



Thermal Treatment Effect on Transcrystalline Interphase and Interfacial Shear Strengths of Glass Fibre/Polypropylene Composites

Yin Li¹, Qunfang Lin², and Xiaodong Zhou^{1*}

(1) State Key Laboratory of Chemical Engineering; (2) School of Materials Science and Engineering, East China University of Science and Technology, Shanghai-200237, P.R. China

Received 24 July 2010; accepted 28 February 2011

ABSTRACT

To investigate the influence of thermal treatment and interfacial crystalline structure on interfacial shear strength of glass fibre reinforced polypropylene composites (GF/PP), the transcrystalline samples were prepared by isothermal crystallization at 135°C, while the quenched samples without transcrystallization were obtained by quick cooling at the rate of 20°C/min. In the next step, the transcrystalline and the quenched specimens were each treated at various temperatures of 70°C, 100°C and 130°C, and the interfacial shear strength (IFSS) of these specimens was measured by the microbond test. The results demonstrated that, before thermal treatment, the interfacial shear strength of the transcrystalline specimen is much smaller than the quenched composite specimen. The shear debonding of the trans-crystalline specimen occurred at the rim where the transcrystalline region met the spherulites, because the shear debonding of the quenched specimen happened at the PP and glass fibres interfaces. Whereas, after thermal treatment the shear debonding of the transcrystalline specimens, as same as quenched specimens, occurred at the interfaces of PP and glass fibres. The interfacial shear strength (IFSS) of the trans-crystalline specimen also increased the same as that of the quenched cases. Therefore, we conclude that transcrystallinity alters the failure location at interfacial region and thus it affects the interfacial shear strength. Both the interfacial shear strength of the transcrystalline and that of the quenched samples can be improved by thermal treatment.

Key Words:

glass fibre;
polypropylene;
interfacial shear strength;
transcrystallization;
thermal treatment.

INTRODUCTION

When glass fibres are embedded in a semicrystalline thermoplastic matrix melt, they may act as nuclei for the growth of spherulites. If there are many nucleation sites along fibre surface, the resulting spherulite growth would be restricted in the lateral direction and a columnar layer, known as “transcrystallinity”, would develop and enclose the fibre [1-4].

The strong orientation of the molecules in the transcrystalline layer in glass fibre/polypropylene composites affects the mechanical properties of the composite materials by altering the interfacial structure and as a result it is the centre of much attention [5-10]. Moon [9] reported that the transcrystallinity formed at the interface of GF/PP improves the

(*) To whom correspondence to be addressed.

E-mail: xdzhou@ecust.edu.cn

interfacial strength when no spherulites develop in the PP matrix, while the transcrystallinity reduces the interfacial strength when the spherulites are well developed. Further on, Moon considered that transcrystallinity may decrease the interfacial shear strength by pull-out tests [9].

The influence of transcrystalline layers on the fibre-matrix bond strength has been widely discussed in the literature [11,12]. Experimental studies on single fibre/polypropylene (PP) composites by the fragmentation test have shown that transcrystallinity increases the efficiency of stress transfer at the fibre-matrix interface [5-8], thus increasing the interfacial shear strength.

Many researchers have also found an increase in radial stress [13] as a result of the anisotropic nature of the transcrystalline interlayer [14,15] and a corresponding increase in interfacial shear strength of the transcrystalline interlayer [5-8]. Based on X-ray diffraction [16-19] and nano-mechanical testing [14,15], it has been suggested that twisting of the lamellae occurs in the transcrystalline interlayer by moving away from the fibre axis, thus giving rise to a gradient of mechanical properties throughout the transcrystalline interlayer. Zhang et al. [20] found that some residual PP adheres to the glass fibre after debonding of the transcrystalline sample.

Thermal treatment is an effective way to eliminate thermal stress which affects the crystalline behaviours and thus, alter the mechanical properties of the composites [21,22]. It has been found that the GF/PP interfacial crystalline structure, such as alpha versus beta crystal form and the orientation of the crystals are the major factors affecting the interfacial shear strength (IFSS). Thermal treatment conditions, such as temperature and time could also play very important roles in determining the interfacial shear strength of GF/PP [9,23].

