Manufacturing Technology of 316L Stainless Steel/Poly(Lactic Acid) Composite Braids and the Induction of Hydroxyapatite Formation on the Braid

Ching-Wen Lou[1], Jin-Jia Hu[2]*, Yueh-Sheng Chen[3]*, Shih-Peng Wen[4], Keng-Chuan Lin[4] and Jia-Horng Lin[4,5,]*

[1]Institute of Biomedical Engineering and Material Science, Central Taiwan University of Science and Technology, Taichung 406, Taiwan, R.O.C.

[2]Department of Biomedical Engineering, National Cheng Kung University, Taiwan, R.O.C

[3]Laboratory of Biomaterials, School of Chinese Medicine, China Medical University, Taichung40402, Taiwan40402, R.O.C.

[4]Laboratory of Fiber Application and Manufacturing, Department of Fiber and Composite Materials, Feng Chia University, Taichung City 407, Taiwan, R.O.C.

[5]School of Chinese Medicine, China Medical University, Taichung 40402, Taiwan, R.O.C.

*corresponding email: [a]jjhu@mail.ncku.edu.tw, [b]yuehsc@mail.cmu.edu.tw, [c]jhlin@fcu.edu.tw

Keywords: Poly(lactic acid), 316L stainless steel, Braiding machine, Electrochemical methods

Abstract.

Many biodegradable synthetic polymers have been used as tissue-engineered scaffolds. The major problem of these polymers to be used in bone tissue engineering is their poor mechanical strength. It is well known that we can deposit hydroxyapatite, a material with strong osteoconductivity, onto a surface using electrochemical methods. These polymers, again, lack electrical conductivity so that deposition of hydroxyapatite onto these polymers is very challenging, if not impossible. Here we presented a novel scaffold for bone tissue engineering based on textile technology. First, we fabricated 316L stainless steel/poly(lactic acid) composite ply yarn by wrapping stainless steel wires and poly(lactic acid) yarn together. A 16-spindle braiding machine was then used to braid the composite yarn layer by layer into a 3-dimensional scaffold for bone tissue engineering. Furthermore, due to the electrical conductivity of 316L stainless steel wires in the composite yarn, we employed an electrochemical method to induce hydroxyapatite deposition on the braid. SEM was used to evaluate the growth of hydroxyapatite formation on the braid.

Introduction

In 1821, Pierre Berthier recognized the corrosion resistance of iron-chromium alloys. Not until 1926, however, was 18Cr-8Ni stainless steel introduced into surgical applications. Two types (19Cr-9Ni and 18Cr-8Ni) of stainless steels were first proposed to use as implants by the American College of Surgeons in 1947. Despite of its long history, stainless steel is still one of the most important metals in biomedical applications. At present the majority of artificial bones is made of metal. Because of the extreme stiffness of the metal compared to the bone, the loading will be distributed unevenly between the metal and the bone after the metal is implanted; the metal will bear most of the loading. The phenomena is the so-called stress shielding effects. Without physiological loading, however, the bone will undergo demineralization, causing the distability of the implant and in the worst case leading to the failure of the graft. Recently, biodegradable polymers have attracted more and more attention along with the development of advanced polymers. These polymers have getting more and more usage in artificial bone scaffolds.[1-3] Poly(lactic acid) (PLA) is a biodegradable polymer with biocompatibility and mechanical properties and can be machined easily. PLA has been used in orthopaedic treatments. In the body, PLA degrades into lactic acids, which is one of the metabolites in the body, and water. Many biomaterials in the form of fibers have been developed and used in many biomedical application, such as wound dressing and bone scaffod. [4-6] In this study, PLA fibers were twisted with 316L stainless steel wires and the resulting yarn was used to fabricate a 3-dimensional bone scaffold using a 16-spindle braiding machine. The stainless steel not only enhances the mechanical strength of the yarn but also makes it electrically conductive so that hydroxyapatite can be induced to deposit on the scaffold via a electrochemical method. The PLA, on the other hand, reduces the mechanical strength of the yarn, and hence reduces the effects of stress shielding.

