Study on the microstructure and binding mechanisms of Selective laser sintered wood plastic composite

Kaiyi Jiang[1,2] ,Yanling Guo[1*], David L. Bourell[2*]
[1.] College of Mechatronics Engineering, Northeast Forestry University, Harbin 150040, P.R.CHINA
[2.] Laboratory for Freeform Fabrication, The University of Texas at Austin, TX 78712-0292, USA

Accepted August 16th 2013

Abstract

A new type of wood plastic composite, Eucalyptus/PES(Polyethersulfone) blend, was developed. It was designed to be used in LS (Laser Sintering) with reduced energy usage with cost savings. The preparation of Eucalyptus/PES blend and part sintering were under fixed processing conditions and parameters. Through mechanical testing, it was discovered that the strength of prototypes is low due to the formation of a segregated structure in the composite and a weak wood fiber–plastic particle interface.

The specific focus of this paper was on investigating the microstructure and binding mechanisms between these two materials. Combining SEM technology and infrared spectrum analysis, the interface binding form of the wood fiber and PES was confirmed to be mechanical interlock. Also the size and distribution of wood fiber in the PES matrix, which can affect the binding and strengthening mechanism, were analyzed during sintering and crack extension.

Introduction

Laser Sintering (LS) is an Additive Manufacturing technology, which was defined as a process of joining materials to make objects from 3D model data. This is usually done layer on layer as opposed to subtractive manufacturing methodologies (ASTM F2792-09, 2009) by international standards. LS not only allows rapid product development and production and variety of material choices, but ensures a reduction of the time to market using geometries and structures impossible to be obtained by traditional processing[1][2]. Most applications of LS are concentrated in industry and research fields because of the high cost. More and more people are becoming concerned about energy usage and environmental issues[3]. So in this project, a biological and sustainable wood plastic composite was developed to be used in LS. It is a cheap composite material made of wood powder, plastic and other additives[4]. Using this material can reduce the production cost and make the prototype with a natural look.

Generally, prototypes fabricated by LS are one piece, end-use parts, so the structure and properties should be considered according to their applications. The mechanical properties of manufactured parts depend on the shape and distribution of unit-cells, the porosity of the structure and the binding mechanism between particles[5][6]. Previous works focused on the development of the material, sintering experiments, post-processing for improving mechanical properties and the application in various industry fields (e.g. investment casting)[7].

This work investigated the binding mechanism and microstructure as it affected the mechanical properties of Eucalyptus/PES specimens manufactured by LS. The influence of material, mixture composition and microstructure on the mechanical properties of the prototypes will be discussed.

Experiment and Procedures

1. Material

Wood flour used is 300 micron mesh eucalyptus powder of De Long new materials Co., Ltd in Jiangxing City, China. The hot melt adhesive used is 120-180 micron mesh PES-Y1201P80 manufactured by Shanghai Yuanzhi Hot Melt Adhesive Co. Ltd. The hot melt adhesive is a thermoplastic copolyester with good fluidity in liquid phase. It has low hygroscopicity and shrinkage rate. It also has a high heat resistance and shock strength. Upon heating it melts and is able to wet the wood powder to achieve the desired bonding to form the part. Additionally, some other components can be also added into the composite such as a viscosity breaker to reduce the viscosity and disaggregate the wood powder. Initiator and coupling agents enhance the bonding of eucalyptus and PES. Light stabilizers as well as other organic fillers improve the molding appearance and durability.

Weight 1kg of Eucalyptus and 4kg of PES using a electronic scale(AND FW-10KA2), respectively. Put the powder into the ceramic grinding jars of the U.S. Stoneware 803 DVM Long Roll Jar Mill(Motor H.P. is 1.) with small ceramic grinding media as they can offer high purity grinding and reduce grind time. Mix up the powder for 5 hours. Take the homogeneous mixing powder out of the jars, then the Eucalyptus/PES blend was prepared.

2. Laser Sintering (LS)

Specimens of Eucalyptus/PES mixtures in the ratio of 1:4 were produced by using a Sinterstation 2000® . Processing parameters were 35W CO_2 laser with a scan speed of 2000 mm/s, scanning space of 0.15 mm, layer thickness of 0.1~0.2 mm and the powder bed temperature of 70ºC.

3. Mechanical analysis

A Regear computer controlled Universal Testing Machine (Shenzhen Regear Instrument Cooperation, China) was used for mechanical property testing.

