

강력 전단력으로 배합된 테트라포드 형태의 Zinc Oxide가 충전된 에폭시 수지의 물성

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The Properties of Epoxy Resins Modified by Tetrapod-like Zinc Oxide Whiskers under Powerful Shear Force Field

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Abstract: The effects of epoxy composites (EP) filled with tetrapod-like zinc oxide whiskers (T-ZnOw) were investigated in this study. The composites were blended by a self-regulation powerful shear mixer at different mixing clearances. The microstructure, curing behavior, mechanical performance, thermal and electrical properties were examined. The results showed that the particles of T-ZnOw are dispersed more uniformly in the epoxy matrix mixed by the powerful shear mixer compared with those mixed by the traditional mixing method. Both the initial reaction of extrapolation temperature (T_0) and the peak temperature (T_p) enlarge with the augment of the clearance (δ). The range of curing temperature narrows down. ΔH , tensile and impact strength of composites decreases with the increasing of δ .

Keywords: epoxy resin, tetrapod-like zinc oxide whiskers, powerful shear mixer, property.

Introduction

Epoxy resins are widely used in numerous areas such as adhesives, coatings, electronic polymeric materials, aerospace industries, especially in electronic packaging materials for their superior mechanical properties, excellent thermal resistance, and good processability.¹⁻³ However, epoxy network is brittle, which restricts its application. Therefore, tremendous studies have been carried out to improve toughness of epoxy resins.^{4,5} The earliest toughening agents of epoxy resin were some elasticizers such as phthalate, phosphate, and fatty acid multi glycidyl. The other toughening agents, such as rubber particle,⁶⁻⁸ organic montmorillonite,⁹⁻¹¹ and nano-TiO₂,¹² were subsequently used to improve the toughness of epoxy resins. Chonkaew and Sombatsompop have prepared the composites of epoxy resins modified by carboxy-terminated butadieneacrylonitrile copolymer (CTBN). They found that mechanical properties of the modified resins were elevated.¹³ They have achieved the desired results.

Tetrapod-like zinc oxide whiskers (T-ZnOw), a typical semi-conducting material with high strength and modulus, has been widely used as reinforcing fillers. Mathioudakis,¹⁴ Chen¹⁵ and Zhou¹⁶ have investigated the electric, thermal and mechanical properties of the T-ZnOw/EP. All the results showed the performances have an excellent advance. However, they all ignored the important effect of T-ZnOw particles distribution in the epoxy matrix on the properties of the epoxy composites.

The epoxy composites with varying contents of T-ZnOw as a toughening agent were prepared in this study. A powerful shear mixer combining with the idea of mechanical shearing dispersion had been designed and manufactured by our group to improve the dispersion effect of T-ZnOw in epoxy matrix. The smaller the mixing clearance is, the higher the shear strength will be. The curing processes of the T-ZnOw/EP composites mixed by the powerful shear mixer were investigated. Furthermore, mechanical properties, thermal and electrical performances of those composites were researched.

Experimental

Materials. Epoxy composites were prepared by employing commercially available material. Epoxy resin (E-44, Nantong

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Table 1. The Formulation of Raw Materials

Category	Name	Type	Dosage (wt%)
Matrix	EP	E-44	100
Component 1	Zinc oxide whiskers	T-ZnOw	0~20
	Silane coupling agent	KH-570	(0~20) ×3
	ZnSt	-	1
	MgSt	-	1
	White carbon black	Analytically pure	0.5
	TBP	Analytically pure	0.5
	Triethanolamine	Analytically pure	0.4
	DBP	Analytically pure	20
Component 2	Curing agent	JA-1	15
	Phenolic resin	Analytically pure	5

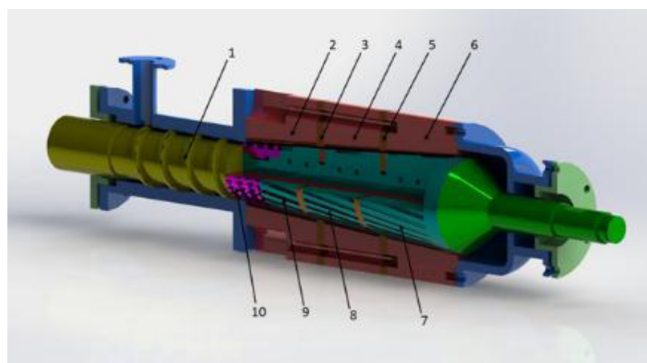


Figure 1. The powerful shear mixer. (1) conveying screw thread; (2) coarse thread casing; (3, 5) baffle; (4) fine thread casing; (6) micro thread casing; (7) micro thread shear blocks; (8) fine thread shear blocks; (9) coarse thread shear blocks; (10) scattered pieces.

Xingchen Synthetic Material Co., Ltd.), tetrapod-like zinc oxide whiskers (T-ZnOw, Chengdu University Jingyu Co., Ltd.), carbon-white (Chengdu Changzheng Huabo Co., Ltd.), curing agent (JA-1, Pujiang Fine Chemical Plant in Sichuan Province). Silane coupling agent (KH-570), ZnSt, MgSt, tributyl phosphate (TBP), triethanolamine, dibutyl phthalate (DBP) and phenolic resin were purchased from Chengdu Kelon Chemical Reagent Factory. The formulation of raw materials is shown in the Table 1.

Apparatus. The powerful shear mixer, used to improve the dispersion effect, was designed by our group based on the idea of mechanical shearing dispersion. The schematic of the machine is shown in Figure 1.¹⁷ It can be found that the mixer is consisted of conveying screw thread, scattered pieces and threaded shearing elements. The threaded shearing elements includes coarse thread shear blocks, fine thread shear blocks

and micro thread shear blocks with their respective thread casings. The material can be made a forward movement from the feed inlet into the barrel by the rotating screw. Then, the materials will be repeatedly divided and dispersed into scattered pieces. The kneaded materials achieve a uniform dispersion state under strong shear kneading elements after being transported into thread shearing segments. The strength of the shearing field can be controlled by changing the clearance between thread shearing segments and their casings. In addition, vacuum drying oven (ZK-82B, Experimental Instrument Factory in Shanghai). The casting mould was produced by ourselves.

