



CELLULAR AND FIBRE-REINFORCED COMPOSITES FOR BONE ENGINEERING

Manuel Bühler, Pierre-Etienne Bourban, Jan-Anders E. Månson
Laboratoire de Technologie des Composites et Polymères,
Ecole Polytechnique Fédérale de Lausanne (EPFL), 1015 Lausanne, Switzerland

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Abstract

Composites based on poly(lactic acid) (PLA) and glass fibre composites were successfully prepared. A specific processing method was developed for consolidated composites exhibiting different fibre concentration gradients. As the conditions to prepare PLA polymer foams with specific porosity morphology were known, this study focused on a fibre-reinforced foam to investigate on a wide range of fibre and porosity fractions. Micrographs confirmed that the distribution and impregnation of fibres could also be controlled at a microscale. Furthermore it was shown that porosity distribution is indeed influenced by the presence of continuous reinforcing fibres. The mechanical properties of such consolidated and cellular composites were determined by three-point bending and compression tests. The results revealed the reinforcing effect of continuous glass fibres. The studied composites exhibit morphology gradients similar to natural bones.

1 Introduction

A large variety of bioresorbable polymers are available. In the development of artificial bioresorbable tissue grafts for bone replacement focus has recently been put on different reinforcement types. Usually a poly(lactic acid) (PLA) foam is chosen as basic material, which may contain fillers such as hydroxyapatite, β -tricalcium phosphate or poly(glycolide) in order to increase the mechanical and biocompatibility performance [1, 2]. The foam porosity enables growth of the natural bone and a uniform bioresorption of the synthetic scaffold. As the mechanical properties of cortical

bone can not yet be achieved by using only fillers into polymers a more efficient porous composite structure was envisaged in this study. Reinforcements with high aspect ratios such as long or even continuous fibres could be of interest. Another appealing idea arising from the use of long fibres is the possibility to design gradient structures by precise placement of the fibres.

2 Materials and experimental

Bioresorbable PLA (Boehringer-Ingelheim GmbH, D; NatureWorks Ltd, USA) was used as the matrix throughout this study. A spinning line (Fourné GmbH, D) was used to process PLA yarns composed of 36 monofilaments, each with a diameter of 13 μm . The melt pressure was set to 30 bar, the melt temperature to 215 $^{\circ}\text{C}$. The rotational speeds of the three successive godets were 340 rpm, 600 rpm and 1864 rpm respectively. Produced fibres were used as the matrix material in the final composites.

The reinforcing glass fibres (DR0300 EC 300-350) were supplied by Glasseiden GmbH Oschatz, Germany.

A specially designed winding apparatus was used to place the fibres and to prepare composite preforms with different fibre distributions. This set-up consisted of a rotational device on which the polymer and glass fibres could be preconsolidated in situ to ensure that the relative fibres position is kept during the following processing steps. Figure 1 illustrates the complete winding apparatus with the mould for the preconsolidation. The rotational speed of the bobbins could be adjusted independently and thus the ratio of reinforcing fibres to matrix fibres could be tailored.

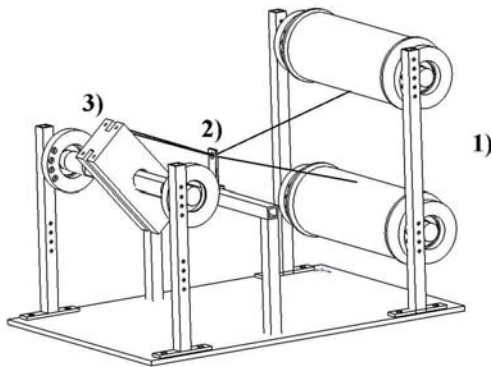
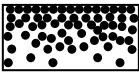
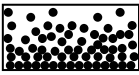
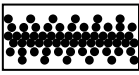
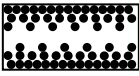


Fig. 1. Winding apparatus 1) bobbins of reinforcing and polymer fibres, 2) mobile fibre guiding device, 3) rotating device and mould for preconsolidation.

