

# INVESTIGATION OF THE EFFECT OF SIZING ON THE TENSILE AND INTERFACE PROPERTIES OF CONTINUOUS BASALT FIBRE AND POLYPROPYLENE

Peter G. Jenkins<sup>1</sup>, Liu Yang<sup>1</sup> and James L. Thomason<sup>1</sup>

<sup>1</sup>Mechanical & Aerospace Engineering, University of Strathclyde, James Weir building, 75 Montrose Street, Glasgow, United Kingdom

Email: peter.jenkins@strath.ac.uk, Web Page:

<http://www.strath.ac.uk/engineering/mechanicalaerospaceengineering/advancedcompositesgroup/>

**Keywords:** basalt fibre, polypropylene, tensile properties, interfacial shear strength, surface topography

## Abstract

The mechanical performance of continuous basalt fibres (BFs) and E-glass fibres (GFs) was investigated using single fibre tensile and microbond interface testing. Fibre surface topography was also studied, using atomic force microscopy. Continuous fibres of identical compositions with various surface sizings were used; interfacial shear strength was investigated using polypropylene (PP) matrix. Data were analysed alongside E-glass fibres with similar surface coatings. The tensile performance of basalt fibres varied significantly depending on the surface sizing applied at point of manufacture but little to no difference was found compared with the similarly sized E-glass fibres. The apparent interfacial shear strength (IFSS) between BFs and PP was significantly improved when sizing was applied. Both unsized BF and GF demonstrated very poor IFSS with PP but the application of silane to GFs produced a significantly greater improvement in the apparent adhesion. Characteristic differences in the surfaces of both unsized and silane-coated BFs and GFs were found by AFM analysis.

## 1. Introduction

Basalt fibre (BF) has been proposed as a potential competitor to the very widely used glass fibre (GF), as a fibrous reinforcement for polymer matrices [1,2]. It has at times been referred to as a ‘natural’ fibre as its singular raw material, basalt, does not require synthesis in the manner that traditional glass fibre formulations do. In terms of mechanical performance, the single fibre strength and Young’s modulus are stated as at least comparable with commonly used GFs such as E-glass: the modulus, in particular, is often quoted as approaching 90 GPa [2,3] which is significantly higher than E-glass fibres.

In addition to the mechanical properties of reinforcement fibres, it is well-established that the interface between fibre and matrix plays perhaps the most critical role in defining the success or failure of a composite [4]. A great deal of research over many years has been carried out into the interface between GFs and a huge range of polymer matrix materials. Due to their relative novelty, there are less data available on BF-polymer interface performance. Greco et al. [5] studied prototype sized and unsized fibres in addition to fully commercially sized BFs. They measured single fibre strength and used single fibre fragmentation test to assess adhesion between these fibres and polypropylene matrix both with and without addition of maleic anhydride grafted PP. They found that unsized BF performed most poorly of the fibres investigated for tensile strength, modulus and apparent adhesion. The addition of maleic anhydride to the PP matrix generally improved adhesion as expected, but the degree of this improvement varied significantly depending on BF selected: in some cases apparent adhesion doubled but in others a more modest increase of no more than 50 % was obtained.

It was also found in the study [5] that the unsized fibre performed almost as well as the various sized fibres investigated, in both maleated and unmodified polypropylene; similar findings have reported by other researchers [6]. The effect that surface silane or sizing has on adhesion between GFs and PP, for example, has been demonstrated [7–9]: with glass fibre that is bare, or whose surface coating is significantly degraded, apparent interfacial shear strength (IFSS) from the microbond test of the order 7 – 9 MPa. Suitable sizing, however, even with unmodified PP can produce IFSS values in excess of 15 MPa.

The study presented here was carried out to systematically investigate BFs of identical chemical composition, to which various surface coatings were applied at point of manufacture. Using this methodology the effect on fibre tensile properties and on interfacial performance of sizing components such as silane and lubricant was investigated. Much of the sizing used on BF is copied directly from GF technology, therefore it is not necessarily optimised for BFs. To investigate this hypothesis, where possible in our study comparisons between BFs and GFs with similar surface coatings were made.

