IMPACT OF IONIC LIQUID ON THE MECHANICAL PERFORMANCE OF MATRIX POLYMER FOR FIBRE REINFORCED MATERIALS FOR ENERGY STORAGE

M. Wienrich^{1*}, G. Kalinka¹, E. S. Greenhalgh², S. Carreyette⁴, M. Bistritz¹, N. Shirshova³, M. Houllé⁵, L. Asp⁶, A. Bismarck³, Q.P.V. Fontana⁴

¹BAM Federal Institute for Materials Research and Testing, Berlin, Germany
 ²Composite Centre, Imperial College London, London, U.K.
 ³Department of Chemical Engineering, Imperial College London, London, U.K.
 ⁴UMECO Composites Technology Centre, Heanor, U.K.
 ⁵Nanocyl s.a., Sambreville, Belgium
 ⁶Swerea SICOMP, Mölndal, Sweden
 *malte.wienrich@bam.de

Keywords: multifunctional epoxy resin, fibre-matrix adhesion, single fibre pull-out test, ionic liquid

Abstract:

For the concept of using structural materials such as carbon fibre reinforced plastics as energy storage devices, new matrix polymers are required. These polymers must provide ionic conductivity as well as adequate mechanical strength. In the EU-Project StorAGE this requirements are fulfilled by adding ionic liquid to commercial polymers. The mechanical properties of these mixtures materials were characterized by using a 3-point-bending device. In addition, single fibre pull test were performed in order to get information on the interfacial shear strength. Adding of ionic liquid has an impact on the mechanical performance of the materials. A decrease of the flexural strength and modulus of less than 10% of the value of the reference materials took part. The interfacial shear strength decreased to a value of around one third compare to the reference material.

1 Introduction

Recent studies show that fibre reinforced polymers can work as both, structural materials and storage for electrical energy, such as a capacitor device reported by Carlson et al. [1]. However, in order to enable the electrical functionality, it is necessary to change the matrix polymer from a nearly perfect insulator to a good ionic conductor (i.e an electrolyte). In the EU-Project StorAGE, dramatic improvements in the ionic conductivity were achieved by adding an well dispersed ionic liquid to the matrix materials. The content of this liquid in the polymer was up to 55 wt%. The ratio of 50:50 (wt%) between the structural polymer and the ionic liquid was a promising compromise between the mechanical and the electrical requirements. Nevertheless, the partial substitution of the solid phase with a liquid leads to a decrease in the mechanical performance. The focus of these investigations was on mechanical performance of the matrix containing ionic liquid and the fibre-matrix adhesion with these materials- The carbon fibres used for this study have a layer of direct grown carbon nano tubes (CNT) on their surface. The CNT layer leads to a significant increase of the surface area, also reported in studies of Qain et al. [2], which improves the electrical properties in the structural energy storage devices. For the mechanical investigations reported in this paper, standard methods like 3-point bending test were used as well as more sophisticated methods such as the single fibre pull-out test to characterise the impact of the ionic liquid as well on overall mechanical properties as of the fibre matrix interphase of future composites.

1.1 Results

The flexural strength and the flexural modulus of four different matrix systems with and without ionic liquid are shown in Figure 1. The measurement conditions of the 3-point bending tests are described below. Both diagrams in Figure 1 illustrate the impact of adding ionic liquid to the base polymer on the mechanical properties. The value of the flexural strength decreases about 90% by adding the ionic liquid. It is the same range for the flexural modulus. Increasing the content of ionic liquid from 45 wt% up to 55 wt% is not leading to significant decrease of the strength and modulus respectively.

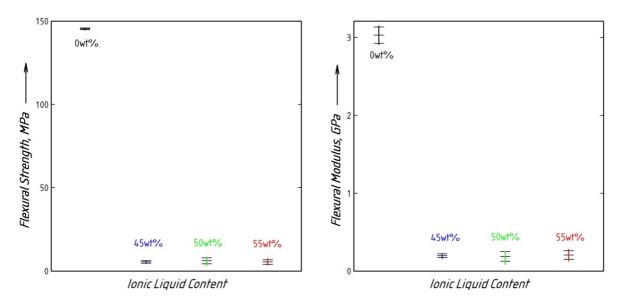
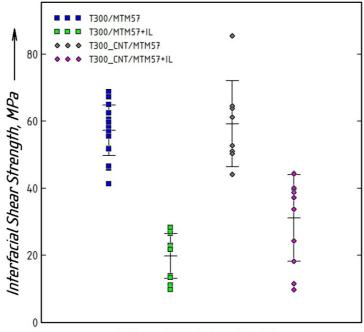


Figure 1. Flexural strength (left) and flexural modulus (right) of four matrix materials with and without ionic liquid

The interfacial shear strength of four different fibre-matrix combinations are shown in Figure 2. The adding of ionic liquid to the polymer leads to a decrease of the interfacial shear strength for both surface modifications. The values drop down to approx. less than 50 % of the values which are obtained from testing with the pure resin. "CNT" modified surface of the carbon fibre leads to a slightly higher value of interfacial shear strength for both, the combinations with and without ionic liquid, compare to the "as-received" modified fibre.