Subsequently this preform was vacuum dried to eliminate water, which at high temperatures would act as a degradation agent [3]. For moulded composite beams, the consolidation of the preforms was achieved within the rotational mould exposed to 163 °C and to a pressure of 5 bar. Table 1 summarizes the investigated geometries with the corresponding designation and variations of fibre fractions through the beam thickness. Four specimen types, each with a different fibre gradation were processed and then tested in flexion. The ASTM norm D 790M – 92 was used for the bending tests. The span width was 32 mm, the sample lengths were 50 mm, thicknesses were 3 mm and widths were 10 mm.

Table 1. Schematic representation of the beam sections with variations of fibre distribution and volume content.

Sample	Beam section	Fibre fraction [vol.%]
F:T		22 (6-42)
F:B		22 (42-6)
F:C		22 (4-40-4)
F:E		22 (30-5-30)

To mimick bone microstructure, composites with high porosity content were processed using a gas foaming method [4-5]. The foaming equipment was composed of a high-pressure chamber and a computerized acquisition data system. Dry glass fibres and PLA pellets or composite preforms made of mingled PLA and glass fibres were loaded in the pressure vessel. The foaming gas was CO₂, the saturation temperature 163°C. An initial depressurization rate of 13 bar/s, controlled by a backpressure regulator, a maximum cooling rate of 5°C/s, and a saturation pressure ranging from 85 to 200 bar were applied to manufacture composites with various pore size and pore distribution. The foaming direction and thus the cell orientation were parallel to the fibre orientation.

Cellular composites with homogeneous fibre distribution and with a combination of porosity and fibre gradient were processed. The specimens denominated as “52% P” and “82% P” contained porosities of 52% and 82% respectively and volume fibre fractions of 18% and 7%, respectively. For both specimens, the ratio of fibres to the volume polymer fraction was 61%.

Compression tests parallel to the direction of the reinforcing fibres were effectuated on cubic samples of 1 cm side length cut with razor blades from large composite samples. The influence of fibre content on the compression moduli of different cellular and fibre reinforced composites could thus be studied.

3 Results and Discussion

As described, the controlled winding of polymer and glass fibres enabled the creation of fibre gradient composites. In Figure 3 it is shown how the fibre volume fraction changed from 42 vol. % at the top to 6% at the bottom. After full consolidation an overall porosity of 0.5% was achieved.

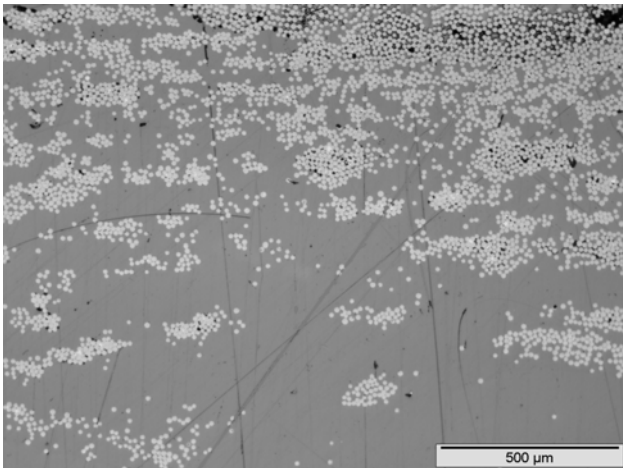
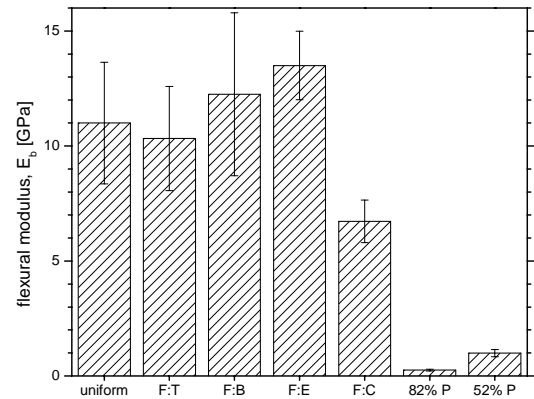