## **2. Experimental**

### **2.1. Materials**

Single end rovings of basalt fiber (800 tex, average diameter  $14.7 \pm 0.4 \mu\text{m}$ ) were provided by Mafic Ireland (Kells, County Meath, Ireland). Four rovings were provided: non-sized or bare (NS), silane coated only (S), silane and lubricant coated (S+L) and fully commercially sized (FS). All fibres were cooled by water spraying under the bushing; for those with surface sizing it was applied using a rolling applicator before strands were gathered and wound into packages.

Boron free E-glass (Advantex) fibres sized with different silane coupling agents supplied by Owens Corning were investigated. Two rovings were studied, produced on the same pilot scale bushing and received as 20kg continuous single-end square edge packages. The rovings had nominal tex of 1200 g/km and an average fibre diameter of  $17.5 \mu\text{m}$ . No sizing was applied to the bare fibres which had only been water sprayed by the cooling sprays beneath the bushing; these fibres are designated GF\_NS. Immediately following the cooling step the chosen sizing was applied to the sized fibres with a normal rotating cylinder applicator. The fibres designated GF\_S were coated with amino-silane only.

The polypropylene (PP) was provided by Braskem as general purpose under the code CP1200B with a nominal flow rate of 230 °C/ 2.16 kg.

### **2.2. Tensile testing**

Tensile testing was carried out using an Instron 3342 single column machine and following the methodology described in ASTM C1557-03. Fibres were tested at 5 gauge lengths between 5 – 80 mm and using a constant strain rate of 1.5 %/min. The process of sample preparation and testing is described in detail in [10].

### **2.3. Microbond interface testing**

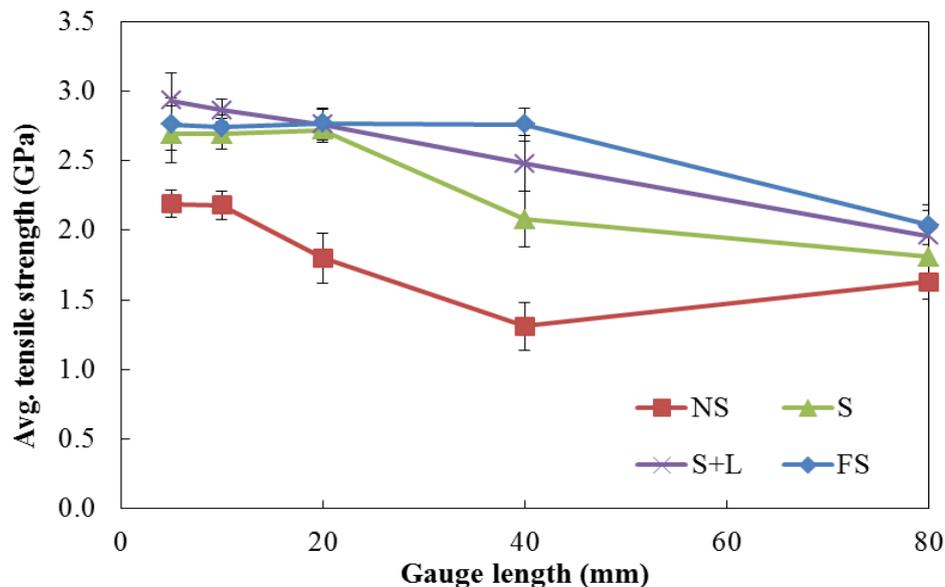
The apparent interfacial shear strength of various BFs and GFs with polypropylene was carried out using the microbond test developed in-house and described in detail by Yang and Thomason [9]. At least 30 individual successful debonds were analysed for BFs and at least 20 for GFs.