Experimental Procedure. The experiment process is shown in Figure 2. Different contents of T-ZnOw (2~20 wt%) modified by KH-570 were added into epoxy matrix with the other constituents of component 1 (Table 1) and then they were stirred mechanically for 30 min. Then a portion of materials were put into the powerful shear mixer and stirred continuously. The colloidal fluid of T-ZnOw/EP was got by changing the clearance of mixing (1, 2, 3 mm). Subsequently, component 2 was added into epoxy matrix. After vacuum defoamation, the samples were prepared by means of mould pressing process at 10 MPa, 30 °C for 2 h and then 50 °C for 1 h. The other portions of materials were casted directly without the powerful shear mixer for comparison.

Measurement and Characterization. **Curing Reaction Test:** The colloidal fluids of T-ZnOw/EP containing various concentrations of T-ZnOw were mixed by the powerful shear mixer or traditional mechanical stir like the description of the experiment process in Figure 2. After vacuum defoamation, the curing reaction heat of the composites was measured by

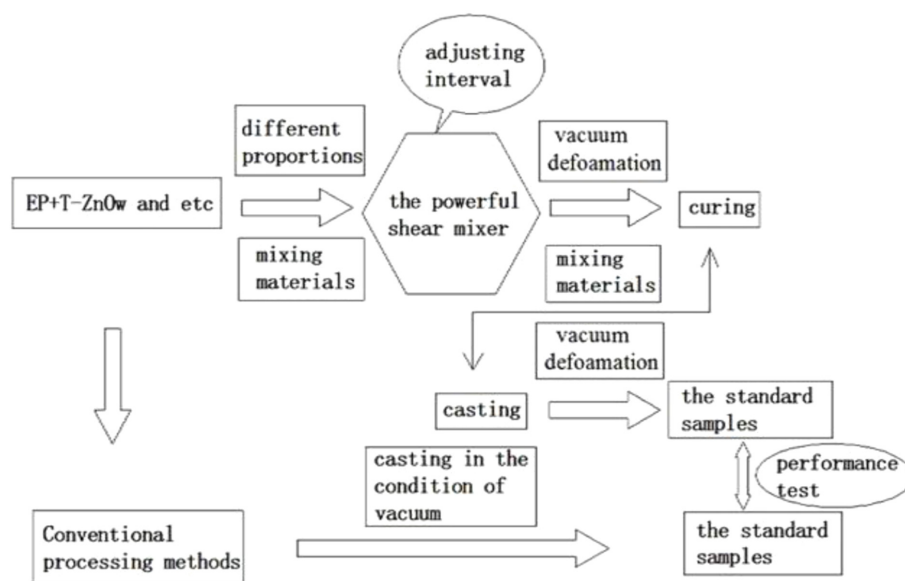


Figure 2. The experimental process.

DSC (TA Q20) at a heating rate of 5~15 °C/min with N₂ flow rate of 20 mL/min.

Dispersion Performance Test: The fracture surfaces obtained from the tensile strength tests were examined by scanning electron microscope (SEM, JSM-7500F, JEOL, Japan) with the acceleration voltage of 5 kv. The fracture surfaces of all samples were prepared and cracked in liquid N₂. Then they were etched by 50% solution of NaOH for 8 weeks. Subsequently, they were dried and sputtered with gold in vacuum before observation.

Mechanical Properties Test: A universal tensile testing machine (AG 710, SHIMADZU Corp, Japan) was used for tensile testing at crosshead speed of 5 mm/min. The impact tests were carried out by using a cantilever beam impact testing machine (XJU 5.5). The dimension of the sample for tensile test and impact test is 150×10×4 mm³ and 80×10×4 mm³, respectively.

Heat-conducting Property Test: The heat-conduction properties were measured by the thermal conductivity meter (2500-OT, Hot Disk Corp., Sweden) at 20 °C, humidity 65%. The dimension of the sample is 40×20×4 mm³.

Electric Property Test: Volume resistivity of those samples were measured by the high resistance meter (ZC36, Shanghai Anbiao electronics Co., Ltd., China) at 23 °C, humidity 60%. Dielectric strength of the samples was measured by a ball-clearance discharge manometer at 20 °C, humidity 65%. The voltage was rising at a constant rate of 20 KV/s until the samples were punctured and the voltage value was fast recorded

then. All experiments were performed in triplicate. The dimension of the samples is 11×11×3 mm³.

Results and Discussion

The Effect of Powerful Shear Field on the Microstructure of T-ZnOw/EP. The concrete structure and dispersion of T-ZnOw particles on microprofile of composites containing different contents of T-ZnOw were characterized by scanning electron microscope (SEM). The influences of shear field generated by the powerful shear mixer on the dispersion of T-ZnOw were discussed.

As we can see from Figures 3 and 4, the part of fracture is

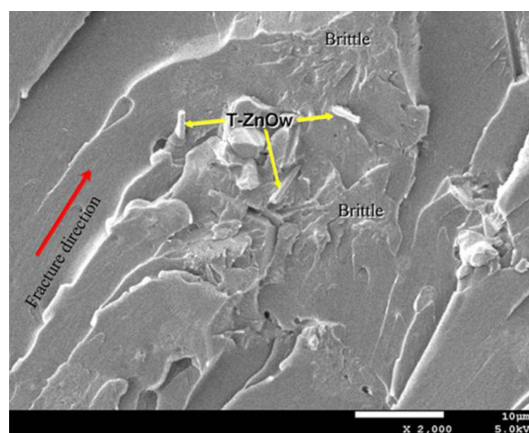


Figure 3. The micrographs of EP/2 wt% T-ZnOw powerful shear mixing ($\delta = 1$ mm).