Materials and Methods

Materials : Polylactic acid (PLA) ply filaments (maximum tensile strength 3.9 g/D, elongation 30 %, UNITIKA LTD., Japan), 316L stainless steel fiber (diameter: 0.06 mm, YUEN NENG co., LTD., Taiwan); Equipments : Doubler, rotor-twister, Textechno statimat, woof machine, 16-spindle braiding machine, Computer Servo Control Materials Testing System (HT-9101).

$$T.P.I = R/T \times D \times \pi \text{ , } T.P.I = \alpha_e \times \sqrt{Ne} \quad (1)$$

where T.P.I is the twist per inch, R is the rotation speed of the rotor (rpm), T is the rotation speed of the take-up roller (rpm), D is the diameter of take-up roller, α_e is the coefficient of twist, and Ne is count.[7]

Our preliminary experiments showed that 225-denier PLA filaments have the optimal properties for further processing. Thus as the first step, we combined three 75-denier PLA filaments by a doubler to make 225-denier PLA filaments. The 225-denier PLA filaments were then twisted by a rotor twister (12000 rpm with coefficients of twist=2, 3, and 4) and preset in an oven (100 ℃) to consolidate the structure. The heat-preset yarns re divided into two groups: in one group the yarns were treated with 1M NaOH and in the other group the yarns were not subjected to 1M NaOH. Maximum tension strength tests and break elongataion tests were performed on yarns from each group according to ASTM D2256 standard.

The braid was then fabricated using a 16-spindle braiding machine, onto which the 316L stainless steel/ PLA ply yarn was loaded. The gear ratio was changed to obtain braids with different braid angles. With an increase of the number of teeth of the take-up gear, the rolling speed of sample becomes slower and the structure of the resulting braid becomes tighter. To investigate the effects of braid angles on the stability of the braid structure, varying ratios of teeth of the take-up gear and braid gear (100/50, 90/50, 80/50, 70/50, and 60/50) were set during braiding. The maximum tention strength tests were then performed using Computer Servo Control Materials Testing System (HT-9101) according to STP35359S braids standard (sample size: 80 mm×4 mm×3 mm). After testing, the best braiding parameters were determined.

After mechanical testing, the composite braids were placed in 0.4 wt% $Ca(OH)_2$ solution for 30 min to allow calcium ion grafted on the braids, which would facilatate the following electrochemical process. The braids were then subjected to the electrochemical process, during which hydroxyapatite was deposited onto the braids. We investigated the effects of electrical currents (100, 200, 300, 400, and 500 mA) on the hydroxyapatite deposition, which was examined by SEM.

Results and Discussion

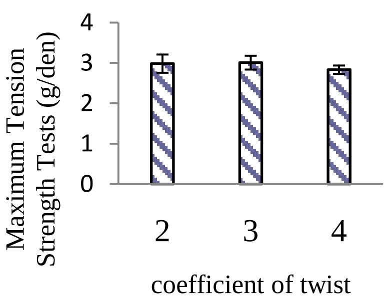

Fig. 1Maximum tension strength Tests of PLA composite ply yarn for rotor spin device 12000 r.p.m with different coefficient of twist

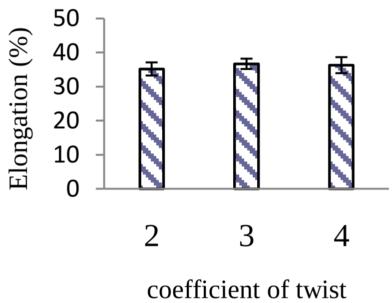

Fig. 2 Elongation of PLA composite ply yarn for rotor spin device 12000 r.p.m. with different coefficient of twist.

Figure 1 showed no obvious change in maxmum tension strength with dfferent coefficients of twist. But the tension strength appeared to slightly decrease when coefficient of twist is 4. In general, torsion force increases as the coefficient of twist increases. When coefficient of twist reaches the limit, however, the extreme torsion force may break down part of the fibers in the yarn. Fig. 2 showed that elongation slightly increased as the coefficient of twist increased. This may be due to fibre cohesion caused by twisting. In order to wash away oiling agent on PLA ply yarn, we immersed PLA ply yarn in NaOH solution. But we do not want the treatment to destroy fibers in PLA ply yarn. With this in mind, the optimal concentration of NaOH solution was investigated. Preliminary experiments showed that the concentrations of the NaOH solution (0.5 M, 1 M, 1.5 M, and 2 M) have no influence on the mechanical properties of yarns. While 0.5 NaOH solution may not be able to remove the oiling agent, there would be more residual NaOH left on PLA ply yarn in the case of high NaOH treatment. We decided to use 1M NaOH and tested the influence of immersion time (10, 15, and 20 min) on the maxmum tension strength of the PLA ply yarn. Figure 3 showed that no obvious effects of 1 NaOH immersion on the mechanical properties of the yarn in comparison with Figure 1. Figure 3 showed as coefficient of twist increased, maxmum tension strength of the PLA ply yarn slightly decreased. This may be due to the torsion force during twisting that destroys part of fibers in PLA ply yarn. Immersion of NaOH may slightly expand the PLA ply yarn, causing maxmum tension strength of the yarn to increase. Figure 4 showed that after NaOH treatment, the maximum elongation of PLA ply yarn increased as coefficient of twist increased. We decided to use coefficient of twist of 3 to twist the filaments and then remove oiling agent by immersing PLA ply yarn in 1 M NaOH solution for 20 min for subsequent experiments as the processing conditions give the best results. Note that coefficient of twist of 3 did not generate the maxmum tension strength but it created a stable structure and reliable maxmum tension strength compared to coefficient of twist of 2 and 4.