There has not yet been research report on the relationship between the thermal treatment and failure mode in the form of interfacial debonding with the existence of transcrystallinity and consequently the interfacial shear strength. In this work, we treated the transcrystalline and the quenched samples at various temperatures of 70°C, 100°C and 130°C, and observed the failure location as interfacial debonding. Furthermore, we measured the interfacial shear

strength (IFSS). The IFSS is frequently characterized via a single fibre pull-out test, although many other techniques such as fragmentation [24] and micro-compression [25] have also been used.

Pull-out tests exist in several different geometries [24, 26-28]. The method developed by Miller et al. [28] is named as the "microbond test" and used in the present work due to its suitability for glass fibres [29,30] to measure the IFSS and thus to determine the effect of transcrystallization on the IFSS via debonding in GF/PP composites before and after thermal treatment [31]. Subsequently, the influence of thermal treatment on the interfacial microstructure was investigated.

EXPERIMENTAL

Materials

The isotactic polypropylene (PP Y1600) was produced by Shanghai Petrochemical Co., China. Melt flow rate was 16 g/10 min. Glass fibres (GF) of average diameter 12 μm were supplied by Jushi Group Co. Ltd., China. Before coating, glass fibres were calcined in a muffle oven at 500°C for 6 h. The strength of a single filament after calcination was 2034.7 MPa. Silane-coated glass fibres were prepared by exposing the fibres to aqueous solutions of γ -methacryloxypropyltrimethoxysilane (MPS, Shanghai Yaohua Chemical Plant, China). The fibres were steeped overnight in this solution and then dried at 100°C in an oven.

A maleic anhydride modified polypropylene (MPP) was produced from the isotactic polypropylene by grafting with maleic anhydride (grafting ratio =1.21% by weight). The samples of PP (90%) were blended with MPP (10%) and extruded.

Interfacial Microstructure Observation on Single Fibre/PP Composite

Single fibre-PP composites were prepared as follows: the treated fibres were placed on a glass slide, and then covered with a piece of PP with MPP (10%) sample. After the polypropylene was heated by an alcohol burner to melt, it surrounded the whole fibres and then it was pressed to a sheet.

All the composites were then transferred to a



Figure 1. Optical micrograph of the border in glass fibre/polypropylene with isothermal crystallization at 135°C.

temperature-controlled oven held at 210°C for 10 min to eliminate the previous thermal history. Then, some samples were cooled to 135°C for isothermal crystallization for 2 h, and then quenched to room temperature (labeled as transcrystalline samples) while others were cooled to room temperature at 20°C/min (labeled as quenched samples).

The interfacial crystallizations of the two samples were recorded by a Nikon polar optical microscope (LV100POL, Japan) which are demonstrated in Figures 1 and 2.

Testing of Interfacial Shear Strength via Debonding

The interfacial shear strength of the fibre/resin

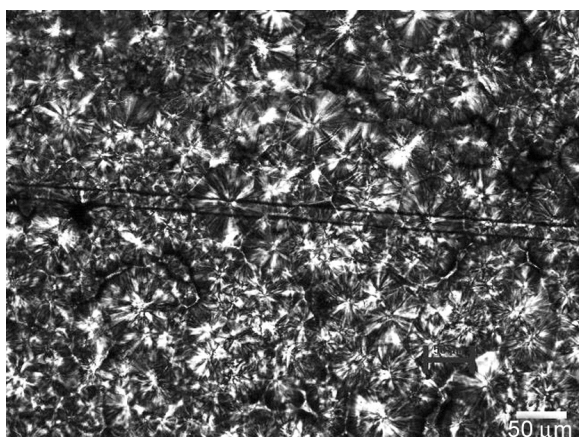


Figure 2. Optical micrograph of the border in glass fibre/polypropylene with cooling rate of 20°C/min.

interface can be determined by measuring the force needed to pull a single fibre axially out of a near sphere (droplet) of the solid matrix, namely the microbond test. The interfacial shear strength τ is calculated as follows:

$$\tau = p / (\pi dl) \quad (1)$$

where p (cN) is the pull-out force, d (mm) is the fibre diameter, and l (mm) is the length of the embedded fibre [29].