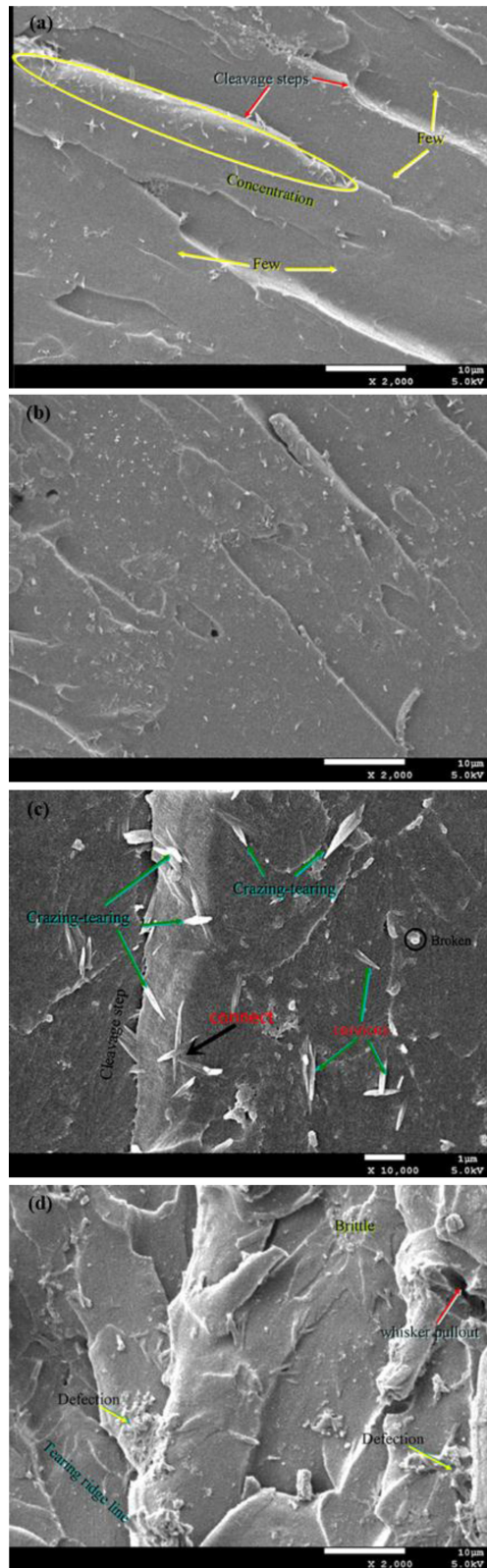


Figure 4. The micrographs of EP/5 wt% T-ZnOw. (a) mechanical mixing; (b, c, d) powerful shear mixing ($\delta = 1, 2, 3$ mm).

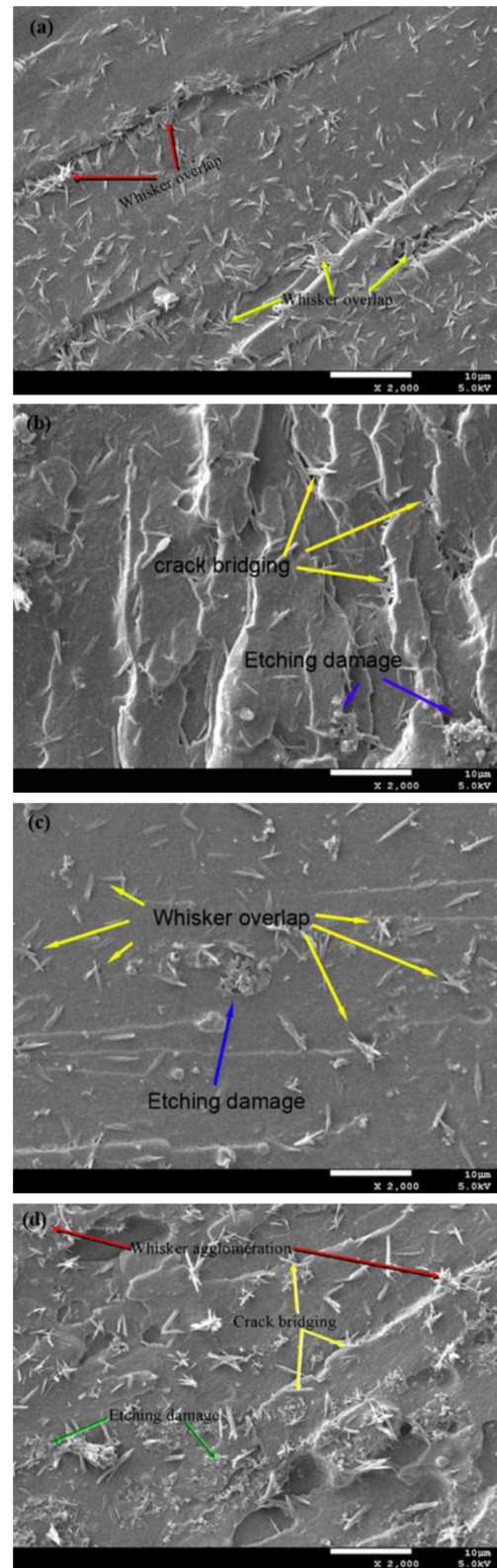


Figure 5. The micrographs of EP/10 wt% T-ZnOw. (a) mechanical mixing; (b, c, d) powerful shear mixing ($\delta = 1, 2, 3$ mm).