Tensile strength: The dog-bone-shaped tensile specimens having a typical dimension of 165 ×13 × 4 mm, were measured according to ASTM D638-2004. A crosshead speed of 5 mm/min and a gage length of 50 mm were used for the test. The results of the testing showed the tensile strength to be 0.220 MPa.

Flexural strength: Specimens measuring 80 × 13 × 4 mm were measured under three-point bending using the same Universal Testing Machine in accordance with ASTM D790-2004. A crosshead speed of 1.9 mm/min and a span length of 64 mm were used for the test.. The result of the testing showed the flexural strength to be 0.360 MPa.

4. FTIR spectra analysis

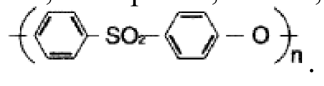
FTIR spectroscopy was used to measure the components of wood powder/PES blend and of the sintered part, respectively. By comparing the two spectrograms, the states of materials were investigated. Furthermore, the changing mechanism (which is chemical or physical) of the material can be resolved.

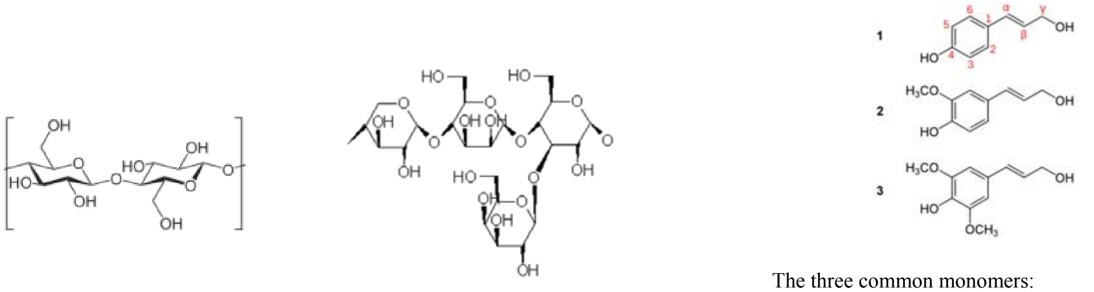
5. Scanning Electron Microscopy (SEM)

The microstructures of the surface of the wood powder/PES specimens in Y-direction and the cross-section of the fractured parts in Z-direction were observed under SEM to investigate the binding mechanism, the fracture surface, particle features and microstructure. The specimens needed to be coated with gold, because the material is non-conductive.

Results and Discussion

1. Materials Analysis with FTIR

PES is a heat-resistant, transparent, amber, non-crystalline engineering plastic having the molecular structure of . Wood is a natural composite material consisting of cellulose, hemicelluloses and lignin. The molecular structures are shown in Fig. 1(a)-(c)[8].



-Xylose-β(1,4)-Mannose- β(1,4)-Glucose-alpha(1,3)-Galactose

The three common monomers:
1,paracoumaryl alcohol ; 2,coniferyl alcohol;
3, sinapyl alcohol

(a) Cellulose (b) Hemicelluloses (c) Lignin

Fig.1 molecular structures of the components in Wood

Fig. 2 presents FTIR spectra of Eucalyptus/PES before and after LS, the ascriptions of characteristic peaks are shown in Table 1.

Results show that the position and the shape of the peaks of the blends before and after LS are basically the same. There is no peak emerging or disappearing. In spite of slight changes in absorption strength of some peaks, which may be caused by the different distribution of the components in the samples, it can be possibly concluded from the FTIR that under LS, Eucalyptus and PES rarely produce new chemical bonds or new material to form a firm connection between them. So the changing processes of the materials during sintering can be considered as physical changes.