To make such pull-out measurements, the length of the embedded fibre should be short enough, for not to break before being pulled out. This is difficult to achieve by the conventional methods with glass fibres of small diameter. The approach in the present work was to dip the fibre into the polypropylene melt to form one or more discrete microdroplets and to cure droplets into the shape of ellipsoids, which concentrically surround the fibre. After solidification of the resin each fibre was examined under the optical microscope. An image of each droplet was captured by a video camera. From these images it was possible to determine the embedded length of fibre in each droplet as well as the fibre length between both ends of the droplet.

All the samples for the microbond test were given the same thermal treatments as for the optical microscope films. We believed that the interfacial crystallizations of these droplet-fibre samples for the microbond test were similar to those of the single fibre-composite films with the same thermal histories.

The microbond tests were performed with an Electronic Single Fibre Strength Tester (Model YG004A, China), attached with a chart recorder (Changzhou No.2 Textile Machinery Co. Ltd, China). A ramp force 1N/min was applied to the fibre until it was pulled out from the droplet and the load-displacement curve was recorded. A schematic representation of a valid microbond test is shown in Figure 3. The failure mode mechanism in this microbond tests has been discussed by Miller et al. [28].

About 30 specimens were tested for each set of conditions. Each specimen was examined by an optical microscopy after mechanical testing to verify that debonding, rather than cohesive failure of the matrix, had occurred.

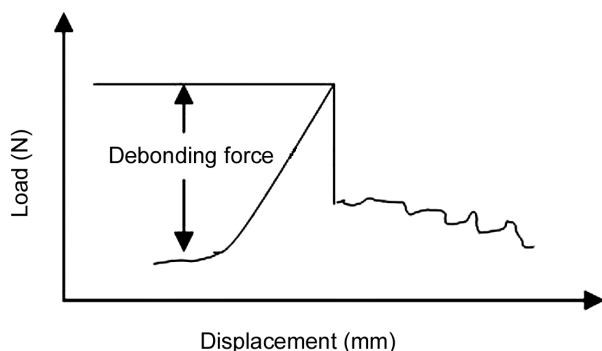


Figure 3. Schematic representation of a typical load-displacement curve obtained from a valid microbond test.

Thermal Treatment

Both types of droplet samples were thermally treated at various temperatures for 20 h in a temperature-controlled oven and then used in the microbond test.

DSC Analysis

The droplet samples were selected for DSC measurement. For the registration of differential scanning calorimetry (DSC) thermograms we used a Perkin-Elmer Diamond DSC instrument (USA) at a heating rate of 10°C/min with nitrogen flow (20 mL/min). Weight fraction crystallinities were derived from the heat of fusion by taking the ratio to the ideal value ΔH^0 (whole enthalpy) of a fully crystalline PP. We selected $\Delta H^0 = 207 \text{ J.g}^{-1}$ as reported by Janimak et al. [32].

SEM Observation

PP/MPP (90/10) was dissolved in xylene at a concentration of 2% (by weight) to reflux for 2 h. The treated fibre was fixed by a double-faced adhesive tape on a glass carrier. The solution of the PP/MPP was spread to cover the fibre horizontally and then

placed in a vacuum oven to cast the film. Then, the film was given the same thermal history as for the optical microscope films and examined by a high-resolution scanning electron microscope (Jeol JSM-6360LV, Japan).

RESULTS AND DISCUSSION

Transcrystallization and the Interfacial Shear Strength in Debonding GF/PP

Figures 1 and 2 show the microstructure of the two composites that were isothermally crystallized and quenched, respectively. In the isothermal crystallization composites a transcrystalline interlayer was formed. No transcrystallization was evident in the GF/PP composites cooled at the rate of 20°C/min, i.e., the quenched composites.

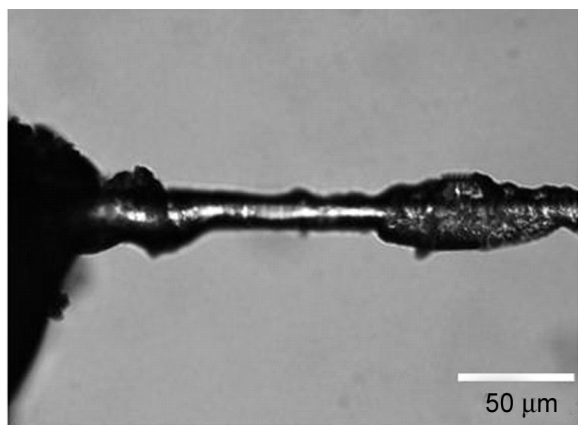
Table 1 shows the mean IFSS via debonding for the two samples. The IFSS was found to be 7.53 MPa for the transcrystalline and 14.69 MPa for the quenched samples.

