lower, resulting in an improved fibre-matrix adhesion and a higher delamination resistance in sandwich panels. Consequently an improved interlaminar shear strength for monolithic and peel torque resistance for sandwich parts were attained.

1. INTRODUCTION

High performance composite materials for aerospace applications are conventionally processed in autoclaves, heating ovens or presses. In ovens and autoclaves the heat is transferred into the part via air or inert gas. The low thermal conductivity of a gas limits the heating rates, which often results in long cure cycle times. Therefore the aerospace industry is investigating in low-cost, rapid and energy efficient solutions for production [1].

A new curing technology called Quickstep™ is based on a fluid as the heat transfer medium. Since fluids provide a much higher thermal conductivity compared to gases, it is possible to increase the heating and cooling rates. Hence, shorter cycle times can be achieved while maintaining the same dwell and curing times as in an autoclave process [2].

Furthermore, the higher heat transfer of the system reduces the risk of exothermic reactions occurring while curing. The speed of the heating of the composite lay-up influences the initial viscosity of the matrix and therefore the quality of the composite part [3]. Properties that are expected to change are degree of cure, fibre-matrix adhesion [4] and fillet forming for sandwich structures due to a lower initial resin

viscosity. These properties have been investigated in this study. With the new curing process, the utilisation of standard materials such as vacuum bagging, prepreg or even infusion technologies can be maintained and no major changes concerning the design of parts have to be done.

For the purpose of the investigation carbon fibre reinforced monolithic and sandwich panels, manufactured with the Quickstep™ technology, have been compared with panels produced in a conventional autoclave. The research focused on the influence of the processing method on the mechanical and physical-chemical properties. The honeycomb sandwich panels were tested on their skin-to-core adhesion. For monolithic panels, tensile strength and interlaminar shear strength were tested. The degree of cure and the glass transition temperature were also analysed to determine the effect on the chemical properties.

2. MATERIALS AND EXPERIMENTAL DETAILS

The monolithic panels, used for interlaminar shear strength (ILSS) and tensile tests, and the facings for the sandwich test panels are made from HEXCEL epoxy carbon fibre prepreg material HexPly M18/1 with a resin content of 43% and a bidirectional [0°/90°], 4 harness satin weave fabric G939. This material is used for structural components in the helicopter industry and has a service temperature ranging from -55°C to 135°C.

The monolithic panels consisted of 8 ply [0°/90°] prepreg material with dimensions of approximately 600 mm x 300 mm. As an un-perforated release film was used for the Quickstep™ plates, no resin bleeding was possible, leading to a thicker plate. Therefore the thicknesses of the monolithic plates were approximately 1.92 mm (autoclave) and 2.10 mm (Quickstep™). In Figure 1 conventional lay-up (left) with standard vacuum bagging (right) can be seen, being placed in the Quickstep™ pressure chamber.

The sandwich panels were laminated with facings of two plies [0°/90°] and a 15 mm Nomex-honeycomb core, with a cell size of 3.2 mm and a density of 48 kg/m³, without any adhesive film in between.

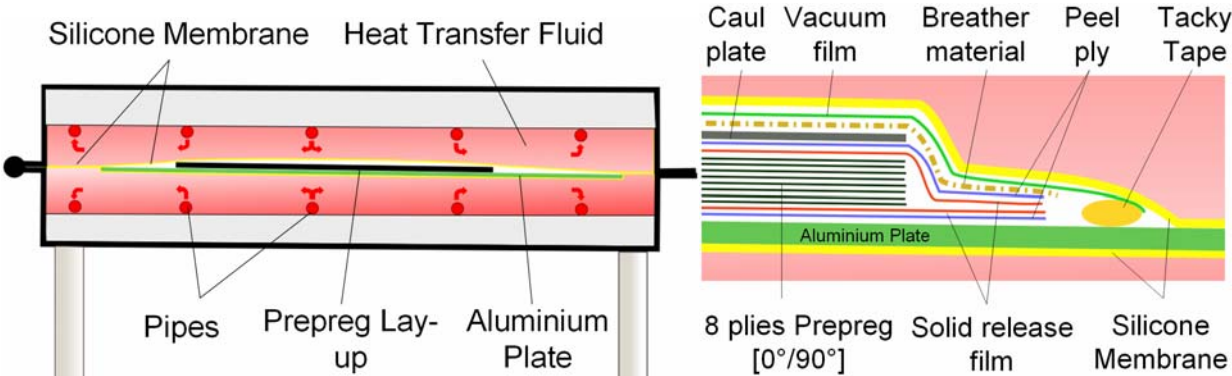


Figure 1: Drawing of the pressure Chamber (left) and schematic Lay-up and vacuum bagging diagram (right)

The laminates were cured in the autoclave applying a cure cycle with a first dwell temperature of 80°C for 1 hour and a second dwell temperature of 180°C for 2 hours. The differential pressure for autoclave parts was higher; approximately 2.5 bar for sandwich and 5.5 bar for monolithic panels, instead of 1.1 bar for both cases with the low pressure process. Due to the lower initial resin viscosity with the Quickstep™ process the pressure was found to be sufficient to consolidate the laminate.