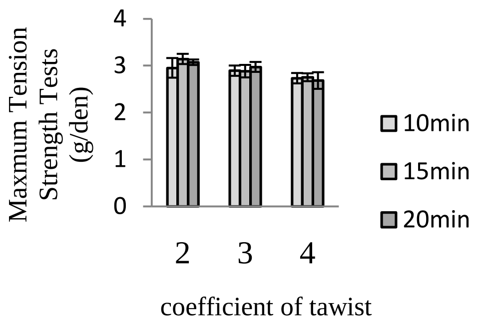

Fig. 3 Maximum tension strength Tests of PLA composite ply yarn for immerse NaOH solution with different coefficient of twist.

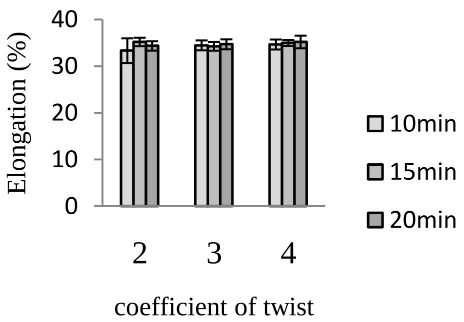

Fig. 4 Elongation of PLA composite ply yarn for immerse NaOH solution with different coefficient of twist.

Table. 1 different gear ratio for braid angle

gear ratio	braid angle 1	braid angle 2	braid angle 3	braid angle 4	average value
100:50	56.63	58.81	58.00	55.94	57.34 ±2.60
90:50	51.75	51.63	53.25	54.75	52.84 ±2.94
80:50	49.63	51.88	49.94	48.31	49.94 ±2.94
70:50	49.13	50.25	49.19	43.31	47.97 ±6.30
60:50	42.69	45.00	39.25	44.31	42.81 ±5.13

The prelimineary experiments showed that the tension strength of the PLA ply yarn is 2.97g/denier.We changed gear ratio of take-up gear and braid gear to obtain a series of 316L stainless steel/ PLA composite braids. The braid angle of the braid was examined by a stereomicroscope (OLYMPUS B061). We found that the braid angle decreased as the number of teeth of take-up gear decreased. The result was expected as the number of teeth of take-up gear decreases the formed braid would move upward with a higher speed reducing the braid angle. Table 1 showed different gear ratio and the corresponding braid angle.

We futher tested the maxmum tension strength of 316L stainless steel/ PLA composite braids with using the Computer Servo Control Materials Testing System. Figure 5 showed that the maxmum tension strength of 316L stainless steel/ PLA composite braids increased as take-up gear decreased and peaked at the gear ratio of 70:50.

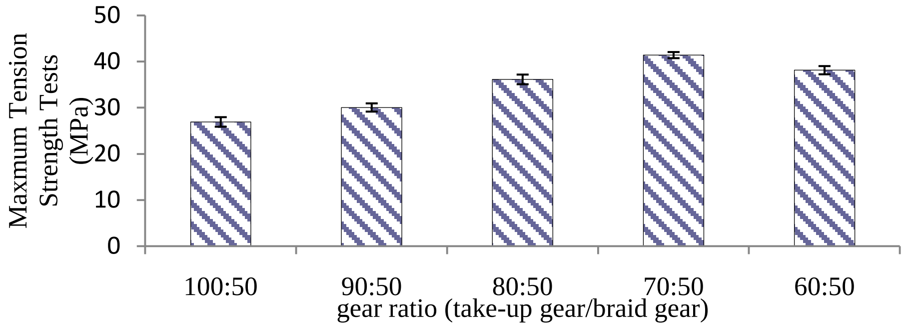

Fig. 5 Maxmum tension strength of 316L stainless steel/ PLA composite braids with different gear ratio.