The surfaces of glass fibres pulled out from the droplets of the transcrystalline and the quenched samples are shown in Figure 4. For the quenched samples, the whole PP droplet was debonded from the glass fibre and there were no traces of debris on the fibre surface. Thus, the shear debonding of the quenched sample occurred at GF/PP interface. However, for the transcrystalline sample, some residual PP adheres to the glass fibre after debonding, consistent with the results of Zhang et al. [20].

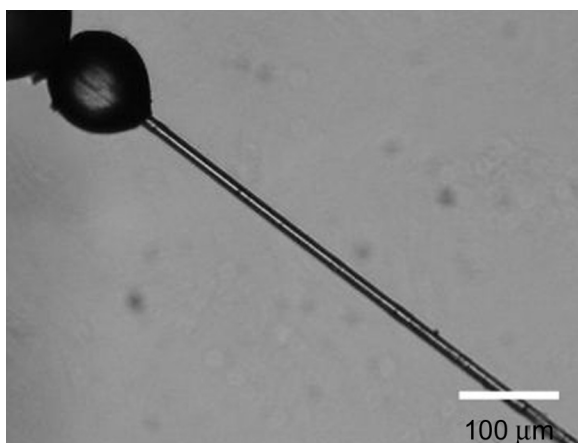
It is highly suspected that the failure could easily take place at the large, presumably amorphous gap between the transcrystalline layer and the spherulites under shear stress, as evident in Figure 5. Therefore,

Table 1. Microbond test results before and after thermal treatment.

Samples	Interfacial shear strength, τ , (MPa) \pm (standard deviation)			
	Before thermal treatment	After thermal treatment at 70°C for 20 h	After thermal treatment at 100°C for 20 h	After thermal treatment at 130°C for 20 h
Quenched sample	14.69 \pm 0.40	16.94 \pm 1.81	18.98 \pm 1.50	19.59 \pm 0.86
Transcrystalline sample	7.53 \pm 0.29	16.89 \pm 1.29	18.72 \pm 0.42	19.07 \pm 0.26



(a)



(b)

Figure 4. Surface of glass fibres pulled out from droplets before thermal treatment: (a) transcrystalline and (b) quenched samples.

the rupture of the transcrystalline sample first occurs at the rim where the transcrystalline region meets the spherulites of the PP matrix. Thus, the interfacial shear strength via debonding for the transcrystalline sample is the physical bonding force and the frictional force between the transcrystalline layer and the spherulites which is lower than the strong interactions needed to pull fibres out of the matrix [33]. Therefore, the value of interfacial shear strength via debonding for the transcrystalline sample is lower than that for the quenched one.

Thermal Treatment and Interfacial Shear Strength in Debonding of GF/PP

Table 1 also shows the mean interfacial shear strength via debonding for these two samples after thermal treatment at various temperatures for 20 h. The IFSS was found to be 16.89 MPa for the transcrystalline

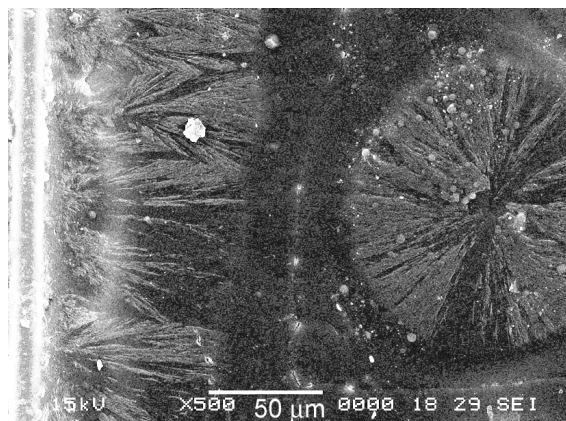
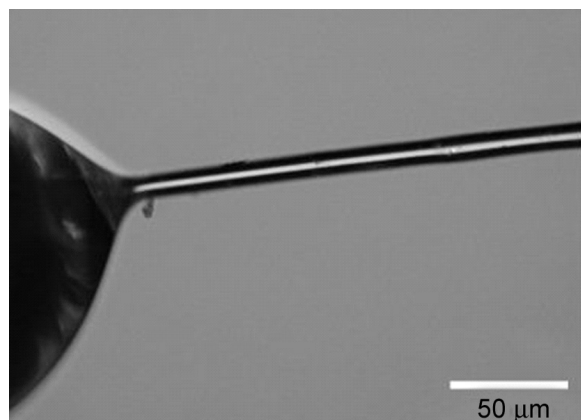


