ISSN 1027-5495. Functional Materials, 26, No.3 (2019), p. 530-533.
doi:https://doi.org/10.15407/fm26.03.530

Preparation and analysis of the structure of the composite material of polypropylene — silicon nanoxide

H.Xu

Department of Basic Science, Henan Polytechnic Institute, Henan, China

Received April 18, 2019

The structure of the composite material of polypropylene — silicon nanoxide obtained by the method of mixing in the melt was investigated. The results showed that nano-SiO_2 was little agglomerated and evenly distributed. Superdispersed SiO_2 dispersed in polypropylene was found to affect the structure of the condensed polypropylene substance, which increases the crystallinity of polypropylene and leads to a decrease in crystal deformation. The distribution of crystalline particles was more compact, which proves that nano-organic solids can simultaneously reinforce and harden the polymer.

Keywords: nano-SiO_2, polypropylene, melt blending, condensed matter.

Исследована структура композитного материала полипропилена — наноксида кремния, полученного методом смешивания в расплаве. Результаты показали, что нано-SiO_2 был мало агломерирован и распределен равномерно. Обнаружено, что ультрадисперсный SiO_2, диспергированный в полипропилене, влияет на структуру конденсированного вещества полипропилена, что повышает кристалличность полипропилена и приводит к уменьшению деформации кристалла. Распределение кристаллических частиц было более компактным, что доказывает, что наноорганические твердые частицы могут одновременно усиливать и упрочнять полимер.

Отримання і аналіз структури композитного матеріалу поліпропілену — наноксіду кремнію. *H.Xu.*

Досліджено структуру композитного матеріалу поліпропілену — наноксіду кремнію, отриманого методом змішування в розплаві. Результати показали, що нано-SiO_2 був мало агломерований і розподілений рівномірно. Виявлено, що ультрадисперсний SiO_2, диспергований у поліпропілені, впливає на структуру конденсованої речовини поліпропілену, що підвищує кристалічність поліпропілену і призводить до зменшення деформації кристала. Розподіл кристалічних частинок є більш компактним, що доводить, що наноорганічні тверді частинки можуть одночасно посилювати і зміцнювати полімер.

1. Introduction

Although polypropylene PP has high strength, good abrasion resistance, light weight, low price, easy to form, and abundant source of raw materials, it has limited application scope because of its low impact strength [1]. Therefore, to study the toughening is important for PP modification. With the appearance of nanomaterials, the studies of PP toughening with nano inorganic particles become more and more intense. Because of small size and a big specific surface area of nanometer particles, they can produce a lot of interface interactions with PP; these can provide this material with inorganic rigidness, thermal stability, dimension stability of nano-particles and good wear-resistance, dielectric properties, and processability of plastic plastic [2]. The problem of high surface energy and easy agglomeration should be solved before the application of nano-level particles. By surface treatment of the nano-level particles, the surface modifier can form an elastic interface layer between the matrix and the filler, which can effectively transfer and relax the stress at the interface, and absorb and disperse the external impact energy better [3].

If the dispersion problem of nano-inorganic particles is solved, the nano-inorganic particles can be widely used in strengthening and toughening of polymers [4, 5]. In [6], cracks and silver grains of plastics reinforced and strengthened by nano-inorganic particles were studied in the process of modifying the PP with a nanofiller. The authors proposed a mechanism for transformation of cracks into silver strias and suggested that the hardening and strengthening of PP were caused by filling the cracks of PP with nanoparticles. In [7, 8] PP/SiO_2-g-pba composites obtained by in situ grafting during melt mixing and their crystallization properties, microscopic morphology, dynamic viscoelastic behavior of the composites, and their mechanical properties were studied. The results show that most of the nanoparticles can be distributed in the amorphous phase of the matrix and play the role of physical cross-links. When the composite is subjected to external forces, the modified nanoparticles act as the stress concentration point. In the process of debonding with the matrix, the strong interface bonding can cause the matrix to undergo plastic deformation and absorb a large amount of energy; thus, the rigidity, tensile and impact properties of the composite are improved [9]. The results [10] showed that the large specific surface area of nano-SiO_2 and the strong force between nano SiO_2 and PP matrix interface were the main factors to strengthen and toughen the PP.

In this paper, the properties of the PP/nano-SiO_2 composite prepared by melt blending were studied. Taking into account the synergistic effect of nano-SiO_2 particles after surface modification and the PP-g-MAH, compatibilizer, the structure of nano-SiO2 particles was deeply analyzed during the application process; this provides a theoretical basis for the reliable strengthening and toughening the polymers by nano-inorganic particles.

2. Experimental

According to the characteristics of nano SiO_2, a special surface treatment agent is designed to make one end of it easily react with a large amount of hydroxyl groups on the surface of nano-SiO_2 and the other end easily compatible with PP. At the same time, the surface treatment agent is specially designed according to the particle size of nano-SiO_2.

The process route adopted in this experiment is shown in the Fig. 1.