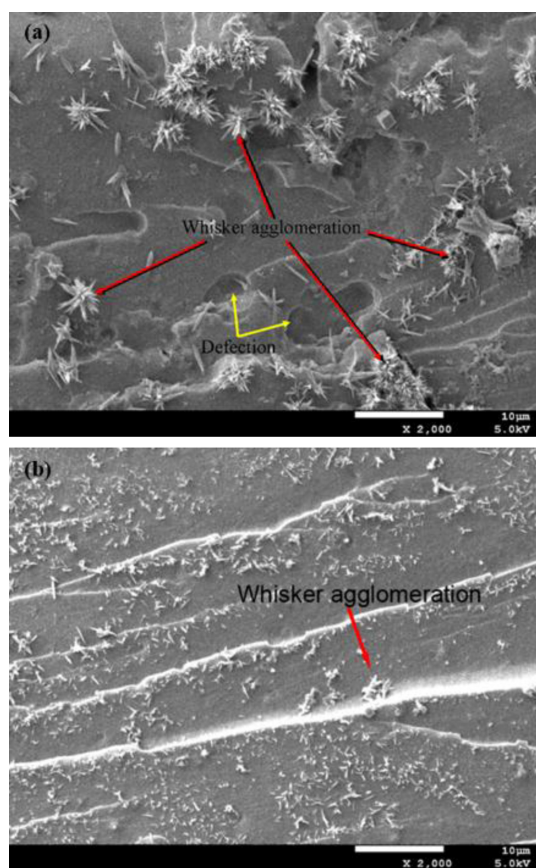


Figure 6. The micrographs of EP/20 wt% T-ZnOw. (a) mechanical mixing; (b) shearing mixing.

concentrated and has cleavage steps and tear ridges. There is no ductile fracture morphology such as a dimple and serpentine glide on the fracture surface. On the contrary, the surface is smooth and glassy. All above results indicate that the fracture of those composites is a typical brittle fracture.^{18,19}

As shown in Figure 3, fewer particles disperse on the micro-profile of 2 wt% T-ZnOw composites. Most particles mixed by the powerful shear mixer are coated by the matrix separately. The whiskers can disperse pressure, inhibit the large deformation in the local matrix and generate micro-cracks, which can improve the toughness of materials.

The whiskers are also completely coated by the matrix and isolate with each other in Figure 4. Few whiskers' tips overlap with another. The interfaces of the matrix and whiskers are vague, which indicates the fine compatibility of whiskers and matrix.²⁰ Micro-cracks appear around the whiskers' tips, as the whiskers' tips are nanoparticles and the surface activity is high. It is in very good agreement with the conclusion drawn by T. H. Hsieh *et al.*, they found that feather markings are visible as

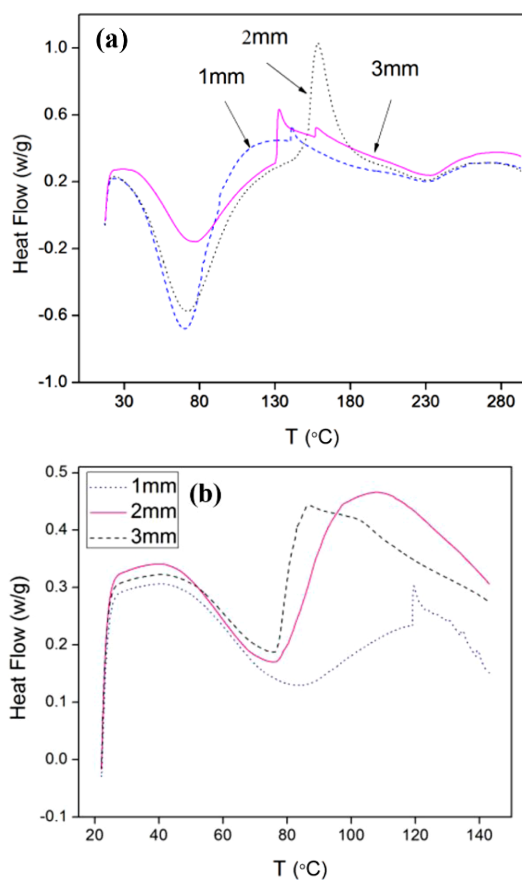


Figure 7. The curing curve of systems measured by DSC at the different mixing interval (a) pure EP; (b) EP/5 wt% T-ZnOw.

steps and changes of the level of the crack. These caused by crack forking due to the excess of energy around whiskers' tips associated with the rapid crack growth that occurs.²¹ In addition, the whiskers mixed by the powerful shear mixer disperse more uniformly than those just mixed by the traditional mechanical stir (Figure 4). Moreover, the smaller the mixing clearance is, the better the dispersion of whiskers will be.

As illustrated in Figure 5, the proportion of broken and coated whiskers grows. A part of the whiskers start to overlap with each other to form physical networks. The whiskers mixed by the powerful shear mixer were dispersed more uniformly comparing with those using mechanical mixing. However, a few aggregations emerge when the mixing clearance is 3mm. Much more aggregations appear in the matrix using mechanical mixing, but the whiskers mixed by the mixer still can disperse uniformly in matrix (Figure 6(b)) when the whiskers content is 20 wt% (Figure 6). According to these results, it is obvious that the whiskers can disperse more uniformly in matrix under powerful shearing field compared to those mixed

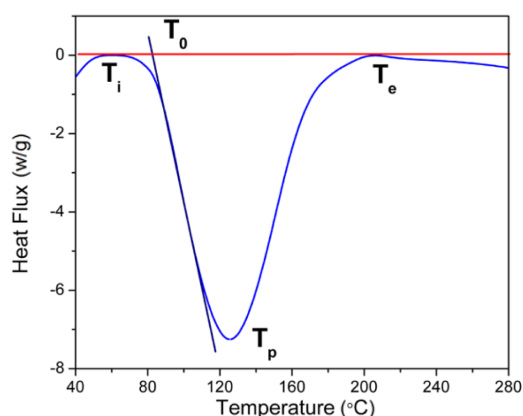


Figure 8. Four characteristic temperatures of curing reaction.

by traditional method.

The Effect of Powerful Shear Field on the Curing Reaction of T-ZnOw/EP. Figure 7 shows that the curing curves of pure epoxy resin and the systems containing 5 wt% T-ZnOw at different δ of the powerful shear mixer. As shown in Figure 7, a strong exothermic peak and a weak endothermic peak appear at 80 and 120 °C, respectively. Their reaction mechanisms seem not to be changed by the presence of T-ZnOw with the same characteristic of curves.²² The main reaction was chain growth in the initial stage of reaction for the composites, which based on the primary amine. At the moment, the reaction rate and heat release were higher until reaching to the second stage of curing reaction (three-dimensional polycondensation, gel and sol coexist). The absorbed heat was larger than the released heat due to a longer second curing reaction stage, leading to a weak endothermic peak on the curve of DSC.