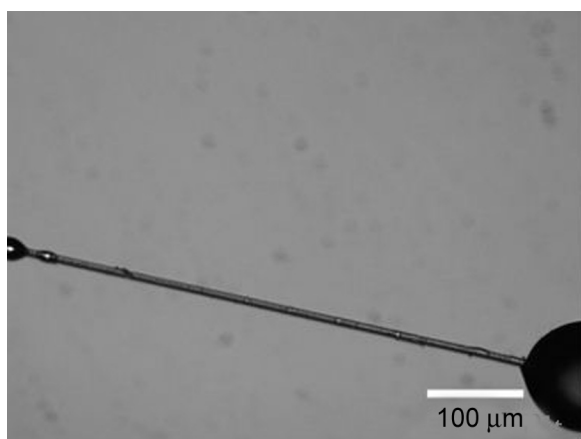
Figure 5. SEM Micrograph of the border in glass fibre/polypropylene of the transcrystalline sample before thermal treatment

and 16.94 MPa for the quenched sample when annealed at 70°C for 20 h; 18.72 MPa for the transcrystalline and 18.98 MPa for the quenched sample at 100°C for 20 h; 19.07 MPa for the transcrystalline and 19.59 MPa for the quenched sample at 130°C for 20 h. It is obvious that the values of IFSS via debonding, for both samples, are essentially the same after the same thermal treatment and they increase as the thermal treatment temperature increases. We suggest that this may be due to the thermal stress formation in the cooling process which can be relaxed at higher temperature of thermal treatment, and it is in favour of IFSS improvement. Moreover, higher temperature thermal treatment results in the perfection of some crystallites and chain diffusion between the noncrystalline and crystalline regions [34].

Figure 6 shows the surface of glass fibres pulled out from the droplets of the transcrystalline and the quenched samples after thermal treatment. Optical microscope observations do not seem to reveal significant differences between the two samples. There are no traces of debris adhering to the fibre surface after debonding in either samples. After thermal treatment the shear debonding of the transcrystalline sample occurs at the interface of the GF/PP composite, not at the rim where the transcrystalline region meets the spherulites of the PP matrix. Thus, the value of interfacial shear strength for the transcrystalline sample is almost the same as the quenched sample after thermal treatment, and increases due to the release of the thermal stress.



(a)



(b)

Figure 6. Surface of glass fibres pulled out from droplets after thermal treatment at 100°C for 20 h: (a) transcrystalline and (b) quenched samples.

Figure 7 depicts the DSC thermograms of transcrystalline specimen before and after thermal treatment. We observe a main endothermic peak and a secondary broad endotherm that develops on the low-temperature side of the main peak and shifts to higher temperatures after thermal treatment which has

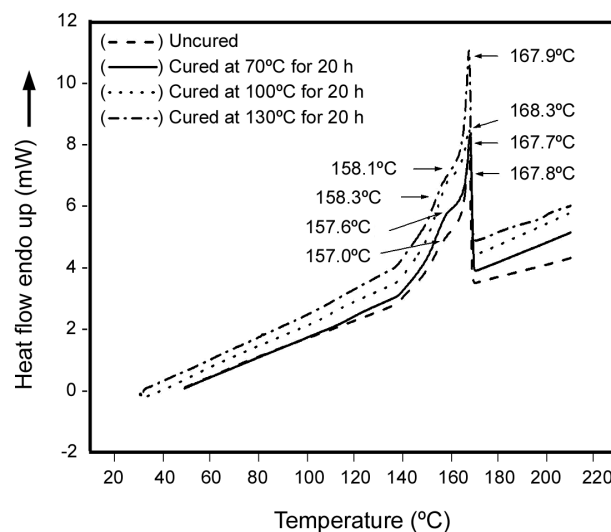


Figure 7. DSC Melting curves of the transcrystalline samples with various thermal treatments (heating rate: 10 K. min⁻¹).

been often observed and reported [35].