### **2.4. Atomic Force Microscopy (AFM)**

Glass fibre surfaces were imaged using a Bruker Innova instrument. Height and phase data were collected in Tapping Mode® using a OLTESPA-R3 visible apex type tip with nominal cantilever spring constant of 2 N/m and resonant frequency 70 kHz. Three fibres were imaged for each condition and 2 images of approximately 3 µm x 3 µm were captured on each of these fibres. Images were obtained at a rate of 0.75 Hz with the fast scan direction being perpendicular to the longitudinal fibre axis. The roughness of each fibre surface image was quantified using root mean square (RMS) roughness; defined as the square root of mean square of the height deviation from the mean elevation plane, after removing the background curvature by polynomial fitting. Statistics generated from raw AFM data were produced using NanoScope Analysis software.

### 3. Results

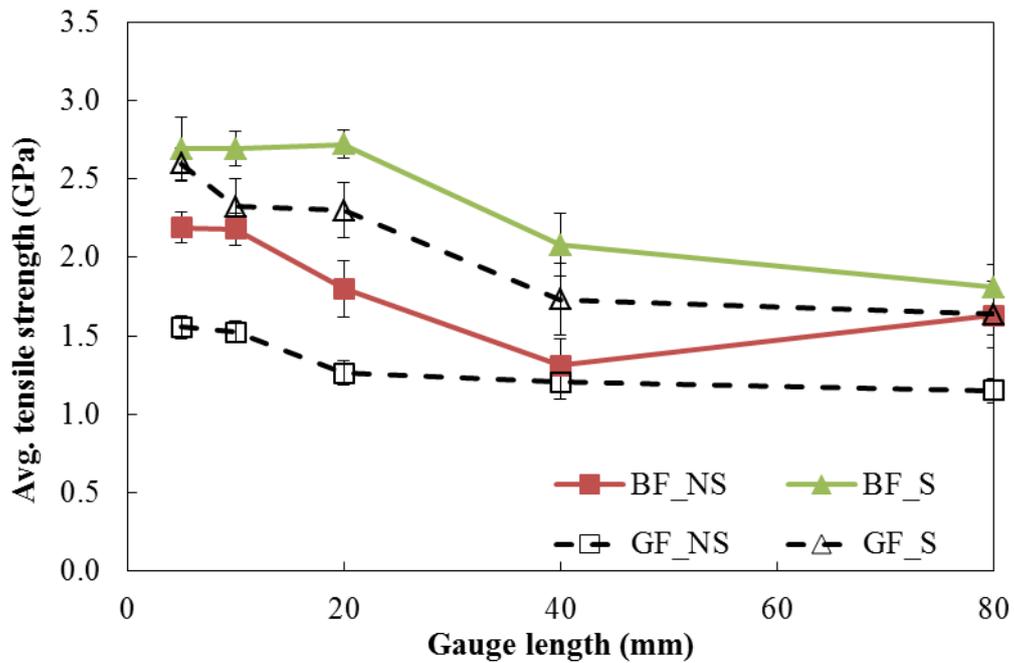
The tensile strength results of the BFs with 4 different sizings are presented in Fig.1. The error bars represent 95 % confidence limits, in this and all subsequent figures.