Nano-SiO_2, after being treated with the surface treatment agent, is mixed with PP powder and other additives in the high mixing machine. Corea TE twin-screw extruder (diameter 34 mm, $l/d = 28$) was used for granulation. Finally, standard test samples were prepared by a Zhende injection molding machine.

3. Results and discussion

SiO_2 nanoparticles have small size, large specific surface area and a large number of hydroxyl groups with different bond states on the surface. Fig. 2 shows the transmission electron micrograph of nano SiO_2. As can be seen from Fig. 2, the nanometer SiO_2 used in this experiment has a large particle size span ranging from tens of nanometers to hundreds of nanometers, with fuzzy surface. Fig. 3 shows the infrared spectrum of nano-SiO_2, and the absorption peak of hydroxyl groups at 3431 cm^{-1}, indicating that the surface of nano-SiO_2 has a large amount of hydroxyl groups.

According to the research, 2 parts of nano-SiO_2 were treated with 1.5 parts of surface treatment agent and mixed with 100 parts of PP powder, and the comprehensive mechanical properties of the material were the best. Compared with pure PP, the tensile strength increased by 5 %, v-notch impact strength increased by 90 %, and bending strength increased by 23 %, thus, the goal to increase strength and toughness of polymer with nano inorganic particles was realized. The observable transparency of 2 nano-SiO_2 samples was close to that of pure PP. The v-notch impact section of the sample was observed by SEM, as shown in Fig. 4. Nano SiO_2 (white dots in Fig. 4) was evenly distributed in the PP matrix, with basically no agglomeration and uniform particle size distribution. Some particle sizes were about 100 nm. The impact fracture surface of the composite material treated with the surface treatment agent has many small silver striations, which can disperse the impact stress and improve the impact toughness of the composite material.

For crystalline polymers, the crystalline state of the material has a great influence on the properties of the material. Inorganic particles are added to PP to promote the heterogeneous nucleation. Therefore, the

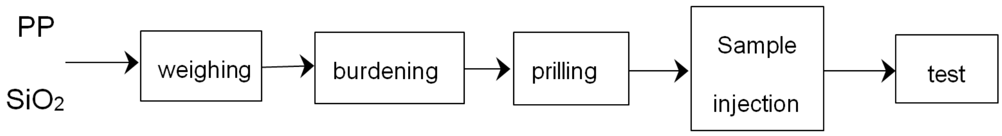

Fig. 1. Process roadmap.

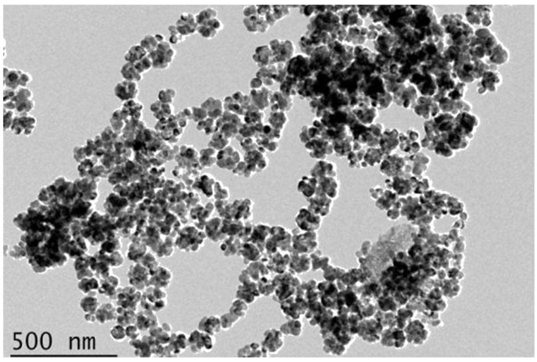

Fig. 2. SEM of nano-SiO_2.

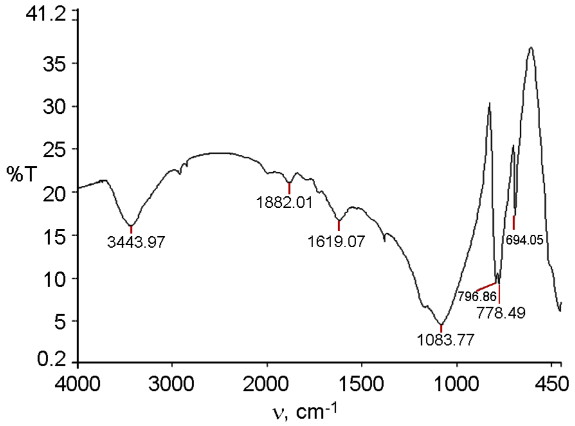

Fig. 3. Infrared spectrum of nano-SiO_2.

crystallization of PP can be affected. The nano SiO_2 particles in 0, 1, 2, 3, 4 and 5 parts respectively treated with 1.5 parts of surface treatment agent for DSC testing. Fig. 4 show the relationship between the melting crystallization peak temperature, crystallinity and the width half-height of the melting crystallization peak of PP and the addition amount of nano SiO_2 particles, respectively. As can be seen from Fig. 4, the melting crystallization peak of the composite material increased after the addition of nano SiO_2 particles. When two parts of nano-SiO_2 is added the melting-crystallization peak temperature increasesd by 5.2° because the bondage of spherical SiO_2 particles of PP chain segments is weaker. The melting-crystallization peak is used to reflect the rate of nucleation. The experimental results show that the added nano-SiO_2 particles have the function of nucleating agent, and the nano-SiO_2 particles are evenly dispersed in PP with without agglomeration. According to the test results, it can also be seen that the crystallinity of the composite increases with the increase of nanometer SiO_2 particle weight portion. The crystallinity of pure PP was 47.5 %. When adding 2 parts of nano-SiO_2, the crystallinity increased to 49.4 %. High crystallinity is accompanied with high density, high hardness, and good tensile strength, bending strength and wear resistance.