As recorded in Table 2, the total heat of the transcrystalline sample is 106.83 J.g⁻¹, indicating a weight fraction crystallinity of $\phi_w = 0.516$ (crystalline weight percentage). After thermal treatment at 70°C, 100°C and 130°C for 20 h, respectively, the degree of crystallinity has changed slightly. There is only a little increase at 130°C, since higher temperature thermal treatment is favourable to the perfection of crystallites.

The mechanical properties of semicrystalline polymers are mainly dependent on their molecular weight, nature of the crystal morphology, lamella thickness, packing molecules, degree of crystallinity and spherulite size. As the crystalline structure and degree of crystallinity only changed slightly after thermal treatment, the differences in the intercrystalline

Table 2. Melting characteristics of the transcrystalline sample before and after thermal treatment.

Thermal treatment	T _{m1} (°C)	T _m (°C)	ΔH _{PP} (J/g) ± (standard deviation)	X _{PP} (%)
Untreated	157.0	167.8	106.4±0.4	51.2
20 h at 70°C	157.6	167.7	107.1±0.3	51.7
20 h at 100°C	158.3	168.3	108.5±1.1	52.4
20 h at 130°C	158.1	167.9	112.8±0.7	54.5

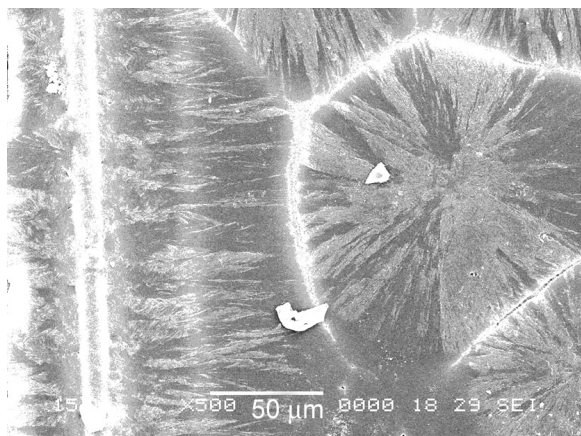


Figure 8. SEM Micrograph of the border in glass fibre/polypropylene of the transcrystalline sample after thermal treatment at 100°C for 20 h.

mechanical interconnections, also called the “semicrystalline structure continuity” [22] must be responsible for the differences in the inter-facial shear strength via debonding. Chain diffusion between the non-crystalline and crystalline regions [34] after thermal treatment also favours the increase in the interaction between the transcrystalline region and the spherulites, because individual chain may connect the lamellae in the transcrystalline layer with those in the spherulites at the contacting region of transcrystalline layer and spherulites.

Figure 8 is an SEM micrograph of the border in GF/PP of the transcrystalline composites after thermal treatment. With the perfection of the crystals, the chains of the lamellae in the transcrystalline layer may connect better with those in the spherulites in the region where the transcrystalline layer meets the spherulites, which improves the physical adhesion between the transcrystalline and the spherulite regions. In fact, the contact surface area of transcrystallization with spherulite increased and the anchorage of spherulite at the transcrystalline layer is favoured. The IFSS measured in the pull-out test is considered to be based on the physical-chemical bonding force and the frictional forces between the fibre and matrix [9]. The change in the IFSS is related to the frictional force as the physical-chemical bonding force is constant for the same fibre surface treatment and matrix resin. Increasing the anchorage of spherulites at the transcrystalline layer is

favourable to the increase of the frictional forces and thus the failure did not first occur at the rim where the transcrystalline region meets the spherulites. Then, the debonding of the transcrystalline samples happens at the interface of glass fibre and matrix, and the IFSS is determined by the adhesion of GF/PP to be increased due to the release of the thermal stress.

CONCLUSION

There is a transcrystalline interlayer in the isothermally crystallized composites, while no transcrystallization occurs in the GF/PP composites when cooled at the rate of 20°C/min. The shear debonding of the transcrystalline specimen occurs at the rim where the transcrystalline region meets the PP spherulites due to the discontinuous gap, consisting of quenched amorphous polymer, between the transcrystalline layer and the spherulites, while the failure of the quenched layer happens at the GF/PP interface. This leads to the IFSS via debonding being only 7.53 MPa for the transcrystalline sample while it is 14.69 MPa for the quenched sample.