Fibre - Matrix Combination

Figure 2. Interfacial shear strength of two surface modifications of the T300 carbon fibre in combination with two matrix materials with and without ionic liquid based on the epoxy material MTM57

1.2 Conclusion

As expected, the mechanical properties of the matrix materials decreased by adding the ionic liquid, due to a significant loss of bulk material. The loss of around 90 % of the mechanical strength is dramatic. However, development on these kind of materials is still under process. In combination with reinforcing carbon fibres it can lead to laminates which properties are a suitable compromise between electrical and mechanical demands. Promising is also the fact that the interfacial shear strength is not decreasing in the same way as the mechanical properties of the matrix materials when a ionic liquid is added. The use of CNT modified carbon fibres lead to a slightly higher value of interfacial shear strength. This result is also been reported by Qian et al.[2]. The fairly good fibre-matrix interaction might help to compensate the loss of mechanical strength.

1.3 Acknowledgements

Research leading to these results was founded by the EU in the 7th Framework programme (Grant Agreement No. 234236).

2 Materials and testing methods

The base polymer for the multifunctional resins is MTM57 which is a commercial prepreg resin of UMECO. To transform the resin in an ionic conducting material, an ionic liquid doped with lithium salt was added. The contents of ionic liquid in the base polymer are 0 wt%, 45 wt%, 50 wt% and 55 wt% respectively. ACG also supplied the carbon fibres T300 in the modification "as-received". Nanocyl s.a. modified the T300 carbon fibres by direct growing of carbon nano tubes on the fibre surface (modification"CNT").

For the mechanical characterisation of the multifunctional resins a 3-point-bending device was used. The device is implemented in a Zwick testing machine and the tests were carried out in a displacement-controlled modus. In order to compensate the variation of the sample shape, caused by the cutting process, the tests were carried out with a constant peripheral strain rate. For this purpose, the anvil speed v was adjusted by using the following equitation (1),

$$v = \frac{\mathcal{E}_{dot} l^2}{6h} \quad [\text{mm/min}] \tag{1}$$

where \mathcal{E}_{dot} [-] is the peripheral strain rate, l is the span [mm] and h is the height [mm]. The 3-pointbending tests were carried out for four different materials with ionic liquid contents of 0 wt%, 45 wt%, 50 wt% and 55 wt% respectively. The samples for the 3-point-bending test were milled from plaques with an air cooled milling device. Afterwards the dimensions of the samples were measured with calibrated measuring equipment.

The single fibre pull-out tests to characterise the fibre-matrix adhesion are carried out with a special testing equipment described by Hampe et. al [3]. The interfacial shear strength τ_{IFSS} was calculated according to the Kelly-Tyson model [4] with the following equitation,

$$\tau_{IFSS} = \frac{F_{\text{max}}}{d_f \pi d_{emb}} \quad [MPa]$$
⁽²⁾

where τ_{IFSS} [MPa] is the interfacial shear strength, d_f is the fibre diameter and l_{emb} is the embedding length. The pull-out tests were carried out with both, the "as-received" and "CNT" modified type of the T300 carbon fibre. As the matrix polymer, the pure MTM57 and a mixture of it with 50wt% ionic liquid was used. The sample preparation for the single fibre pull-out test was done with a semiautomatic embedding device. Before pull out test, the fibre diameter was determined with a Zeiss microscope.

References

- Carlson T., Ordeus D., Wysocki M., Asp L.E. Structural capacitor materials made from carbon fibre epoxy composites. *Composites Science and Technology*, **70**, pp. 1135-1140 (2010)
- [2] Qian H., Bismarck A., Greenhalgh E.S., Shaffer M.S.P. Carbon nanotube grafted fibres: A study of wetting and fibre fragmentation. *Composites Part A*, **41**, pp. 1107-1114 (2010)
- [3] Hampe A., Kalinka G., Meretz S., Schulz E. An advanced equipment for single-fibre pull- out test designed to monitor the fracture process. *Composites*, **26**, pp. 40-46 (1995)
- [4] Kelly A., Tyson W.R., Tensile Properties of Fibre-Reinforced Metals: Copper/Tungsten and Copper/Molybdenum. J. Mech. Phys. Solids, 13, pp. 329-350 (1965)