

# GLASS/CBT LAMINATE PROCESSING AND QUALITY ASPECTS

R.T. Durai Prabhakaran, Tom Løgstrup Andersen, Aage Lystrup

*Materials Research Division, Risø National Laboratory for Sustainable Energy,  
Technical University of Denmark (Risø DTU), Frederiksborgvej 399, 4000 Roskilde,  
DENMARK. Email: rtdp@risoe.dtu.dk*

**ABSTRACT:** This article reports details on manufacturing and quality of laminates based on glass fibre fabric and glass fibre roving in combination with cyclic butylenes terephthalate (CBT) matrix, which is suitable for resin infusion. At this stage, Cyclics<sup>®</sup> CBT 160 resin in powder form was used to manufacture a 5 mm thick laminate by alternating layer of CBT powder and glass fibre No-Crimp-Fabrics (NCF) or filament wound Unidirectional (UD) lamina. The lay-ups were vacuum bagged and consolidated into laminates by vacuum consolidation. The present article investigates the process difficulties, control of parameters, and the microstructure of thermoplastic Glass/CBT laminates. The study shows the difference in the laminate quality produced using NCF and UD glass fibre roving wounded onto a frame. The microscopy study reveals the material quality for both laminates as well as the process intricacies.

**KEYWORDS:** CBT resin, Vacuum consolidation, Glass roving, Quality control, Filament winding, Voids

## INTRODUCTION

Few researchers have reported in the literature that thermoplastic polymers are upcoming resins in developing large sized composite structures using resin infusion techniques. Compared to thermosetting, the thermoplastic polymer matrix materials for fibre composites have some promising advantages. One of the principal advantages of traditional thermoplastic polymers is their ability to melt, flow and impregnate fibres at elevated temperatures, although it is not so easy as the melt viscosity is a magnitude higher than the viscosity of thermosetting polymers. However, this melt impregnation possibility also limits the upper temperature at which thermoplastic can be used [1-3].

Cyclic butylenes terephthalate (CBT) is a low viscosity cyclic oligoester which polymerizes, after adding a catalyst, into the better known thermoplastic polybutylene terephthalate (PBT). The low viscosity makes the resin suitable for resin infusion. The main objective of this study is to conduct manufacturing trials using different glass fibre forms and CBT 160, which is a powder based resin from Cyclic Corporation, USA [4]. The laminate dimensions considered for this study are 300 x 300 x 5mm and 580 x 490 x 5mm. Vacuum consolidation technique is used to estimate various process parameters and to control the quality of the laminates. Standard microscopy was used to evaluate the quality (voids) of the laminates.

## EXPERIMENTAL WORK

The first laminate is manufactured from a no-crimp-fabric (NCF) of glass fibres from Devold AMT with the Cyclics CBT 160 resin. The laminate is symmetric with the stacking sequence  $[0^0/90^0/CSM]_{3S}$ . The second laminate is a unidirectional laminate made of glass fibre roving (PPG 4586 2400TEX). The glass fibre roving is wound on a metal frame using filament winding technique. The volume fraction of fibres is 50% in both laminates. CBT 160 resin is a powder based resin, which can be distributed uniformly at the time of laying up the glass fibres (see Fig. 1). Both laminates are consolidated by vacuum consolidation technique. The process is monitored using thermocouples, which are placed at the centre of the stacked laminas as well as on the top of the lay-up and bottom tool plate.

Both laminates are processed under full vacuum throughout the cycle. The process cycle starts with heating from room temperature to 120<sup>0</sup>C, which is hold for 1.5 hrs to remove all moisture from the material and vacuum bag. Next steps are heating from drying temperature to 200<sup>0</sup>C and holding at this temperature until the inside of the laminate has seen 190<sup>0</sup>C for 1 hr. Heating further to raise the temperature to 230<sup>0</sup>C in 15min and holding it for 45min. At last, cooling down to room temperature as fast as possible as shown in the process cycle diagram (see Fig. 2).



Fig. 1 Lay-up and the oven (in this case an autoclave) used for the vacuum consolidation process