As shown in Figure 8, four characteristic temperatures are defined: T_i , T_e , T_p and T_0 to investigate curing process expediently. They represent the initial reaction temperature, the reaction-end temperature, the peak temperature and the initial reaction of extrapolation temperature, respectively.

As observed from Table 2, the heat of the system reduces significantly after adding T-ZnOw into the epoxy matrix. The maximum reaction heat of compound system containing

5 wt% T-ZnOw is 193.1 J g⁻¹ lower than pure EP, which suggests that oxide whiskers can promote the curing process. The mechanism is in accordance with the curing reaction of T-ZnOw/EP composites, as the ZnO content increases, less energy is needed for the onset of co-operative motion of large parts of the polymer chains.⁹ In addition, T_0 for 5 wt% T-ZnOw/EP is 9.5 °C above the average for those of pure EP. As the oxide whiskers increase the viscosity of the compound adhesive solution and the silicon hydroxyl on the surface of particles experience an interaction with the epoxy molecules, which limits the diffusion movement of the resin molecules, the reaction needs to proceed at higher temperature. Just like the investigation of Yuan *et al.*,²³ the whiskers influence the curing behavior doubly,²⁴ which results in that the particles could promote the curing process of resin only at a higher temperature.

T_0 and T_p of pure EP and 5 wt% T-ZnOw/EP on the non-isothermal curing curves of DSC are made linear regression with δ , which is shown in Figure 9.

The slope of fitting straight line is a positive value, which indicates that the activation temperature of curing system rises with the enlargement of δ . It was caused by a series of reasons, the main reason was that the distribution and division of the whiskers in the epoxy matrix became poorer with the increasing of δ . Therefore more energy must be absorbed to make the system more uniform and support the diffusion movement of molecule, if the reaction state of molecule need to be attained. It indicates that the fine dispersion of the whiskers in matrix is in favor of the curing reaction under powerful shear field.

The Effect of Powerful Shear Field on Mechanical Properties of the Composites of T-ZnOw/EP. The Tensile Strength Analysis: Figure 10 shows the tensile strength of T-ZnOw/EP composites at various mixing clearances (δ) of the powerful shear mixer. 0 mm represents mechanical mixing. According to the curves, we note that the tensile strength of the composites with whiskers in the range of 2 to 10 wt% is the highest when the δ is 1 mm, whereas the tensile strength is lowest when δ is 3 mm. The smaller δ is, the higher the shear strength will be. The particles were dispersed in the epoxy

Table 2. The Data of DSC for EP and EP/5 wt% T-ZnOw

Mixing interval (δ) (mm)	EP			5 wt% T-ZnOw/EP		
	T_0 (°C)	T_p (°C)	ΔH (J g ⁻¹)	T_0 (°C)	T_p (°C)	ΔH (J g ⁻¹)
1	36.16	67.98	207.4	44.98	73.57	34.93
2	37.2	70.49	183	47.95	73.66	26.03
3	39.92	75.34	114.8	48.7	80.02	14.31

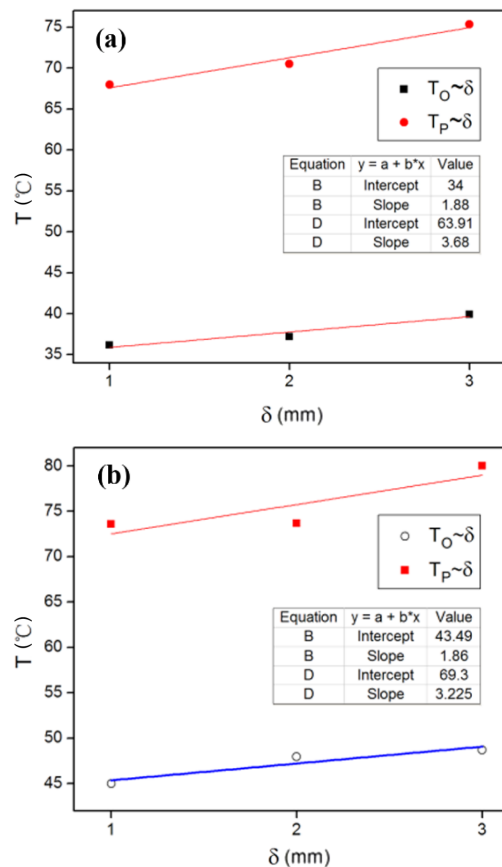


Figure 9. The fitting chart of T_0 and T_p . (a) pure EP; (b) EP/5 wt% T-ZnOw.

matrix uniformly, which could transfer stress quickly and evenly, made the materials have a compatibility of deformation.²⁵ That is why the tensile strength of the composites became better with the decrease of δ . However, the performances of the systems containing 20 wt% whiskers become better with the increasing of δ . The reason is that the particles in the matrix were interspersed with each other and stacked forming some aggregates (Figure 6) when the content of whiskers was too much, even if they had been mixed under powerful shear field. The aggregates acting as defects in matrix induced cracks, which made the materials damaged even in the condition of a small tension. In addition, the clearances around the aggregates dispersed much more uniformly with the decrease of δ , which made the cracks run through the matrix more easily.