The schematic cure cycle, vacuum and pressure development for the monolithic panels under both processes are shown in Figure 2. The first and second dwell times were 1 hour and 2 hours respectively. A reduction in time of 175 minutes was achieved by applying higher cooling and heating rates (of approximately 6-9 °C/min instead of 1.5 °C/min with the autoclave). This results in a time saving of 43% in comparison to the autoclave process.

The chemical properties, such as glass transition temperature (T_g) and the degree of cure, were determined using TA Instruments Q1000 Modulated Differential Scanning Calorimetry (MDSC). The specimens were heated at a constant rate of 10°C/min.

The interlaminar shear strength was determined according to the standard DIN EN 2563. For statistic reasons the results are quoted as the mean of five samples for each panel.

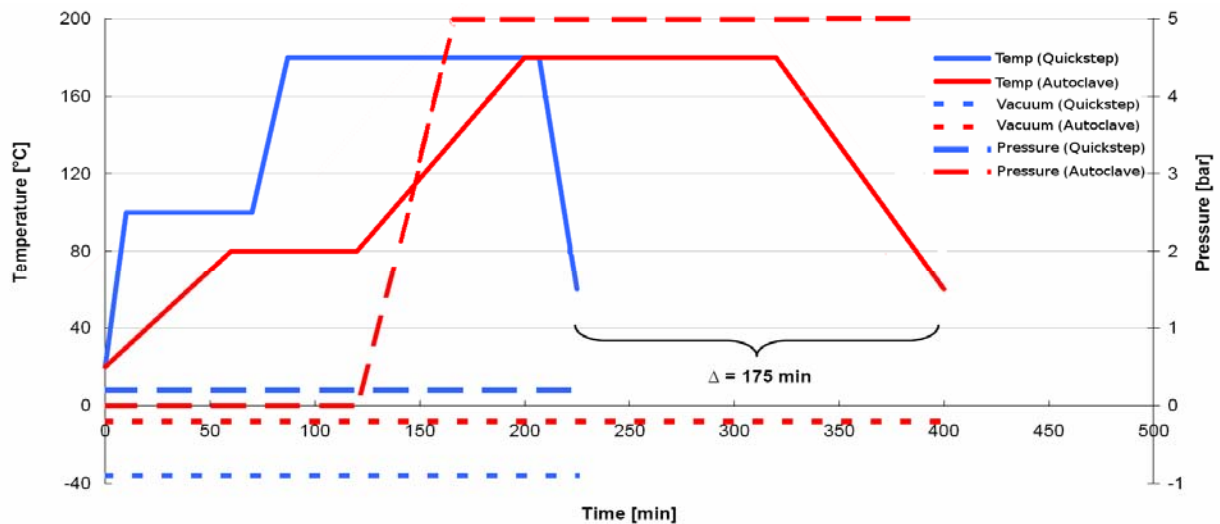


Figure 2: Cure cycle for monolithic panels Autoclave - Quickstep™

Tensile tests were performed on a Schenck-Trebel testing machine with a speed of 2 mm/min, according to the standard EN2561. Within each cure method five specimens measuring 10mm x 250 mm were produced and tested. Glass fibre epoxy tabs [+/-45°] were bonded with 3M adhesive film AF 3109-2 onto the specimen, which resulted in a free testing length of 120mm. Strain gages were attached to record the strains and to calculate the elongation in testing direction. Although the same number of plies were used, a variation in specimen thickness was observed between the autoclave and Quickstep™ panels ($t_{AC}=1.92\text{mm}$ and $t_{QST}=2.10\text{mm}$). For this reason, the ultimate tensile strength related to the fibre cross-sectional area was calculated according to the EN3783 standard.

The climbing drum peel test was performed on a Zwick 1475 testing machine according to the standard DIN 53295, a test method for determining the skin-to-core bond strength. The specimen size was 75mm x 300mm. The face sheets were peeled from the core by being rolled onto a 100 mm radius cylinder with a cross head speed of 25 mm/min.