After thermal treatment the shear debonding of the transcrystalline specimen also occurs at the interface of GF/PP composite, just like the quenched specimen. Furthermore, IFSS values of the transcrystalline and the quenched samples are almost the same and increase to nearly 20 MPa when they were annealed at 130°C. The frictional forces between the transcrystallization and spherulites regions increase after thermal treatment and then the failure does not occur at the rim where the transcrystalline region meets the PP spherulites under stress. Thus, the debonding of the transcrystalline specimen happens at the interface between glass fibres and matrix and as a result the interfacial adhesion increases.

REFERENCES

1. Na K, Park H-S, Won H-Y, Lee J-K, Lee K-H, Nam J-Y, Jin B-S, SALS study on transcrystallization and fiber orientation in glass fiber/polypropylene composites, *Macromol Res*, **14**, 499-503, 2006.

2. Wang YM, Tong BB, Hou SJ, Li M, Shen CY, Transcrystallization behavior at the poly(lactic acid)/sisal fibre biocomposite interface, *Compos Part A-Appl S*, **42**, 66-74, 2011.
3. Wang C, Liu F-H, Huang W-H, Electrospun-fiber induced transcrystallization of isotactic polypropylene matrix, *Polymer*, **52**, 1326-1336, 2011.
4. Masoomi M, Ghaffarian SR, Mohammadi N, The effect of processing conditions and matrix type on the interface of polyethylenefibre/polyethylene-matrix composites, *Iran Polym J*, **13**, 381-387, 2004.
5. Wang K, Guo M, Zhao DG, Zhang Q, Du RN, Fu Q, Dong X, Han CC, Facilitating transcrystallization of polypropylene/glass fiber composites by imposed shear during injection molding, *Polymer*, **47**, 8374-8379, 2006.
6. Heppenstall-butler M, Bannister DJ, Young RJ, A study of transcrystalline polypropylene/single-aramid-fibre pull-out behaviour using Raman spectroscopy, *Compos Part A-Appl S*, **27**, 833-838, 1996.
7. Huang Y, Young RJ, Interfacial behavior in high temperature cured carbon fibre/epoxy resin model composite, *Composites*, **26**, 541-550, 1995.
8. Nielsen AS, Pyrz R, Study of the influence of thermal history on the load transfer efficiency and fibre failure in carbon/propylene microcomposites using Raman spectroscopy, *Compos Intreface*, **6**, 467-482, 1999.
9. Moon CK, The effect of interfacial microstructure on the interfacial strength of glass fiber/polypropylene resin composites, *J Appl Polym Sci*, **54**, 73-82, 1994.
10. Devaux E, Caze C, Evolution of the interfacial stress transfer ability between a glass fiber and a polypropylene matrix during polymer crystallization, *J Adhes Sci Technol*, **14**, 965-974, 2000.
11. Varge J, Karger-Kocsis J, The occurrence of transcrystallization or row-nucleated cylindrical crystallization as a result of shearing in a glass-fiber-reinforced polypropylene, *Compos Sci Technol*, **48**, 191-198, 1993.
12. Varga J, Karger-Kocsis J, Interfacial morphologies in carbon fibre-reinforced polypropylene microcomposites, *Polymer*, **36**, 4877-4881, 1995.
13. Nielsen AS, Pyrz R, A Raman study into the effect of transcrystallisation on thermal stresses in embedded single fibres, *J Mater Sci*, **38**, 591-601, 2003.
14. Amitay-Sadovsky E, Cohen SR, Wagner HD, Anisotropic nanoindentation of transcrystalline polypropylene by scanning force microscope using blade-like tips, *Appl Phys Lett*, **74**, 2966-2968, 1999.
15. Amitay-Sadovsky E, Cohen SR, Wagner HD, Nanoscale shear and indentation measurements in transcrystalline α -isotactic polypropylene, *Macromolecules*, **34**, 1252-1257, 2001.
16. Assouline E, Fulchiron R, Gerard JF, Wachtel E, Wagner HD, Marom G, γ -transcrystallization in isotactic polypropylene-based composites promoted by aramid fibers, *J Polym Sci Pol Phys*, **37**, 2534-2538, 1999.
17. Assouline E, Pohl S, Fulchiron R, Gerard JF, Lustiger A, Wagner HD, The kinetics of α and β transcrystallization in fibre-reinforced polypropylene, *Polymer*, **41**, 7843-7854, 2000.
18. Assouline E, Wachtel E, Grigull S, Lustiger A, Wagner HD, Marom G, Lamellar twisting in α isotactic polypropylene transcrystallinity investigated by synchrotron microbeam X-ray diffraction, *Polymer*, **42**, 6231-6237, 2001.
19. Assouline E, Grigull S, Marom G, Wachtel E, Wagner HD, Morphology of α -transcrystalline isotactic polypropylene under tensile stress studied with synchrotron microbeam X-ray diffraction, *J Polym Sci Pol Phys*, **39**, 2016-2021, 2001.
20. Zhang MQ, Xu JR, Zhang ZY, Zeng HM, Xiong XD, Effect of transcrystallinity on tensile behaviour of discontinuous carbon fibre reinforced semicrystalline thermoplastic composites, *Polymer*, **37**, 5151-5158, 1996.
21. Drozdov AD, Christiansen JC, The effect of annealing on the time-dependent behavior of isotactic polypropylene at finite strains, *Polymer*, **43**, 4745-4761, 2002.
22. Chen F, Shanks RA, Amarasinghe G, Structural and mechanical properties changes of ethylene-olefin copolymer blends induced by thermal treatments and composition, *Macromol Mater Eng*, **289**, 552-561, 2004.
23. Casellas JJ, Frontini PM, Carella JM, Fracture