It can be seen from Fig. 4 that the width at half height of the PP melting crystallization peak decreased when nano SiO_2 was added. When the addition amount is 2 parts, the temperature drops to the lowest value and the half-peak width is 4.3 f. When the addition amount was 3 parts, the half-height width of crystallization peak increased in 5.7 f, and the growth rate of crystal nuclei slowed down. When the added amount of nano-SiO_2 particles is too much, the distribution of crystal nuclei will be hindered. The half-height width and narrow tip indicate that the grains are formed in a short time and the size is uniform, which can effectively improve the impact strength of the composite.

The X-ray diffraction test results the composites doped with 2 parts of nano-SiO_2 are shown in Table 1. The crystal volume of PP decreased with the addition of 2 parts of nano-SiO_2. For the pure PP 100 crystal, 2 is 14.350°, width at half height is 0.960 f. Addition of 2 θ portions of nano - SiO_2 to the *P* 100 crystal results in 2 θ increased to 14.415° and half high width of 1.060 f. According to the formula calculation, the crystal volume of PP is 90% of pure PP after adding of 2 portions of nano-SiO_2.

When SiO_2 was added into the pure PP, the crystals of the PP became smaller and the impact strength was greatly improved. The crystallinity was slightly higher than that of pure PP, and the tensile strength increased accordingly. The experimental results show that the strength and toughness of PP can be improved simultaneously by using nano SiO_2 at the same time. With the improvement of preparation methods of nano-inorganic particles, the cost of nano-inorganic particles will be lower and the particle size will be smaller.

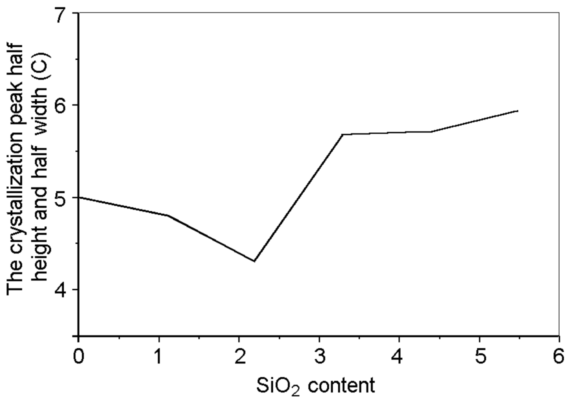

Fig. 4. Relationship between the crystal peak half-height of the composite material and the amount of SiO_2.

4. Conclusions

The following results can be obtained from this study: the better the dispersion of nano SiO_2 particles in PP matrix, the more fuzzy the boundary of PP spherulites, and the finer and more uniform the size of the spherulites; the better the dispersion of nano SiO_2 particles in PP matrix, the more obvious the increase of PP phase crystallization and melting temperature in PP/nano-SiO_2 composite. the toughness of PP/nano-SiO_2 fused composites was significantly improved.

Although many researchers have explored the application of nano SiO_2 to modify PP to achieve the strengthening and toughening of PP, at present, the preparation technologies are difficult to achieve industrial production, so it is difficult to obtain the real nano composite materials. To industrialize the achievements of scientific researches is the problemof great importane should be attached great importance in academic and industrial circles. With the continuous improvement of preparation technology and the improvement of dispersion and interfacial adhesion of nano particles, the PP/nano-SiO_2 composites will have a large space for development.

Table. X-ray diffraction results of PP/nano-SiO_2 composites

Program	2θ, °	Half high width, f
PP	14.350	0.960

References

1. Y.Zhao, X.Qi, D.Yu, *Tribology Intern.*, **103**, 599 (2016).
2. Z.Luo, Z.Hua, X.Gao, *Energy Build*, **147**, 41 (2017).
3. X.Zhang, P.Dong, B.Zhang, *J Alloys Comp.*, **671**, 465 (2016).
4. S.Rezaei, H.Ziari, S.Nowbakht, *Liquid Fuels Techn.*, **34**, 1195 (2016).
5. J.Chen, X.Zhou, H.Ge, *Polymer Composites*, **37**, 334 (2016).
6. S.Safi, A.Zadhoush, M.Ahmadi, *Plastics Rubber & Composites*, **46**, 1 (2017).
7. Z.Wang, Z.Wang, H.Hu, *J. Mater. Engin. Perform.*, **25**, 3880 (2016).
8. M.Farsi, *J. Polymers. Environment*, **25**, 1 (2016).
9. Y.Wang, X.Gao, C.Peng, *Appl. Therm. Engin.*, **96**, 699 (2016).