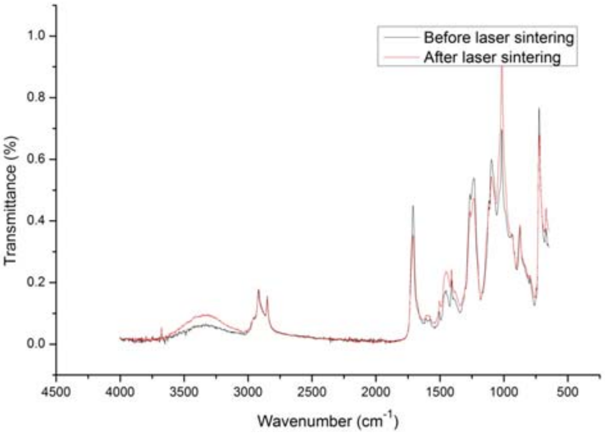


Fig.2 Eucalyptus/PES blend FTIR before and after Laser Sintering

Tab.1 Ascriptions of characteristic peaks

Wavenumber	characteristic group and vibration mode
3250-3500	-O-H stretching
2750-3000	Aliphatic C-H stretching
1700-1800	&=O of Hemicelluloses stretching
1550-1600	C=C of aromatic benzene ring twisting
1299-1323	O=S=O antisymmetric stretching
1200-1300	Aromatic =C-O-C stretching
1100	O=S=O symmetric stretching
1025-1000	-S- stretching Accompanying aromatic =C-O-C stretching
880-860	C-H in benzene ring twisting
730-700	C-S stretching

2. Microstructure observation and analysis

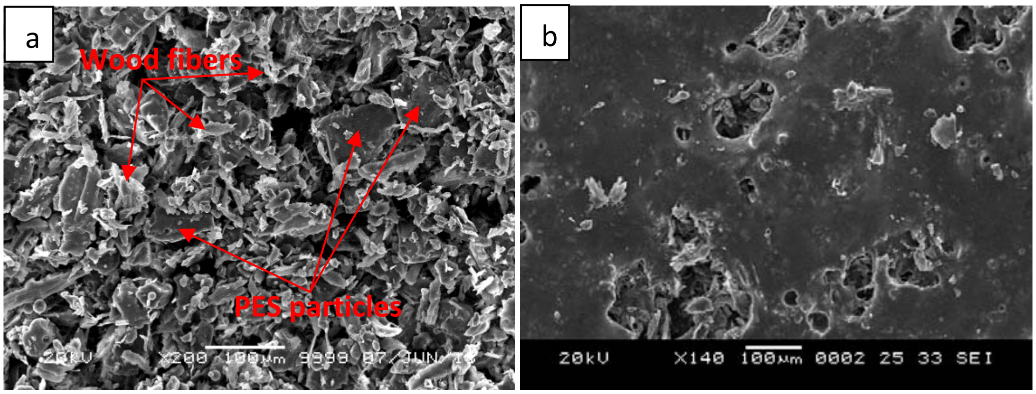
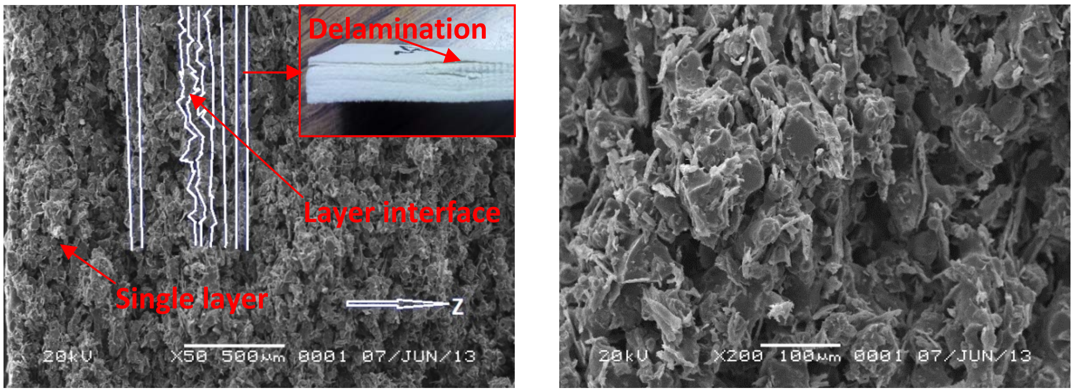


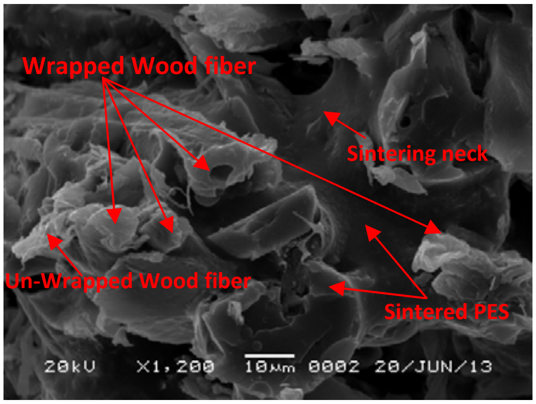
Fig. 3 Micrographs for : (a) Eucalyptus/PES blend, (b) Surface of the fractured bar (X130)

The microstructure important for the mechanical properties of Eucalyptus/PES parts was observed in thin section. Figs. 3a and 3b and Fig.4 are the Eucalyptus/PES blend, surface and the cross-section micrographs of the fractured bar, respectively.