3. Results

3.1. Glass transition temperature and degree of cure of monolithic panels

The monolithic panels manufactured in the autoclave and Quickstep™ processes provided almost similar values for the degree of cure and the glass transition temperature (T_g). The results of the DSC tests are presented in Table 1. The values for the degree of cure for both processes, as well as the T_g for the autoclave panels, are based on the average of four specimens. The Quickstep™ average T_g was calculated from two measurements.

Despite having being exposed for 175 more minutes the degree of cure of the autoclave samples is just 0.5% higher than under the shorter Quickstep™ curing process. Moreover, the T_g of 212.5°C for the autoclave specimens is slightly lower compared to the 213.5°C obtained from the quicker cured panels. Based on these results, cure cycle time reduction achieved with Quickstep™ has no major negative affect on the physical-chemical properties of the resin matrix.

	Autoclave	Quickstep™
Degree of cure	94,0%	93,5%
Glass Transition Temperature	212,5 °C	213,5 °C
Total Cycle time	~ 400 min	~ 240 min

Table 1: Degree of cure and glass transition temperature

3.2. Monolithic Plates - Tensile and ILS test

Tensile Test:

The ultimate tensile strength was calculated as a function of the fibre cross-sectional area. The mean ultimate tensile strength resulting from each process is presented in Figure 3. The obtained average tensile strength for the autoclave-processed specimens is 832.4 MPa with a standard deviation of ± 35.5 MPa. The Quickstep™ specimens were in the range of the autoclave values and have an average of 822.2 MPa with a slightly lower standard deviation of ± 32.4 MPa.

The differences in failure/fracture mode surfaces of the specimens can be observed clearly in Figure 4. The autoclave specimen fibres were pulled out of the laminate and create a brush-like cross section with fraying fibres. This indicates a more brittle failure behaviour compared to the Quickstep™ specimens, which show a more uniform failure along the cross section. The different mode of failure is caused by shear lag. This effect is introduced by some initial fibre breakages occurring just before the whole specimen fails. Between these fibres shear tension peaks arise leading to frayed out fibre failure if the matrix can not withstand the shear forces. Therefore the mode of failure represents either a tougher matrix or an enhanced fibre to matrix adhesion for the Quickstep™ specimens, which is most likely caused by the lower initial viscosity of the resin. This improvement leads to an increasing capability to resist shear forces.

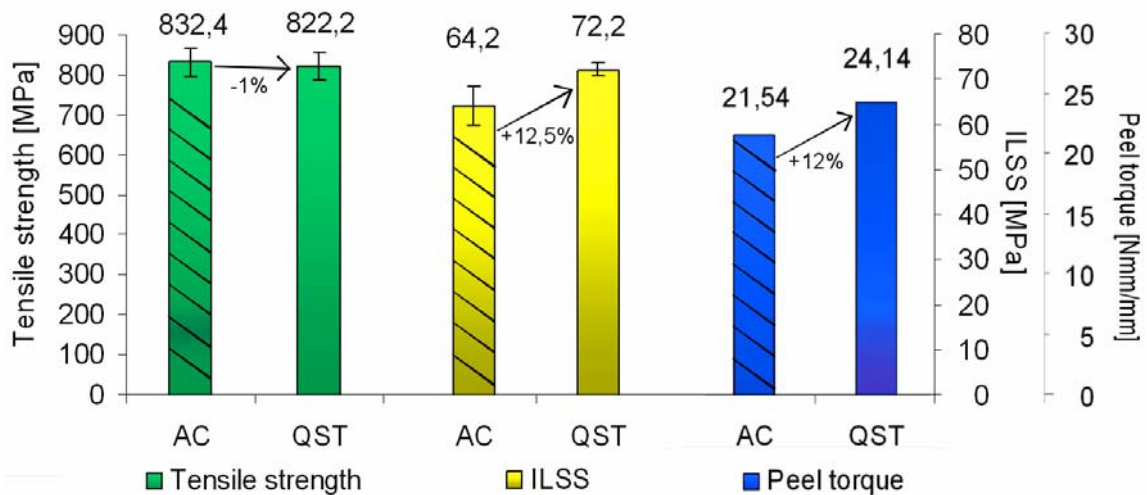


Figure 3: Results of Tensile, ILS and Climbing drum peel test - Autoclave (AC) and Quickstep™ (QST)