Furthermore, as illustrated in Figure 10(b), the strength presents a trend of increasing first and then decreasing with the addition of T-ZnOw. The strength is the highest when the content was 5 wt% and the value is 37.26 MPa, which is higher

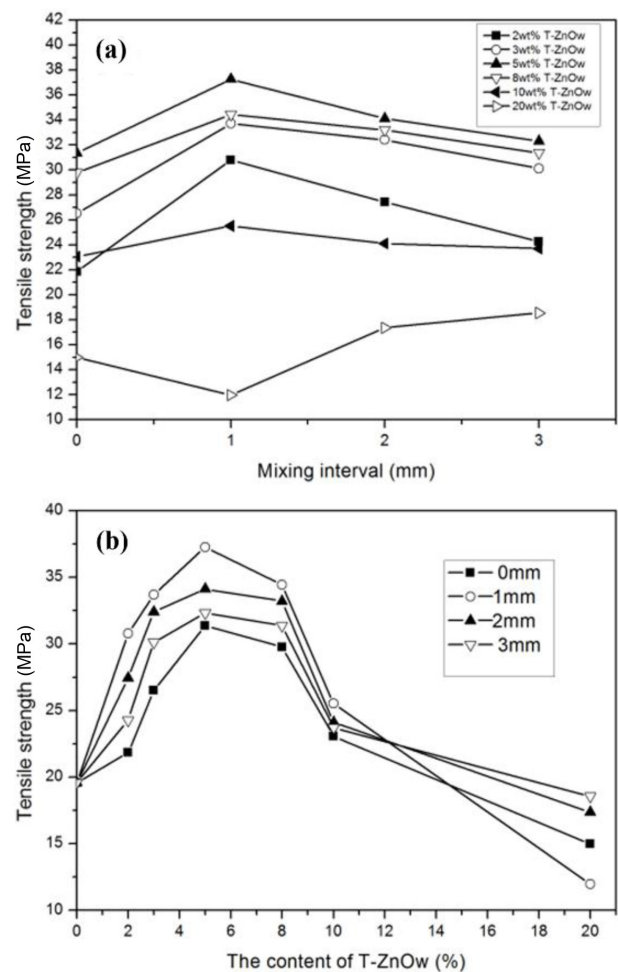


Figure 10. The tensile strength curves of EP/T-ZnOw. 0 mm represents mechanical mixing and 1, 2, 3 mm are mixing intervals of powerful shear mixer.

than the epoxy composites reinforced by short carbon fiber.²⁶ Since the length of T-ZnOw is in the scope of 10 to 50 μm , the matrix around 4.2×10^{-6} to $5.2 \times 10^{-4} \text{ mm}^3$ would be connected by only one whisker particle (Figure 4(c)). As a result, the mechanical strength of materials is improved significantly.

The Impact Strength Analysis: Figure 11 shows the impact strength of the composites containing different contents of whisker at various mixing clearances. As shown in Figure 11, the impact strength of materials decreases gradually with the enlargement of δ . Since δ is small, strong shearing, distribution and dispersion of whiskers are fine. The gap derived from the micro-cracks around the particles was difficult to connect, the matrix could withstand a greater impact load, and the toughness of composites was improved. After δ increased, the dispersion of whiskers became heterogeneous, the whiskers

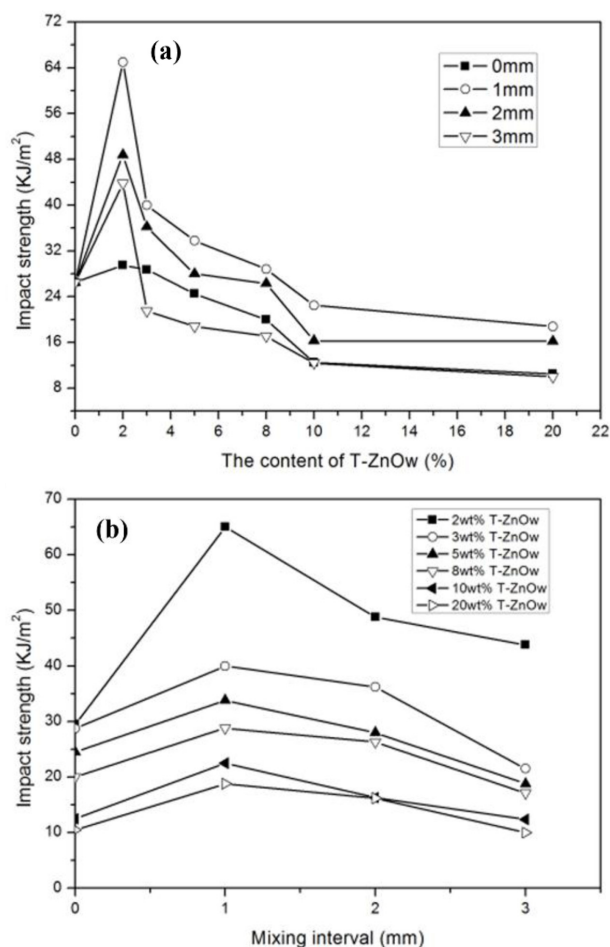


Figure 11. The impact strength curves of EP/T-ZnOw. 0 mm represents mechanical mixing and 1, 2, 3 mm are mixing intervals of powerful shear mixer

stack and some aggregates were emerged, which could weaken the strength.

In addition, the toughness of the materials was improved obviously after adding the whiskers into the matrix. The impact strength of systems is the highest when the content of T-ZnOw is 2 wt%, the value is 65 KJ/m². However, the impact strengths of epoxy resins modified with highly epoxidized polysiloxane and dendritic polysiloxane are just 44 and 33 KJ/m².^{20,25} Then the strength of the matrix declines sharply with the increasing of whiskers after the addition of T-ZnOw is over 2 wt%, which is in line with the changing rule of toughening brittle polymer materials with the rigid fillers. Shen *et al.* have studied the mechanical properties of epoxy resins modified by carbon nanotubes. They found that the movement of chain segment was restricted with the addition of much more carbon nanotubes, leading to a decreasing of the smaller inner area

surrounded by the impact load versus strain, which was the reason of decreasing impact strength.²⁷

In general, the effect of powerful shear field on mechanical properties of T-ZnOw/EP reflected in the morphology and distribution of whiskers and shearing aspect of the matrix molecule.

(1) In the mixing process, the smaller the clearance is, the stronger the shear action on T-ZnOw can be. T-ZnOw under powerful shear mixing elements produced different degrees of distributive and dispersive state. The smaller the shear clearance can produce a more intense mechanical shear force, such that the shear stress was generated in large whiskers making the whiskers destroyed, the distributive state of the whiskers became better; Meanwhile, the whisker particles were more uniformly dispersed in the epoxy matrix when the mixing clearance became smaller, the presence of such force transmission whiskers “crosslinking point” led to a higher strength of the composite material.