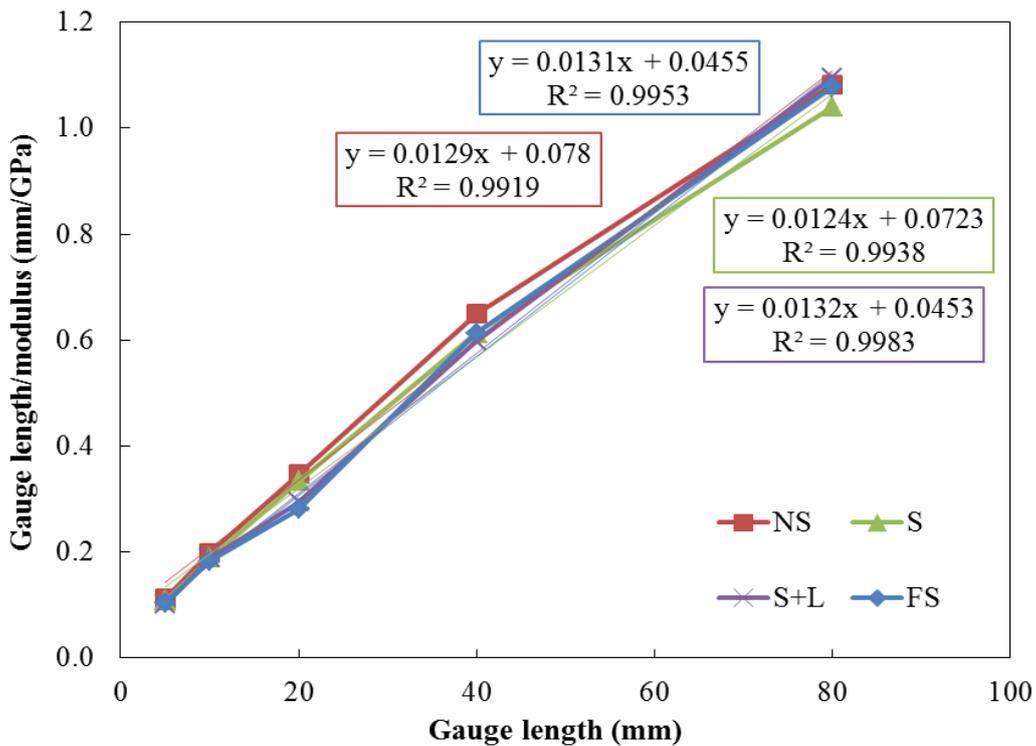
**Figure 1.** Average tensile strength of BFs against sample gauge length

These results clearly indicate the critical importance of suitable fibre surface coating during manufacture to maintain fibre strength; the NS sample was significantly the weakest at every gauge length. At shorter gauge lengths of 20 mm and below the protective effect of silane, silane and lubricant and full sizing was almost indistinguishable. At the shortest gauge length, however, FS samples appeared to be strongest which fits with expectations given the much greater protection from mechanical degradation of strength that this thicker multi-component size should provide.

The strength of unsized and silane-coated BFs is compared with similarly surface-coated GFs in Fig. 2.



**Figure 2.** Tensile strength of unsized and amino-silane coated BFs and GFs at gauge lengths 5 – 80 mm (GF strength data reproduced from [10])



**Figure 3.** Transformed Young's modulus of BFs against sample gauge length

The strength of GFs was lower than the BFs with corresponding surface coating at the majority of gauge lengths tested. Similar to the data in Fig. 1, the importance of fibre coating to the retention of fibre strength is evidenced in Fig. 2. Unsized GF was significantly weaker than BF particularly at short

gauge lengths. Conversely, the strength of silane-coated GF and BF was similar at 5 mm and was within 0.4 GPa or less at any gauge length.

The results of the gauge length study of Young's modulus are given in Fig. 3. The ordinate has units of transformed Young's modulus in mm/GPa such that a linear trend may be plotted; from the gradient of this line the compliance corrected value of Young's modulus is obtained, following the procedure outlined in greater detail in [10].