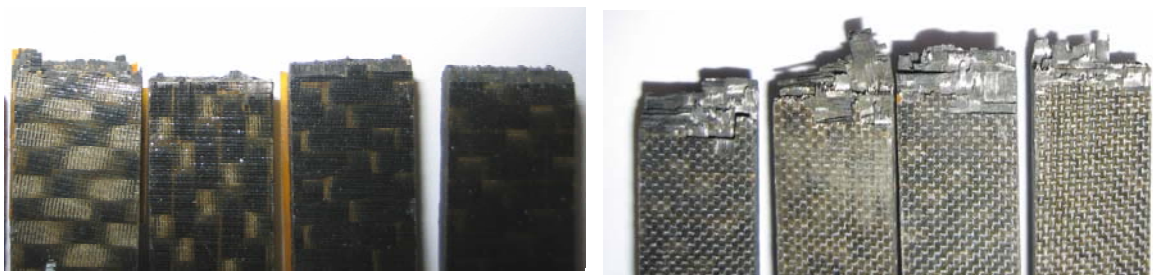


Figure 4: Failure surfaces of Quickstep™ (left) and more brittle autoclave (right) tensile test specimens

ILS test:

The interlaminar shear test (ILS) was chosen to estimate the strength of the fibre-matrix adhesion. The specimens must fail in shear, approximately at the neutral fibre axis and not by flexure or by plastic deformation. The interlaminar shear strength (ILSS) values of the Quickstep™ specimens are on average 12.5 % higher (72.2 MPa) than the autoclave results (64.2 MPa), as illustrated in Figure 3. The standard deviation is also lower, ± 1.5 MPa for the Quickstep ILSS average values compared to ± 4.2 MPa for the conventional process. This shows that the fibre-matrix adhesion increases with the faster curing process. Additionally the repeatability was improved, as indicated by the lower standard deviation.

3.3 Sandwich plates - Climbing drum peel test

The strength of the face-to-core adhesion, tested using the climbing drum peel test, increased with the faster curing process. The fillet forming of the resin between the core and the face-sheets improved due to lower resin viscosity (see Figure 3). This phenomenon resulted in a better adhesion and a higher delamination resistance of approximately 12% (from 21.5 Nmm/mm with the autoclave to 24.1 Nmm/mm with Quickstep™). Also the standard deviation is lower, ± 5.1 Nmm/mm compared to ± 8.7 Nmm/mm with the autoclave. With the utilization of self-adhesive prepreg systems,

weight and cost reduction could be achieved through the omission of adhesive film due to the better face-to-core adhesion with Quickstep™. In Figure 5 the improved face-to-core adhesion for the Quickstep™ specimens are demonstrated. Also 4-point bending tests were conducted with sandwich panels yielding to results which are in the range of the autoclave specimens.

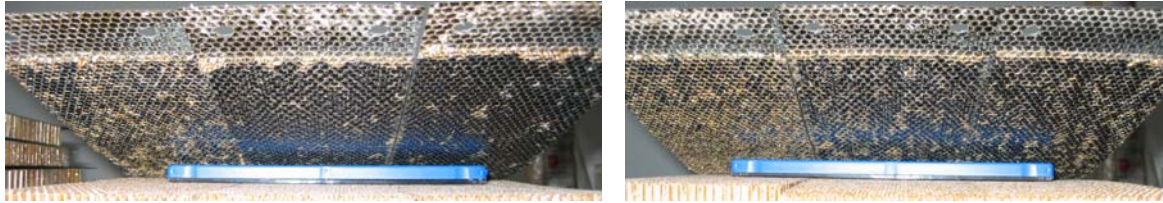


Figure 5: Climbing-drum-peel specimens - Autoclave (left), Quickstep (right)

4. CONCLUSION

The tensile strength, interlaminar shear strength, degree of cure and glass transition temperature of carbon epoxy (HexPly M18/1-43%-G939) monolithic composite and the peel torque resistance of a Nomex-honeycomb sandwich plate were investigated. The results of the physical properties of the out-of-autoclave Quickstep™ tests were in the range or superior to the autoclave ones. Despite the shorter total curing time, the glass transition temperature and the degree of cure had the same values as the autoclave specimens. With Quickstep™, faster heating (and cooling rates) are possible, which lowers the initial viscosity of the resin. This mechanism is believed to improve the fibre wetting and the fibre-matrix adhesion, as observed in the ILS test results and the failure surfaces of the tensile test specimens. The lower viscosity leads also to a better fillet forming between the face sheet and the honeycomb core, resulting in a higher peel torque resistance for sandwich panels. With the new process, a time saving of 43% compared to the conventional autoclave cure cycle was realised. The Quickstep™ process has the potential to reduce the cycle time and therefore the cost for manufacturing parts.

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