- characterization of low-density polyethylenes by the essential work of fracture: changes induced by thermal treatments and testing temperature, *J Appl Polym Sci*, **74**, 781-796, 1999.
24. Piggott M, Why interface testing by single-fibre methods can be misleading, *Compos Sci Technol*, **57**, 965-974, 1997.
 25. Mandell JF, Grande DH, Tsiang TH, McGarry FJ, Modified microdebonding test for direct in situ fiber/matrix bond strength determination in fiber composites, *Material & Technik (Duebendorf, Switzerland)*, **893**, 87-108, 1986.
 26. Chua P, Piggott M, The glass fibre-polymer interface. II: work of fracture and shear stresses, *Compos Sci Technol*, **22**, 107-119, 1985.
 27. Eagles DB, Blumentritt BF, Cooper SL, Interfacial properties of Kevlar-49 fiber-reinforced thermoplastics, *J Appl Polym Sci*, **20**, 435-448, 1976.
 28. Miller B, Muri P, Rebenfeld L, A microbond method for determination of the shear strength of a fiber/resin interface, *Compos Sci Technol*, **28**, 17-32, 1987.
 29. Wong S, Shanks RA, Hodzic A, Effect of additives on the interfacial strength of poly(lactic acid) and poly(3-hydroxy butyric acid)-flax fibre composites, *Compos Sci Technol*, **67**, 2478-2484, 2007.
 30. Houshyar S, Shanks RA, Interfacial properties of all-polypropylene composites, *e-Polymers*, **33**, 1-13, 2010.
 31. Li Y, Lin QF, Chen LX, Zhou XD, Effect of Transcrystallization and Thermal Treatment on Interfacial Bond Strength of Glass Fiber/Polypropylene Composites, *5th East-Asian Polym Conf*, 304-305, 2008.
 32. Janimak JJ, Cheng SZD, Crystallization studies in isotactic polypropylene fractions, *J Polym Eng*, **10**, 21-69, 1991.
 33. Li Y, Chen LX, Zhou XD, Interfacial crystalline behavior in glass-fiber/polypropylene composites modified by block copolymer coupling agents, *J Mater Sci*, **43**, 5083-5091, 2008.
 34. Yao YF, Graf R, Spiess HW, Rastogi S, Restricted segmental mobility can facilitate medium-range chain diffusion: a NMR study of morphological influence on chain dynamics of polyethylene, *Macromolecules*, **41**, 2514-2519, 2008.
 35. Iijima M, Strobl G, Isothermal crystallization and melting of isotactic polypropylene analyzed by time- and temperature-dependent small-angle X-ray scattering experiments, *Macromolecules*, **33**, 5204-5214, 2000.