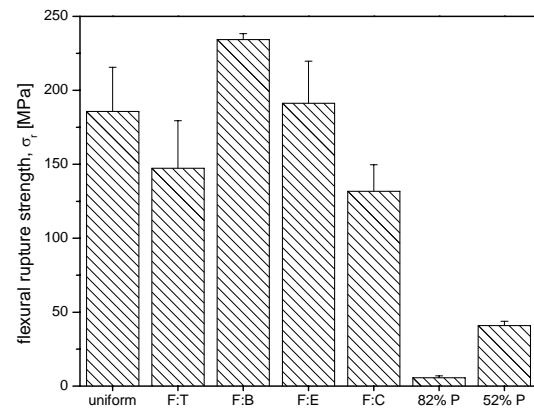
Fig. 2. Optical microscopy image of the cross section of sample with gradient F:T, fully consolidated.

Using three-point bending tests, the flexural modulus and the flexural failure strength of the beams were determined. The results are represented in Figure 3. Among the consolidated specimens (porosity of 0.5%) both properties were the smallest for the gradient composite F:C, whereas the composite F:E, with a flexural modulus of 13.5 GPa, showed the best performance because the reinforcing fibres were at a larger distance from the neutral axis of the beam. No significant difference was recognized for the other composite types, what is in accordance with other studies [6]. The flexural modulus of the composite beam with 52% of porosity was around 1 GPa decreasing to 0.26 GPa when the porosity reached 82%. Variations similar to those noted for the flexural modulus were also observed for the strengths.

It is interesting to notice that the moduli obtained for the consolidated composites are close the moduli of the cortical bone while the moduli of the porous or cellular composites is in the same range as the one of the trabecular bone.



a)



b)

Fig. 3. Flexural properties of PLA-GF gradient composites. a) flexural modulus, b) flexural strength.

Cellular composites were produced using either PLA pellets and neat glass fibres or PLA spun fibres mingled with glass fibres.

The Figure 4 depicts a scanning electron micrograph of a PLA-foam reinforced with continuous glass fibres. This composite foam was obtained with PLA pellets, full intra-fibre bundle impregnation did not really occur during the processing. Glass fibres were into cell walls but only partially impregnated.

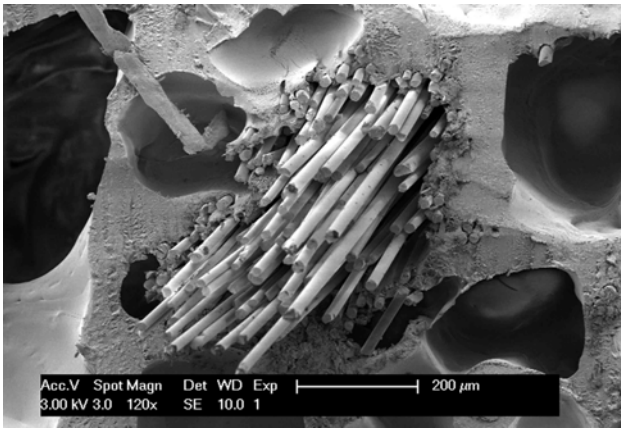


Fig. 4. Continuous glass fibre reinforcing PLA-foam when foaming PLA granulates around glass fibre bundles.

Fibre distribution and impregnation were much better when a composite preform made of spun PLA fibres mingled with glass fibres was used. In this case, the single fibres of the glass fibre bundles were impregnated and embedded in cell walls. Although there are regions with higher and lower fibre concentrations, the macroscopic distribution was homogenous. A section of a composite prepared by this second technique is illustrated in Figure 5.

