

Polystyrene Nanocomposite Materials—Preparation, Mechanical, Electrical and Thermal Properties, and Morphology

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ABSTRACT: This work prepared polystyrene resin nanocomposites with antistatic properties, by melt-blending polystyrene with nanoscale zinc oxide. The effect of nanoscale zinc oxide on the electrical and physical characteristics of polystyrene nanocomposites was investigated. Two kinds of nanoscale powders, spherical zinc oxide (s-ZnO) and zinc oxide whisker (w-ZnO), were selected. The coupling agents, vinyltriethoxysilane and phenyltriethoxysilane, were utilized to improve the compatibility between nanopowders and polystyrene resin. Adding spherical zinc oxide and zinc oxide whisker improved the antistatic characteristic of materials. The surface resistivities of s-ZnO and w-ZnO nanocomposite were significantly reduced, by mod-

ification with vinyltriethoxysilane and phenyltriethoxysilane. Adding zinc oxide nanopowder increased the flexural modulus and reduces flexural strength. Silane coupling agent improved the flexural properties of nanocomposite. The glass transition temperature and thermal degradation temperature of zinc oxide/polystyrene nanocomposite increased with ZnO content. Treatment with silane increased the glass transition temperature and thermal degradation temperature of composite. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 100: 508–515, 2006

Key words: zinc oxide; nanocomposites; surface resistivity; antistatic property

INTRODUCTION

According to the properties of antistatic materials, one can divide these materials into the following three categories:

1. Antistatic agents, such as nonionic surfactants or hydrophilic polymers, can be applied to the surface of products, or blended into raw materials, to form antistatic materials.^{1–3}
2. The polymer matrix can be blended with conductive additives, such as carbon black,^{4–6} metal powder,^{7,8} or conductive fibers,^{9,10} to form the antistatic polymer.
3. A material whose molecular structure is originally conductive, such as conducting polymer, can be used to prepare antistatic materials.^{11–14}

Antistatic agents can also be used as lubricants, which offer conductive channels. Antistatic agents, used as lubricants, can lower the coefficient of friction of plastics, and thus decrease the gathering of static

charges around the surface.¹ This function, however, cannot disperse the static charges. A second function, instead, can offer conductive channels, which help to disperse the charges. Besides, it can further release the charges. The main function of the antistatic agent is to offer conductive channels, in which a continuous water layer is usually formed because of the attachment of water vapor to the surface of the antistatic agents.² From the earlier discussion, it is known that proper absorptivity is a necessary property of antistatic agents. The conductivity of the water layer increases when the amount of ions increases. As a result, the greater the ionic property of the antistatic agents, the better the antistatic property of the composite materials.³

Owing to its excellent properties and lower price, carbon black, in past decades, has been blended into polymer materials, to provide the conductivity of materials.^{4–6} Although carbon black can serve as a good conductive additive, it will darken the surface of plastic products, a major defect that makes coloring of polymers more difficult.

Much research indicates that polymer composites, prepared as a mixture of conjugated conducting phase and classical nonconducting phase, can show quite good electrical conductivity at a relatively low content of the conducting phase.^{11,12}

Furthermore, some research shows that compositions of coating solutions with conductive metal oxide

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semiconductors, such as tin dioxide, indium(III) oxide, and zinc oxide, dispersed in a binder, may be either a nonconductive polymer or a polyelectrolyte. The coating layer gives excellent antistatic properties, even under low humidity.¹⁵

This study intended to prepare antistatic polystyrene nanocomposite materials, by incorporating nanoscale zinc oxides. Two kinds of zinc oxide were used in this study, namely, spherical zinc oxide and zinc oxide whisker. Two kinds of silane coupling agents, namely vinyltriethoxysilane (VTES) and phenyltriethoxysilane (PTES), were utilized to improve the compatibility of inorganic zinc oxide and polystyrene resin. They provide chemical (C—C bonding) and physical (π — π interaction) force, respectively. Electrical, mechanical, and thermal properties and morphology of the nanocomposite materials are discussed in this research.

EXPERIMENTAL

Materials

The general-purpose polystyrene pellet, with M_w 150,000 g/mol used, was received from the Chi-Mei Co., Taiwan. Two kinds of silane coupling agents, VTES ($C_8H_{18}O_3Si$) and PTES ($C_{12}H_{20}O_3Si$), were purchased from Lancaster Synthesis Ltd., Newgate, England and Acros Co., Janssens Pharmaceuticaan 3A 2440 Geel, Belgium, respectively. Two kinds of nanoscale powders, i.e., spherical zinc oxide and zinc oxide whisker, were used. Spherical zinc oxide (s-ZnO), about 31 nm in particle size and a specific surface area of about 11.1 m²/g, was purchased from Nanophase Technologies Corp., Romeoville, IL. ZnO whisker (w-ZnO), about 30 nm in particle size, specific surface area of about 40–60 m²/g and aspect ratio of about 5, was purchased from Nanjing High Technology Nano Co. Ltd, Nanjing, China.