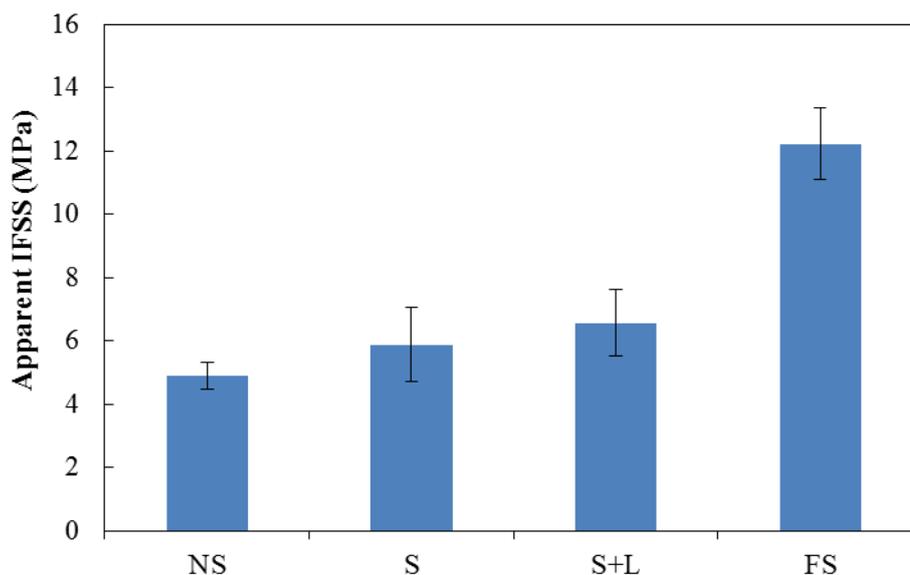
Calculated from the inverse of the respective gradients of trend lines, the Young's moduli of the basalt fibres are presented in Table 1.

**Table 1.** Compliance corrected Young's modulus of BFs

Fibre type	NS	S	S+L	FS
Young's modulus (GPa)	77.6	80.7	75.6	76.4

The values of compliance corrected Young's moduli for the BFs were relatively similar; the silane-coated fibre produced the highest value of 80.7 GPa while the lowest was the silane and lubricant coated sample at 75.6 GPa. In comparison, the unsized and amino-silane coated GFs for which strength data were presented in Fig. 2 yielded compliance corrected moduli of 78.7 and 76.9 GPa respectively.

The apparent IFSS of the 4 types of BF investigated in this study with unmodified polypropylene matrix are presented in Fig. 4.



**Figure 4.** Average IFSS of BFs in PP matrix [NS = no sizing, S = amino-silane, S+L = silane and lubricant, FS = full sizing]

Interfacial strengths obtained for bare fibre was poor as expected at around 5 MPa. The application of silane or silane and lubricant to BF produced only a small increase in IFSS, although from the error

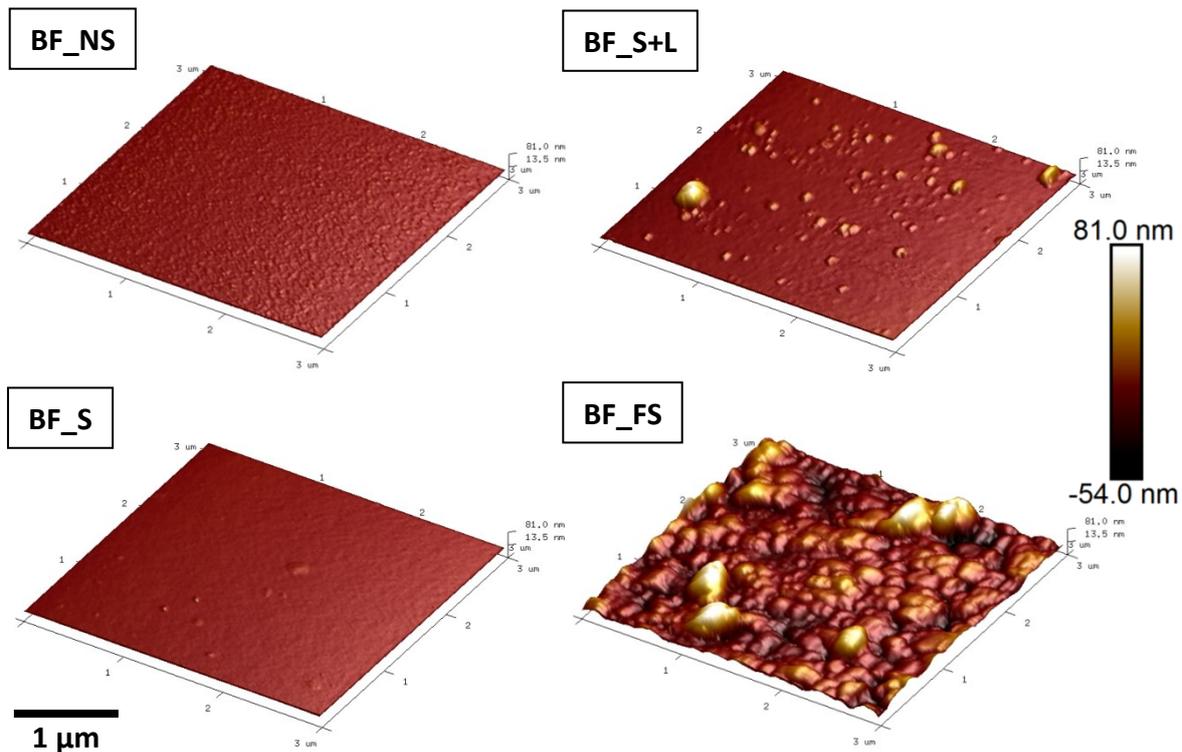
bars in Fig. 4 it is shown that this increase was significant. The highest IFSS was measured for fully commercially sized BF at approximately 12 MPa.