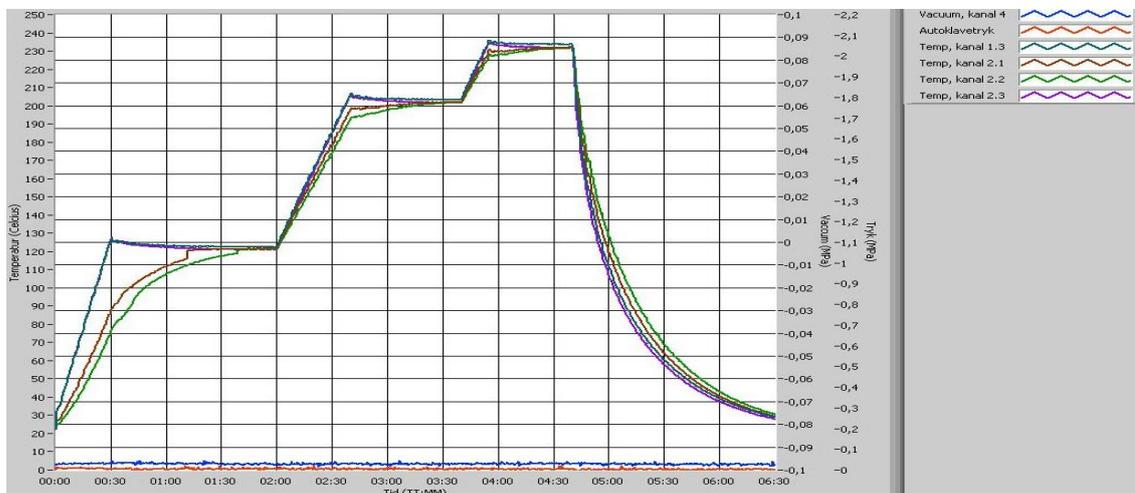


Fig. 2 Process cycle – vacuum consolidation process

The quality of the manufactured laminate depends mainly on the process parameters, quality of the used semi-raw materials, impregnation characteristics, and the tooling. The section below describes the quality aspects of consolidated laminates using microscopic analysis.

### **MICROSTRUCTURAL ANALYSIS**

Characterization of the microstructure of the consolidated laminates helps to get a better understanding of the relationships between mechanical properties microstructural homogeneity, and fibre-matrix interface. Two specimens from each of the laminates were investigated. One of the specimen contains fibres perpendicular to surface (Images A, C, E, G as shown in Fig. 3), and the other specimen has fibres parallel to the surface (Images B, D, F, H as shown in Fig. 3). Both polished and unpolished cross sections are investigated.

The impregnation quality was investigated by optical microscopy and very few inter-bundle voids are observed (Fig. 3C), whereas many roving has larger areas of intra-bundle dry fibres (porosities) as shown in Fig. 3A, 3E and 3G. Most images show resin rich areas, and Fig. 3D, 3G and 3H clearly show big cracks running through the matrix and into the fibre roving (Fig. 3G).

The process cycle and the slow cooling rate for the thick laminate create a resin which is highly crystalline and brittle, and the many micro cracks are formed during cooling to room temperature. These cracks can be seen in Fig. 3B and 3D. From this microscopic study, it appears that the CBT/Glass NCF and CBT/Glass UD laminates processed by vacuum consolidation technique have defects such as resin rich areas, voids, dry fibres (porosity) and matrix cracks running into the fibre bundles. Based on the defects observed at the entire inspected areas of the specimens, the laminate with NCF fabric shows defects like resin rich areas, crack running through the matrix and into the fibre roving whereas the UD laminate shows defects like porosities, voids, intra-bundle dry fibres and inter-bundle voids.

From ongoing work (not reported here) at Risø DTU various testing results indicate that the bond between fibre and matrix leads to relatively high ILSS and Compression strength, but as shown in Fig. 3 the microstructure reveals many cracks in the matrix, which indicate high level of residual stresses and/or a brittle matrix, respectively. The ILSS fracture are more like a true interlaminar shear fracture as for ordinary thermosetting fibre composites, which also indicate that CBT is more brittle than the other typical types of thermoplastic. If CBT resins are crystallized from a truly random melt at a sufficiently high cooling rate, they will behave as classical injection moulded PBT.

The future work includes estimating of the molecular weights and degree of crystallization using gel permeation chromatography (GPC) which will give information on the polymerization aspects related to the polymer (CBT) used in this study. The continued work will also include the study of the structure and properties relationship of the G/CBT composites, as per the quantitative structure-property relationship (QSPR) studies [5].