(2) The smaller mixing clearance made a strong shear acting on epoxy molecular chain in the mixing process, resulting in a higher degree of epoxy molecular chain scission. The resulting free radicals cause chain epoxy resin scission and long-chain reduction, and the three-dimensional network structure formed by crosslinking reaction became more intensive. Thus, the force can be dispersed throughout the material’s molecular chain network as far as possible. The more even force distribution in materials is, the higher mechanical properties can be.

The Heat-conducting Properties of T-ZnOw/EP: Figure 12 shows the thermal conductivity of the compound systems containing different whiskers contents at various mixing clearances. According to Figure 12, the thermal conductivity of pure epoxy is $0.204 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$. It is less than $1 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ for the compound systems and grows with the increasing of T-ZnOw content. These results are in accordance with Lewis-Nielsen model.²³ The thermal conductivity of all composites would be influenced mainly by heat transport of the highly conductive materials. The heat conductivity changes most obviously in Figure 12(a) when the content of fillers increases from 5 to 8 wt%.

Additionally, the thermal conductivity of the materials with the same component has a small difference at different mixing clearances (δ). The strong mixing process made whiskers uniformly dispersed, which failed to make the whiskers to contact with each other when the content was less than 5 wt%. After the content was over 5 wt%, the whisker particles overlap in

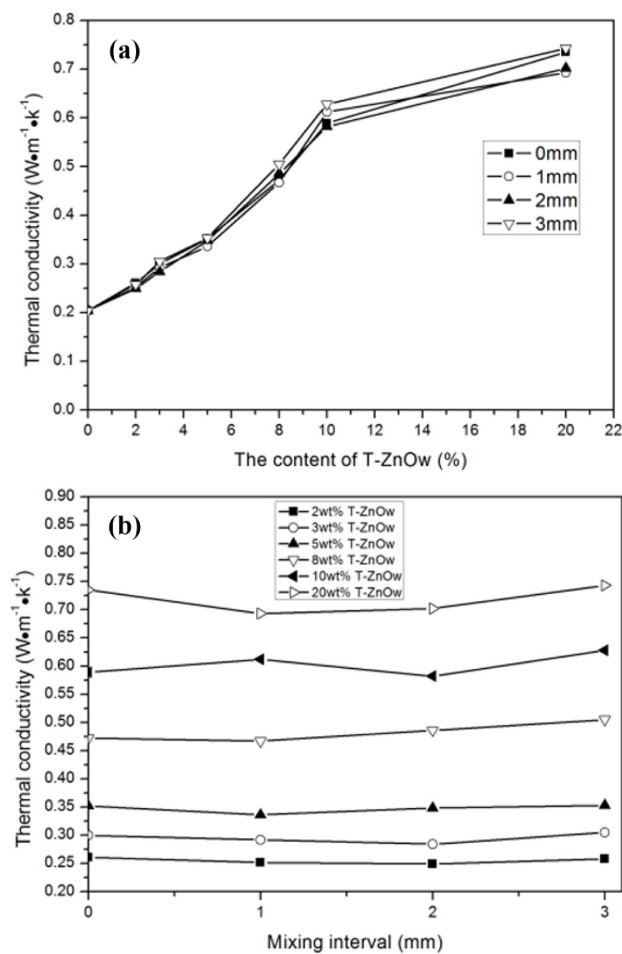


Figure 12. The thermal conductivity of the compound systems. 0 mm represents mechanical mixing and 1, 2, 3 mm are mixing intervals of powerful shear mixer.

matrix by themselves, and the strong mixing process promoted the formation of local heat networks by improving the dispersion of the whiskers, which also increased system thermal conductivity. When the content raises from 5 to 8 wt%, the thermal conductivity increases 38.8% on average. The number of physical network increased with the continually increasing of particles content, the volume becomes large, even the heat-conducting network is through the material. Then the contribution of whiskers on the heat transfer is dominant, the destruction and regeneration process of thermal weak the strong network of shear mixing effect.

The Dielectric Strength Analysis: Figure 13 shows that the dielectric strength of the compound systems containing different whiskers contents at various mixing clearances. Dielectric breakdown strength is defined as the highest voltage which samples can stand before they fail electrically, divided by sam-

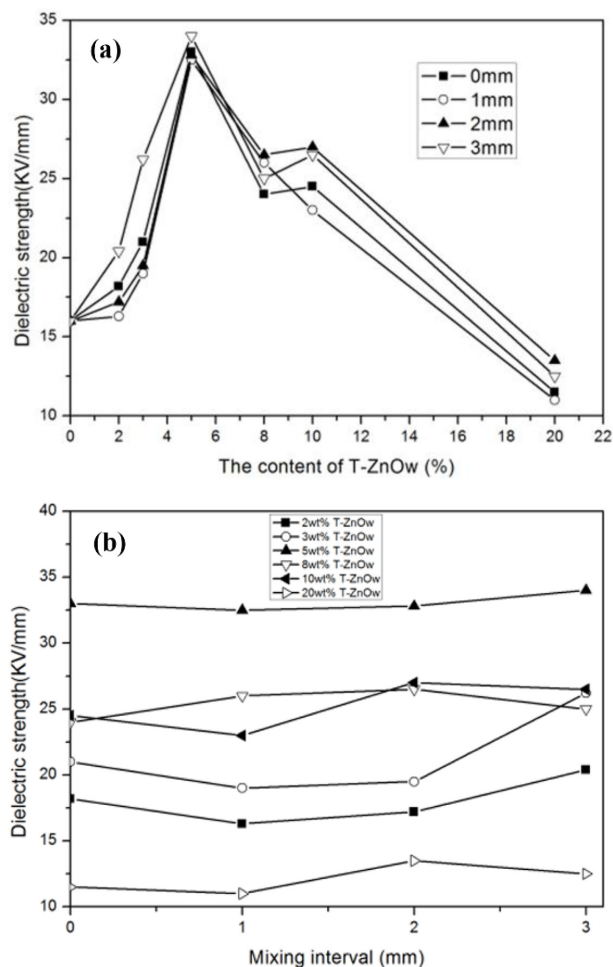


Figure 13. The dielectric strength of the compound systems. 0 mm represents mechanical mixing and 1, 2, 3 mm are mixing intervals of powerful shear mixer.