Results of the roughness analysis from AFM data are presented in Table 2.

**Table 2.** Average RMS roughnesses (nm) of BFs from AFM analysis

Sizing			
<i>NS</i>	<i>S</i>	<i>S+L</i>	<i>FS</i>
0.94	0.47	2.79	25.39

Typical plots of the topography of BFs with each of the 4 coatings used in the study are shown in Fig. 5.



**Figure 5.** 3D topography plots of BFs [NS = no sizing, S = amino-silane, S+L = silane and lubricant, FS = full sizing]

The surface of unsized BF was found to be homogeneous, but relatively rough. Application of amino-silane to these fibres produced a clear effect in the topography data; it appeared that the rough surface structure was obscured by the silane layer and this significantly reduced the RMS roughness. No evidence was found of the apparent ‘islands’ of amino-silane that have been reported on the surface of glass fibres [11]. The surface of BFs coated with both silane and lubricant were the least consistent in terms of topography and roughness. There was some evidence of segregation of the sizing coating components; raised features were of a different phase to the surrounding surface. The coating on fibres with full commercial sizing was significantly thicker and with very different morphology.

#### 4. Discussion

The single fibre tensile data presented demonstrate the importance of proper sizing to the strength of fibres, in addition to role played in forming a successful interface. Data in Fig.1 showed that unsized fibre performs poorly; the addition of silane alone greatly improved fibre strength but a thicker multi-component sizing produced the best performance particularly at longer gauge lengths. A comparison of the strength of unsized and amino-silane coated BF and GF was presented in Fig. 2. In both cases BF was found to be stronger than GF; the strength of bare BF was about 40 % greater than GF at all gauge lengths investigated, except 40 mm. This discrepancy is also notable as the relationship between decreasing strength with increasing gauge length is not followed in the data for BF\_NS at 40 and 80 mm. The strength of silane-coated BF was at most 20 % higher than GF and at the majority of gauge lengths significantly less so. Our tensile strength results support the assertion that has been made in the literature that BF strength is slightly higher than that of GF [12]; the difference, however, was found to be relatively moderate.

Regarding the Young's modulus of BF and GF, our results suggested that there was no significant difference, in contrast to some published data [2,12]. A range of 75.6 – 80.7 GPa was measured for the 4 BFs; for GF, values within this range of 76.9 & 78.7 GPa have been reported [10]. It is not expected that surface coating should affect fibre modulus, and the ranges obtained are likely due to spread in the raw modulus data.