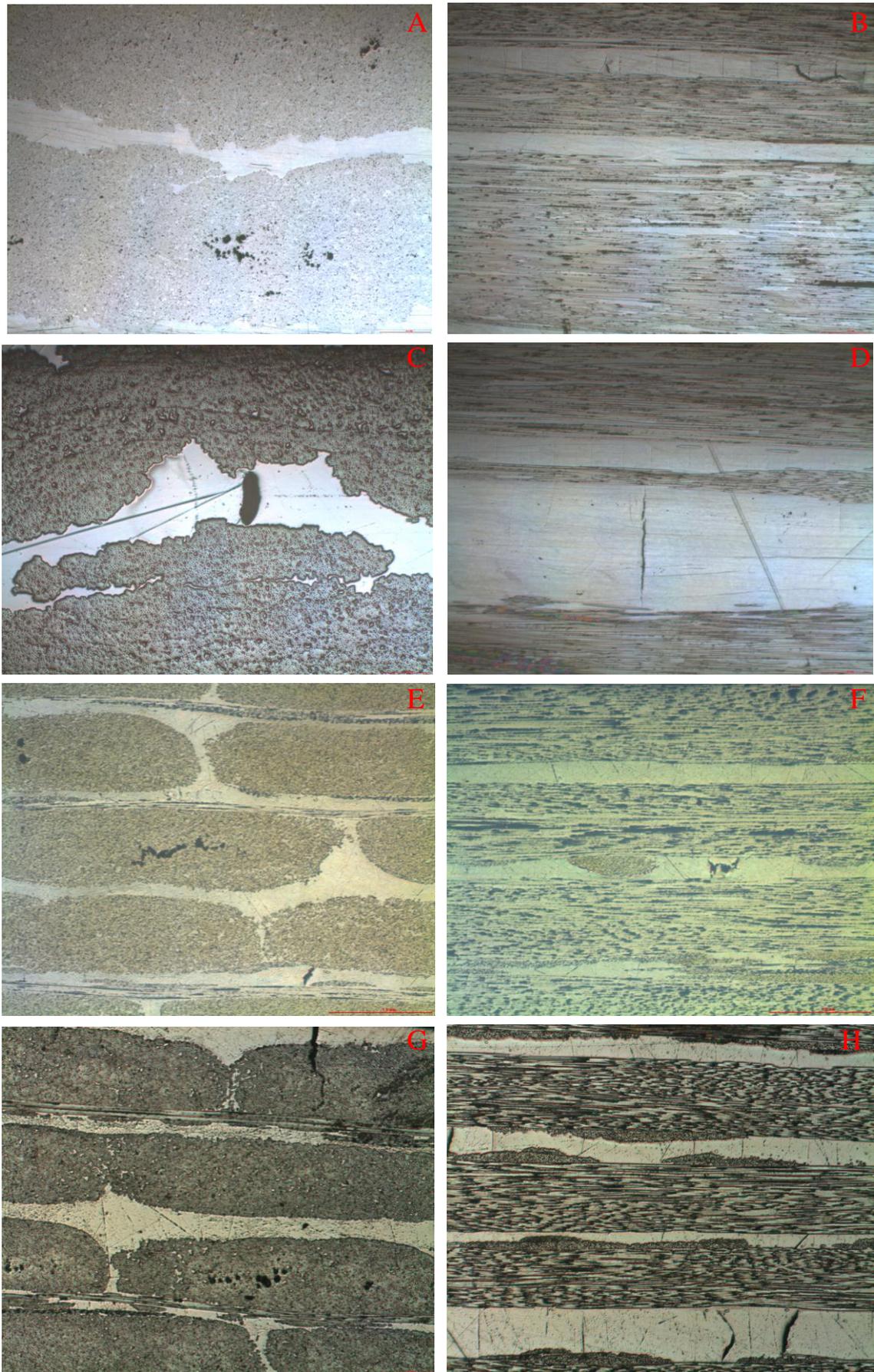


Fig. 3 A 30 x optical micrograph of polished and unpolished cross-section of a G/CBT symmetric and unidirectional laminates (1<sup>st</sup> and 2<sup>nd</sup> row unpolished and polished unidirectional G/CBT; 3<sup>rd</sup> and 4<sup>th</sup> row unpolished and polished symmetric G/CBT NCF)

## CONCLUSIONS

Procedure for manufacturing thermoplastic fibre composite laminates from glass fibre No-Crimp-Fabric or filament wound UD glass fibre roving and Cyclics<sup>®</sup> CBT 160 powder based resin is explained, and the following are the critical finding:

1. The viscosity is sufficient low at a temperature of 200 °C to impregnate the fibres.
2. Even though the polymer were melted (temperature raised to 230 °C) before cooling, the formed matrix becomes highly crystalline and brittle.
3. Microscopic study reveals that, the processed material have many defects such as resin rich areas, voids, dry fibres (porosity) and matrix cracks running into the fibre bundles.
4. The quality of both laminates produced by the vacuum consolidation technique is compared with respect to process induced defects.

## ACKNOWLEDGMENTS

This work is a part of the 'BLADE KING' project supported by the Danish National Advanced Technology Foundation. The other project partners are LM Glasfiber A/S (Project leader), Comfil ApS, and Aalborg University (Department of Mechanical Engineering). Special thanks to the partners, for their support provided for our research studies at Risø National Laboratory for Sustainable Energy, Technical University of Denmark (Materials Research Division) within the Blade King Project. The authors would also like to thank the material (resin) supplier Cyclics Corporation, USA for their support and useful discussion in carrying out this study.

## REFERENCES

1. C.D. Rudd, A.C. Long, K.N. Kendall and C.G.E. Mangin, "Liquid Moulding Technologies", *Woodhead Publishing Ltd, Cambridge, England*, (1997).
2. K. Van Rijswijk and H.E.N. Bersee, "Reactive processing of textile fiber-reinforced thermoplastic composites - An overview", *Composites Part A: Applied Science and Manufacturing*, Vol. 38, no. 3, pp. 666-681 (2007).
3. H. Parton, J. Baets, P. Lipnik, B. Goderis, J. Devaux, I. Verpoest, "Properties of poly(butylenes terephthalate) polymerized from cyclic oligomers and its composites", *Polymer*, Vol. 46, pp. 9871 – 9880 (2005).
4. <http://www.cyclics.com/>
5. R.T. Durai Prabhakaran, B.J.C Babu, V.P. Agrawal, "Quality Evaluation of Resin Transfer Moulded Products", *Journal of Reinforced Plastics and Composites*, Vol. 27, No. 6, pp. 559-581 (2008).