THE EFFECT OF PROCESSING PARAMETERS ON THE INTERLAMINAR PROPERTIES OF AUTOCLAVE CURED GLASS FIBRE/EPOXY COMPOSITES

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1 Introduction

Nowadays, polymer composites are becoming more and more common. In areas with the highest quality requirements, composite components made of prepreg are the most frequently used. Prepregs can be used to produce highly reproducible, high-performance composites, but this requires a tightly controlled processing environment. The process parameters have a significant influence on the structure and properties of the manufactured composite [1]. The heating rate and the isothermal phase, dwell, included in the thermal cycle have a significant influence on the behaviour of the resin during the curing process, they determine the viscosity drop of the matrix and the progress of the crosslinking reaction [2]. The level of pressure applied during the cure cycle might also have a significant effect e.g. on the extent of resin leakage, thus the fibre content of the composite and the amount and size of voids in the product [3]. Due to the structural differences, the mechanical - especially interlaminar - properties of composites produced with different parameters can also vary considerably.

2 Experimental

2.1 Concept

The main research question is, what the often inevitable deviations from the parameters specified by the technical data sheet and the alternative cycles allowed in some cases by the prepreg manufacturer will result in. In this work, the effects of the heating rate, the amount of pressure applied to the composite during the cycle and the use of a temperature plateau have been investigated in the case of autoclave production of glass fibre/epoxy composite plates. In order to specify the influence of the application and the temperature of the isothermal dwell, the viscosity of the resin was monitored during different thermal cycles. Furthermore, the effects of overpressure, the plateau used and the heating rate on the structure and properties of the composite were determined by investigating the interlaminar properties of plates manufactured with different parameters.

2.2 Materials and sample production

For the research, Hexcel's UD S-glass/913 epoxy prepreg was used. The samples for the viscosity measurement were layered from 35 µm thick 913 epoxy resin film. Composite plates with 3 mm thickness were cured in Olmar ATC 1100/2000 autoclave using four different cure cycles. The first was the general cycle recommended by the prepreg manufacturer with 2°C/min heating rate, 125°C cure temperature, 60 min cure time and 7 bar pressure, vacuum was used until the pressure in the autoclave exceeded 1 bar. Each of the other cycles differed in one parameter. The second was conducted with only 1 bar pressure, the third with only 0,5°C/min heating rate and the fourth with a 25 min long dwell at 90°C.

2.3 Test methods

The viscosity of the resin during different thermal cycles was measured by an oscillatory rheometer (TA AR2000) with 10 rad/s angular frequency and 2% controlled strain. The fiber content of each plate was measured by thermogravimetry using a TGA Q500 device (TA Instruments). Theoretic bulk density was calculated from the results. The real density was measured based on the ASTM D792 standard by immersion of samples in distilled water. The real density allowed the conversion of the results of TGA measurements to fiber volume fraction. From the

theoretic and the real density, porosity was calculated according to ASTM D2374 standard.
Short-beam shear tests were conducted with ISO 14130 type test specimens in order to determine the interlaminar shear strength of the composites manufactured with different cure cycles.
After that the fracture toughness $G_{II,C}$ for mode II failure was investigated with uniaxial, quasi-static tensile tests using specimens with cut central plies.
Short beam shear and tensile tests were conducted on a computer-controlled Zwick Z250 universal electro-mechanic test machine, fitted with a regularly calibrated 250 kN load cell. The strains during the tensile tests were measured with a Mercury RT type optical extensometer system with a 5 MPixel Mercury Monet camera (Sobriety). This camera was also used for monitoring the failure modes of the short-beam shear test.

2.4 Results and discussion

Oscillatory rheometry revealed that the application of the dwell included in the thermal cycle limits the viscosity drop, which might result in a smaller extent of resin leakage (see Fig. 1.). The application of a dwell with a higher temperature may cause the opposite effect and help to maintain low viscosity values longer than in the case of a thermal cycle without dwell.

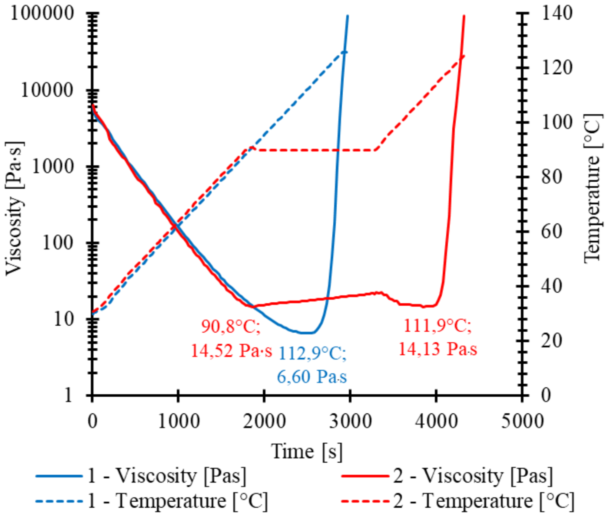

Fig. 1. Viscosity measurements of two different cure cycles, 1) without dwell, 2) with a 25 min long dwell at 90°C

The results of thermogravimetry showed that the general cycle caused the largest extent of resin leakage, which resulted in the highest fiber volume fraction. The reduction of pressure or heating rate and the application of the dwell causes smaller resin leakage, thus lower fiber volume fraction. Density measurements showed that higher pressures applied during the cure cycle reduce porosity.
The short-beam shear tests (see Fig. 2.) and the tensile test with cut plies specimens revealed that a higher applied pressure or higher resin content result in higher values of interlaminar shear strength and mode II fracture toughness.

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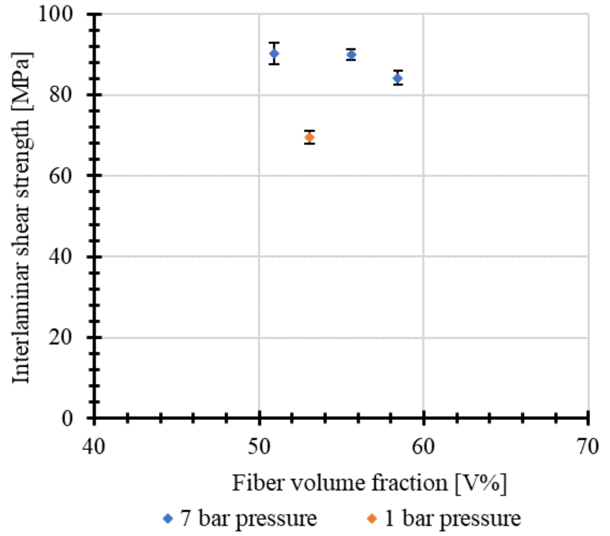

Fig. 2. Fiber volume fraction- interlaminar shear strength diagram of the different composite plates