The effect of sizing on the interfacial shear strength of BF with unmodified PP was demonstrated by the data presented in Fig. 4. Bare fibre performed poorly as expected, but neither the application of amino-silane nor silane and lubricant produced more than a slight improvement. This result was somewhat unexpected as the application of coupling agent is anticipated to improve the BF-PP interface and produce significantly higher IFSS, in a similar manner as it does with GF-polymer interfaces. An improved IFSS of above 12 MPa was obtained only for fully sized fibre; the PP-compatible sizing present on these fibres may contain numerous additional elements, such as maleated PP, which could have produced this increase. In order to investigate the lack of efficacy of amino-silane to improve IFSS, studies using maleic anhydride grafted-PP as matrix are proposed, as well as comparison with the performance of GFs in these same matrices.

The AFM data presented showed clearly the physical changes at the fibre surface due to additional elements in the fibre coating. Correlation between surface roughness and IFSS was not found; in fact, IFSS increased slightly as roughness decreased due to application of silane to the BF surface.

#### 5. Conclusion

The tensile properties of basalt fibres with various coatings were investigated and compared with glass fibre. The strength of unsized BF was low but improved with the addition of amino-silane and subsequently increased further as additional sizing components were included in the fibre coating. Unsized and silane-coated BFs and GFs were compared: average tensile strength of BFs was found to be slightly greater than GFs, but this difference decreased when silane coating was present. Significantly greater Young's modulus of BF, which has been suggested in the literature, was not found in our study; rather, the modulus of BFs and GFs were effectively the same in the approximate region 75 – 80 GPa. Interfacial shear strength between BF and unmodified PP was surprisingly low when amino-silane was present; it performed only marginally better than unsized BF. Further investigation of this finding is necessary as it is important to establish whether silanes, such as amino-silane, perform in the same manner on BFs as it is generally accepted that they do on GFs to enhance interfacial properties.

## References

- [1] Vikas G, Sudheer M. A Review on Properties of Basalt Fiber Reinforced Polymer Composites. *Am J Mater Sci* 2017;7:156–65.
- [2] Asadi A, Baaij F, Mainka H, Rademacher M, Thompson J, Kalaitzidou K. Basalt fibers as a sustainable and cost-effective alternative to glass fibers in sheet molding compound (SMC). *Compos Part B Eng* 2017;123:210–8.
- [3] Fiore V, Scalici T, Di Bella G, Valenza A. A review on basalt fibre and its composites. *Compos Part B Eng* 2015;74:74–94.
- [4] Thomason JL, Adzima LJ. Sizing up the interphase : an insider’s guide to the science of sizing. *Compos Part A Appl Sci Manuf* 2001;32:313–21.
- [5] Greco A, Maffezzoli A, Casciaro G, Caretto F. Mechanical properties of basalt fibers and their adhesion to polypropylene matrices. *Compos Part B Eng* 2014;67:233–8.
- [6] Zykaite R, Purgleitner B, Stadlbauer W, Burgstaller C. Microbond test development and interfacial shear strength evaluation of basalt and glass fibre reinforced polypropylene composites. *J Compos Mater* 2017;51:4091–9.
- [7] Nagel U, Yang L, Kao CC, Thomason JL. Effects of Thermal Recycling Temperatures on the Reinforcement Potential of Glass Fibers. *Polym Compos* 2016.
- [8] Thomason JL, Yang L. Temperature dependence of the interfacial shear strength in glass–fibre polypropylene composites. *Compos Sci Technol* 2011;71:1600–5.
- [9] Yang L, Thomason JL. Development and application of micromechanical techniques for characterising interfacial shear strength in fibre-thermoplastic composites. *Polym Test* 2012;31:895–903.
- [10] Yang L, Thomason JL. Effect of silane coupling agent on mechanical performance of glass fibre. *J Mater Sci* 2012;48:1947–54.
- [11] Turrión SG, Olmos D, González-Benito J. Complementary characterization by fluorescence and AFM of polyaminosiloxane glass fibers coatings. *Polym Test* 2005;24:301–8.
- [12] Fiore V, Di Bella G, Valenza A. Glass-basalt/epoxy hybrid composites for marine applications. *Mater Des* 2011;32:2091–9.