Evenly distributed Eucalyptus and PES with average size of 40 µm and 85 µm, respectively, have irregular particle shape as shown in Fig.3a, micrograph of the blend. With the processing parameters used in LS, PES bonded part of the wood fiber, joined by extensive co-continuous phase formation. The surface is relatively smooth and some bonded wood fiber is partly exposed on the surface. While there are also some pores of different sizes existing in the surface, the micrograph shows there are some wood fibers encapsulated in the pores which adhered to the PES partly. These pores were formed under the moderate viscous flow of PES and the block of the wood fiber during sintering.



(a) Magnified by 50 (b) Magnified by 200



(c) Magnified by 1200

Fig. 4 Cross-section micrographs of the fractured bar

Fig.4a shows a compact cross-section. The slices typical of layer manufacturing can be observed in this photo. However, among the connections between layers, some have clear and straight edges, which will result in delamination as shown in Fig.4a. Others have fuzzy and uneven margin which will have a stronger bonding force with the adjacent layers to improve the whole part's mechanical properties.

Figs. 4b and 4c show the micrographs of the fractured bar cross-section magnified by 200 and 1200 times. In the microstructure of the Eucalyptus/PES specimen, part of the wood fiber is wetted and bonded by PES as shown in Fig.4c, while another formation of PES co-continuous phase occurred, with a number of wood fibers adhered to it. This indicates the insufficient melt flow of PES under the process LS. Also the existence of wood fiber domains encapsulated in regions of PES coalescence and the interconnected pores demonstrated the low wettability and interaction between PES and Eucalyptus fiber. Actually the main reason resulting in low attraction force and immiscibility is the two components in the blend are polarity-reversed.

3. Interfacial Bonding

As composites, the interfaces between the components, which are formed based on different binding mechanisms, are important to the strength of parts[9]. For the Eucalyptus/PES blend, the interfacial bonding is due to adhesion between the Eucalyptus as reinforcement and the PES as the matrix. During laser sintering, PES is often in a condition by which it is capable of flowing. Its behavior approximates that of a liquid. It can then spread over the wood fiber flowing over the rough surface. This then can be defined as wettability.

Once PES has wet wood fiber, it is therefore in intimate contact with the wood fiber. In this condition, bonding occurs. Due to the shrinking of the plastic and the difference in elasticity modulus between the components, there are interfacial stresses in composites. Therefore, an efficient binding mechanism is needed to transfer the stress, for the purpose of obtaining a part with good strength. In other words, the binding mechanism is the essence of the mechanical properties of prototypes.

In this research, by comparing the two FITR curves of blends before and after laser sintering, and microscopic analysis, it can be concluded that the main interfacial bonding between the wood fiber and PES particulate is mechanical interlocking.

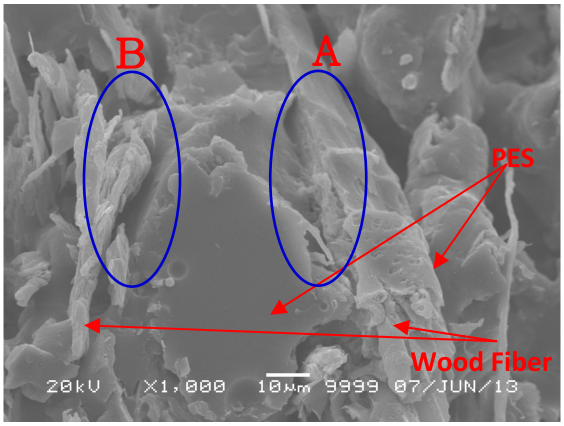


Fig. 5 Interfacial microstructure

Mechanical interlocking[9] is a kind of linkage phenomenon that occurs under laser sintering. PES, wetting into and spreading over the wood fiber which is porous, produces a structure after hardening which can also be seen as mechanical anchoring. Actually, the adherence between PES and wood fiber is accomplished by the interpenetration of the two components in a mechanical form, shown as area A in Fig. 5. Then, the PES outside the fibers would bond with other wrapped fibers producing the prototype. The wood particle is continuous but variously shaped rough fiber, whose adherence to PES is proportional to its surface area. From this point, it is clear that the rougher the interface, the greater is the interlocking. However, the 'rugosity' is a positive factor only insofar as the reinforcement such as wood fiber is perfectly wet by the matrix just like vitreous PES. As a matter of fact, if the vitreous PES cannot penetrate into the asperities of the Eucalyptus fiber, the wood particle would become a barrier blocking the PES bonding. The hardening of the resin is accompanied by the formation of interfacial cavities which are liable to initiate the failure of the interfacial bond.