ple thickness.²⁸ As shown in Figure 13, the dielectric strength (E_b) of the compound materials increases first and then decreases with the addition of whiskers. The maximum value is 34 KV/mm when the content of whiskers is 5 wt%. Yu *et al.* investigated the dielectric strength of epoxy modified with Al₂O₃ particles and hyperbranched aromatic polyamide grafted Al₂O₃ nanoparticles. The values measured by them were 29.40 and 32.83 KV/mm, respectively.²⁹

E_b of the compound systems containing the same components rise gradually with the augment of δ when the content of whiskers is low (2~5 wt%). But after filling more particles (8~20 wt%), E_b grows first and then reduces with the increasing of δ . Under the effect of powerful shear force, abundant whiskers' tips were broken off, which made it easier for a single tip to move in the adhesive solution, promoting junctions' growth, which made the dielectric strength of the compound

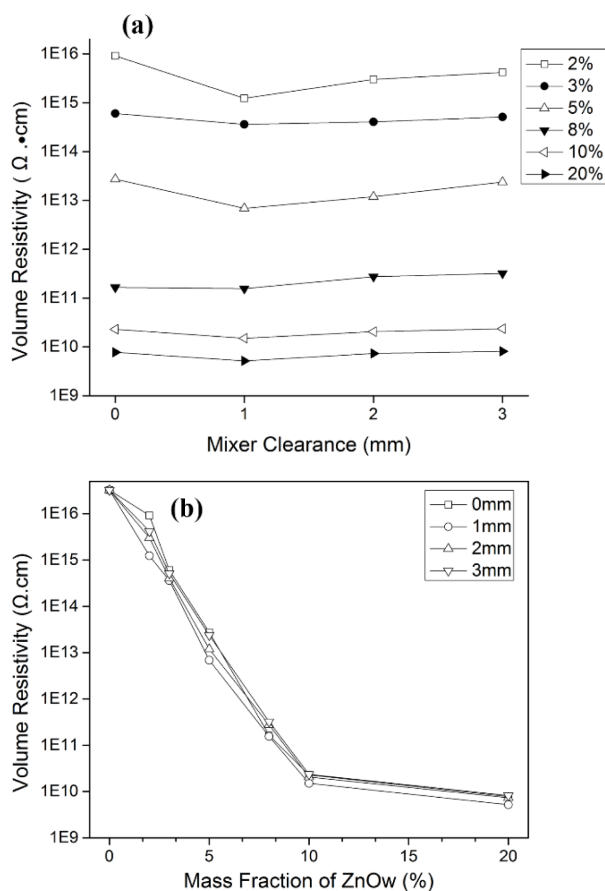


Figure 14. The volume resistivity of the compound systems. 0 mm represents mechanical mixing and 1, 2, 3 mm are mixing intervals of powerful shear mixer.

systems increase instead. However, the dielectric strength of the systems which contained more whiskers ($>8 \text{ wt}\%$) rises first and then declines with the increasing of δ caused by all comprehensive factors.

The Volume Resistivity Analysis: Figure 14 shows the volume resistivity of the compound systems containing different whiskers contents at various mixing clearances. As shown in Figure 14, the volume resistivity of the compound systems reduces significantly with the increasing of whiskers. The volume resistivity of materials is more than or equal to $10^{10} \Omega \cdot \text{cm}^{-1}$ and meets anti-static requirements of epoxy insulating material when the content of whiskers is less than or equal to 10 wt%. On account of the nanometer activity of whiskers' tips, the electronic vacancies around atoms in the surface of whisker' tips have certain potential barrier. The tips of the compound materials containing whiskers have vast charges gathering under the effect of electric field, a strong

electric field is excited correspondingly, generating a tunneling effect and some conductive paths in the matrix, which made the volume resistivity reduce rapidly. Shingh *et al.* added carbon nanotubes into epoxy matrix and found that the added carbon nanotubes formed new links within epoxy matrix, the volume resistivity declined with the increasing of carbon nanotubes content leading to the increase in the number of conducting links.³⁰ However, the volume resistivity was affected slightly by the powerful shear force. The powerful shear field made the particles disperse more uniformly and form the connected electrical network, which resulted in reducing the volume resistivity with the decreasing of δ .²³ But the influence of mixing clearance on the volume resistivity becomes weaker when the content of whiskers is higher ($\geq 8 \text{ wt}\%$), because abundant whiskers could generate a conductive network easily, even cause the particles less uniform.

Conclusions

1) After mixed by the powerful shear mixer, the dispersion of the whiskers in EP matrix have reached an ideal state. Larger particles are cracked by strong shearing force, resulting in a smaller and more uniform particle size.

2) The strong shearing action promotes the curing reaction without changing the mechanism. The curing reaction process tends to be gentle and takes a long time under the experimental condition.

3) The tensile strength of the composites is 2.4 times higher than pure EP when the T-ZnOw is 5 wt%. And the strength grows with the decreasing of δ .

4) The impact strength of the composites is 2.5 times higher than pure EP when the T-ZnOw is 2 wt%, and it reduces sharply with the whiskers increasing. In addition, it is advantageous to improve strength by reducing the mixing clearance.

5) The thermal conductivity can be improved by increasing T-ZnOw content, and destruction as well as regeneration of the conductive network of whiskers weak the effect of the powerful shear mixing.

6) The E_b can be improved by adding T-ZnOw and reaches the maximum at 2.1 times higher than pure EP when the content is 5 wt%.

7) The volume resistivity of pure EP is $3.2 \times 10^{16} \Omega \cdot \text{cm}^{-1}$, but it reduces substantially with the increasing of the T-ZnOw content. However, the influences of the powerful shear force is weak.

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