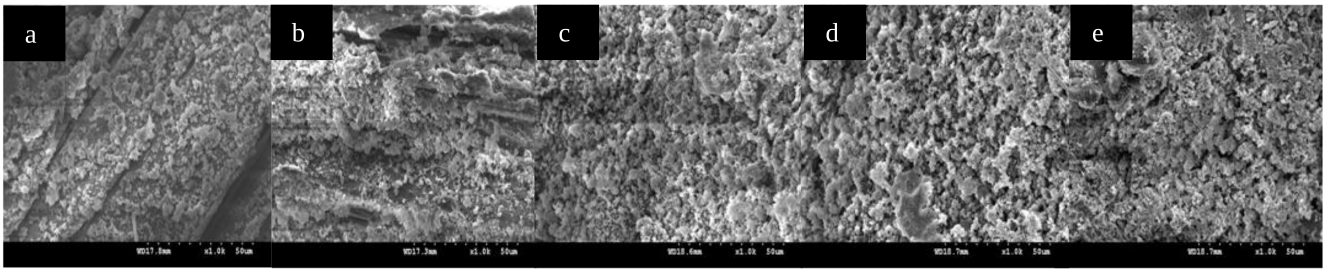

Fig. 6 Growth of hydroxyapatite formation on the braid by electrochemical methods for 60 min (a: 100 mA, b: 200 mA, c: 300 mA, d: 400 mA, e: 500 mA) 1000X.

To evaluate effects of electric current on the deposition of hydroxyapatite on the braids, we subjected the composite braid to a series of electric currents of 100 mA, 200 mA, 300 mA, 400 mA, and 500 mA for 60 min.[8, 9] Figure 6(a)(b) showed that hydroxyapatite deposition was incomplete in the cases of 100 mA and 200 mA. This is because of the insufficient electrons that participate in the reaction. However, in the case of 300 mA, the deposited hydroxyapatite almost covered the surface of composite braid (see Figure 6(c)). Figure 6(d)(e) illustrated that in the cases of 400 mA and 500 mA, the crystals of hydroxyapatite started to aggregate in the forms of blocks, which may increase the surface area of hydroxyapatite. We found that the weight of the composite braid decreased if the electric current exceeded 300 mA; the fibers in the composite yarn may be damaged at high current.

Conclusion

In this study, manufacturing technology of 316L stainless steel/PLA composite braids was developed. The ability of deposition of hydroxyapatite on the composite braid was demonstrated.

Acknowledgement

This work would especially like to thank National Science Council of the Republic of China, Taiwan, for financially supporting this research under Contract NSC 99-2622-E-039-004-CC3.

References

[1] C.C. Huang, C.W. Lou, C T. Lu, S.H. Huang, C.Y. Chao and J.H. Lin: Advanced Materials Research Vol. 123-125 (2010), p. 975-978.

[2] C.C. Huang, C.T. Lu, C.Y. Chao, T.C. Huang, C.W. Lou and J.H. Lin: Advanced Materials Research Vol. 97-101 (2010), p. 2249-2252.

[3] M.C. Lu, Y.T. Huang, J.H. Lin, C.H. Yao, C.W. Lou, C.C. Tsai and Y. S. Chen: J Mater Sci-Mater M Vol. 20 (2009), p. 1175-1180

[4] C.Y. Chao, C.W. Lou, H.W. Sung, Y.S. Chen and J.H. Lin: Advanced Materials Research Vol. 123-125 (2010), p. 979-982.

[5] C.W. Lou, C.W. Lin, Y.S. Chen, C.H. Yao, Z.S. Lin, C.Y. Chao and J.H. Lin: Text Res J Vol. 78 (2008), p. 248-253.

[6] J.H. Lin, C.H. Yao, Y.S. Chen, K.C. Yen, C.Y. Chao, C.W. Lou: Advanced Materials Research Vol.97-101 (2010), p.2253-2256.

[7] J. H. Lin, I. S. Tsai, and W. H. Hsing: The Journal of The Textile InstituteVol.89 (1988), p.266-273.

[8] W.E. Teo, S. Liao, C. Chan and S. Ramakrishna: Acta Biomater Vol.7 (2011), p. 193-202.

[9] Q. Yuan and T. D. Golden: Thin Solid Films Vol.518 (2009), p. 55-60.