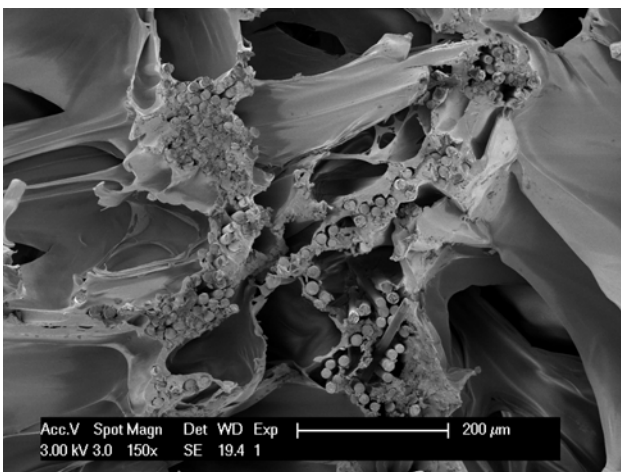


Fig. 5. Continuous glass fibre reinforcing PLA-foam, when foaming mingled PLA and glass fibres.

During the preparation of this second preform type, it was possible as well to build continuous composition changes in fibre fraction and porosity. Thus, the existence of abrupt variations of properties could be avoided. Several gradient composites were processed successfully. Figure 6 depicts one example of such a composite. Its volume fibre fraction increases from 0% in the internal part to

around 10% in the external parts, whereas its porosity decreases from 85% in the internal part to 65% in the external parts. From such composites samples were cut for three point bending and compression tests. Simply by changing the preform structure and composition it was possible to process other gradient composites.



Fig. 6. Composite with a gradient of porosity and fibre fraction.

As the presence of continuous fibres does not change the local viscosity of the molten polymer to be foamed, higher fibre volume fractions than in the case of short fibres or filled polymers could be achieved.

To estimate the influence of the fibre fraction on the composite stiffness, a relative compression modulus, E^* , was defined as the ratio of the compression modulus to the porosity. Figure 7 illustrates for example the increase in E^* observed when composites with about 75% porosity were reinforced with up to 10% of continuous fibres. Although the scatter was very large, a net increase of E^* with the fibre fraction was observed. Compared to PLA foams reinforced with ceramic particle (also with a porosity close to 80%) [1] the modulus was more than three times higher at fibre fractions of 5% for example. A combination of the two material types can be envisaged for the next generation of composite scaffolds for bone engineering. The process is also appealing for other cellular composites for applications in lightweight and packaging industry.

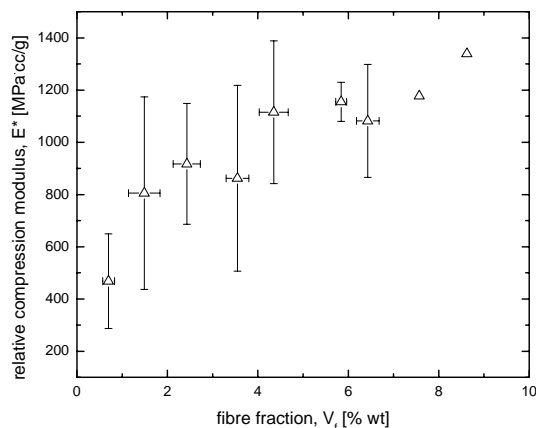


Fig. 7. Relative compression modulus versus fibre volume fraction for composites with approximately 75% porosity.

4 Conclusions

This study demonstrated that high porosity thermoplastic composites reinforced with continuous fibres can be processed successfully when adequate commingled yarns and gas foaming method are used. As the described technique allowed creation of property gradients throughout the composite, new designs for bone implants can be realised. PLA foams reinforced with continuous glass fibres were prepared with fibre content ranging from 0 to 10 % vol. and porosity varying from 45 % to 92 %. With up to 1.5 GPa these composites showed higher longitudinal compression modulus than previously suggested foams made of bioresorbable polymer materials. Indeed, the developed process can be used with biocompatible or bioresorbable materials to produce scaffolds for applications in bone tissue engineering. Cellular composite scaffolds with continuous fibres oriented in specific directions can be envisaged to tailor bending and torsion properties.

5 Acknowledgments

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6 References

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