Conclusions

A type of low-cost and sustainable wood plastic composite, Eucalyptus/PES, was used in LS in this research. Specimens were produced to be used for mechanical testing. However the results showed the sintered part exhibited a low mechanical strength. Therefore the experimental plan provided a specific focus on the essential factors influencing mechanical properties of the Eucalyptus/PES parts fabricated by LS.

This research combined FTIR and SEM technologies. The original microstructure and bonding mechanism of the prototype was determined. It not only explained the low mechanical properties of the Eucalyptus/PES part, but also gave direction for follow-on research to improve the strength.

FTIR of the wood plastic composite before and after LS proved that there was rarely new material or chemical bond produced by laser scanning of these two components. This indicated that the whole bonding is physical. After being melt by the laser, molten PES can spread over the wood fiber. Then the fiber became partly wet and bonded. Furthermore, it is conceivable that adding some specific additives for building a 'bridge' between wood powder and plastic by chemical bonding can improve the mechanical properties.

Through SEM the microstructure of the sintered part was observed. Micrographs showed PES partially wet and bonded the wood fibers. There were many relatively large co-continuous phases of PES with some wood fiber adhering to the outside. Also there were some interconnected pores with some wood fiber encapsulated in the microstructure showing its porosity. After FTIR and SEM were performed, the bonding mechanism of Eucalyptus and PES were confirmed as mechanical interlocking. Also it was seen that the rheologic behavior of PES during the processing played a very important role in the blended microstructure formation.

In conclusion, the manufacturing of Eucalyptus/PES blend using a laser sintering process demonstrated that it is possible to use a low-cost and sustainable wood plastic composite in additive manufacturing to produce parts with the appearance of wood. The performance of parts produced through LS confirm a strong dependence on the microstructure, the bonding mechanism and on the powder characteristics.

Acknowledgments

This work was supported by National Natural Science Foundation of China (51075067), China Scholarship Council, Natural Science Foundation of Heilongjiang Province of China (E201050), Educational Commission of Heilongjiang Province of China (12511145).

References

[1] Bernard, A., Taillandier, G. and Karunakaran, K.P. (2009), Evolutions of rapid product development with rapid manufacturing: concepts and application", International Journal of rapid Manufacturing, Vol. 1 No.1 PP. 3-18

[2] J.J. BEAMAN, J.W. BARLOW, D.L. BOURELL, et.al. (1997), Solid Freeform Fabrication: A New Direction in Manufacturing, Kluwer Academic Publishers Press, Boston, MA

[3] R. Sreenivasan, A. Goel, D.L. BOURELL (2010), Sustainability issues in Laser-based Additive Manufacturing, Physics Procedia, Vol.5, partA, PP. 81-90

[4] Guo Yan-ling, Jiang Kai-Yi, Yu Zhi-Xiang, et al (2012), The preparation technology and forming properties of wood-plastic composite powder used in selective laser sintering, Journal of Shanghai Jiaotong University, Vol. 45 No. 9, pp. 1327-1331

[5] J-P. Kruth, P. Mercelis and J. Van Vaerenbergh, (2005), Binding Mechanisms in Selective Laser Melting, Rapid Prototyping Journal, Vol. 11, No. 1, pp. 26-36

[6] Elena Bassoli and Andrea Gatto (2012), Joining mechanisms and Mechanical Properties of PA Composites obtained by Selective Laser Sintering, Rapid Prototyping Journal, Vol. 18, No. 2, pp. 100-108

[7] K. Jiang, Y. Guo, D.L. Bourell (2013), Study on Selective Laser Sintering of Eucalyptus/PES Blend and Investment Casting Technology, Procedia CIRP, Vol. 6, pp. 511-515

[8]http://en.wikipedia.org/wiki/Cellulose,http://en.wikipedia.org/wiki/Hemicelluloses, http://en.wikipedia.org/wiki/Lignin

[9] Soo-Jin Park and Min-Kang Seo (2011), Interface Science and Composites, Academic Press, Great Britain