Surface modified with coupling agents

Nanoscale zinc oxide powders were dried for 24 h, and then put into the reactor. VTES ($C_8H_{18}O_3Si$) and PTES ($C_{12}H_{20}O_3Si$) were used as the coupling agents and mixed with nanoscale zinc oxide powder, respectively. The weight ratio of the coupling agent and nanoscale zinc oxide powder was 20:1. THF was used as solvent and small amount acetic acid potassium was used as catalyst, and the mixture was stirred at 60°C for 24 h. After the reaction was complete, the mixture was separated by centrifuging three times, and then dried in a vacuum oven.

Preparation of zinc oxide/polystyrene nanocomposites

Zinc oxide/polystyrene nanocomposites were obtained by mixing polystyrene pellet with 0, 5, 10, 20,

and 30 wt % of ZnO filler by means of a HAKKE mixer, Rheomix 600p, Thermo Electron Co., Hamburg, Germany, at 160°C for 5 min. Following this process, the mixture was compressed into plaques for 15 min, at 160°C under 10 MPa.

Characterization property measurements

Fourier transform infrared spectroscopy

Fourier transform infrared (FTIR) spectra of the samples were recorded between 4000 and 400 cm⁻¹ on a Nicolet Avatar 320 FTIR spectrometer, Nicolet Instrument Co., USA. Thin films were prepared, by mixing the finely ground solid sample with powdered potassium bromide, and the mixture was pressed under high pressure at 8000–10,000 kg/cm². A minimum of 32 scans was signal-averaged, with a resolution of 2 cm⁻¹ at the 4000–400 cm⁻¹ range.

Scanning electron microscope

The morphology of the fractured surface of the nanocomposites was examined by scanning electron microscope (SEM; JEOL JSM 5300, Japan), to study the dispersion and size of nanoscale zinc oxide powder in the polymer.

Flexural strength and flexural modulus test

The flexural strength and flexural modulus were measured according to the testing procedure of ASTM D 790, with test specimen bar of 100 mm in length, 13 mm in width, and about 3 mm in thickness. The supporting span was 60 mm, and the rate of crosshead was 5 mm/min.

Surface resistivity test

The surface resistance of the composite was tested on a super high resistance meter, Megohmmeter SME-8311, Dkk-Toa Co., Tokyo, Japan, according to ASTM D 257, with a test thin film, 40 mm in length and 40 mm in width. The test was conducted with a voltage of 1000 V; charging for 30 s; temperature at 25°C; and relative humidity at 50%.

Differential scanning calorimeter analysis

The differential scanning calorimeter (DSC) analyses of the samples were carried out under N₂ atmosphere, with a TA Instruments DSC 10 (USA). Samples were first placed in a vacuum oven at 100°C for 24 h, before being sealed in an aluminum sample cell, held at 100°C for 10 min, and quickly put into liquid nitrogen until the temperature was -100°C, to obtain amor-

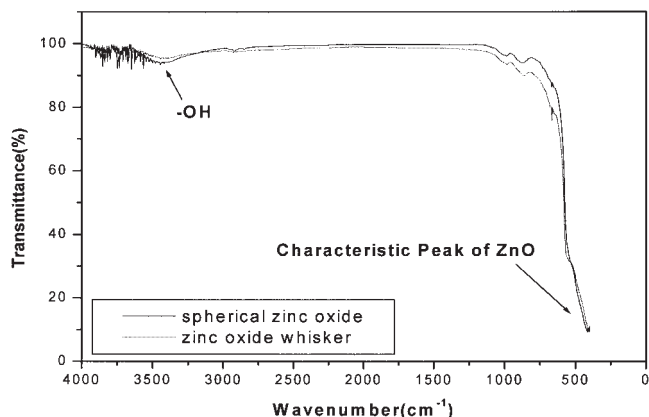


Figure 1 FTIR spectrum of nano scale zinc oxide.

phous samples. These amorphous samples were then heated again to 100°C at a heating rate of 10 K/min.

Thermogravimetric analysis

Thermal degradation properties of these nanocomposites were measured by TGA (DU-Pont-951) from room temperature to 600°C, with a heating rate of 10 K/min, under a N₂ atmosphere. The weight loss-to-temperature curves were recorded using 6–10 mg samples.

RESULTS AND DISCUSSION

Analysis of the functional group on the surface of zinc oxides

Figure 1 presents the FTIR spectra of nanoscale spherical zinc oxide and zinc oxide whisker. The spectra exhibit a weak absorption peak at 3400 cm⁻¹. The absorption peak ranges from 3000 to 3600 cm⁻¹, a range that corresponds to the hydroxy group, —OH, confirming that the surfaces of the two selected nanoscale zinc oxide powders contain —OH groups. Furthermore, strong absorption, which appears at about 400 cm⁻¹, is a diagnostic absorption peak of ZnO.

Surface resistivity of zinc oxide/polystyrene nanocomposite

Figure 2 depicts the surface resistivity of two zinc oxide/polystyrene nanocomposite materials. The surface resistivities fall, as the ZnO increases. Adding 30 wt % of spherical zinc oxide and zinc oxide whisker reduced the surface resistivities of materials from 1.0 × 10¹⁶ to 8.98 × 10¹² Ω/cm² and to 9.57 × 10¹⁰ Ω/cm², respectively. The amount of ZnO in polystyrene resin can be gradually increased to form a conductive network in materials.

The surface resistivity of the zinc oxide whisker/polystyrene nanocomposite materials is two orders

less, than that of spherical zinc oxide/polystyrene nanocomposite materials, perhaps for the following reasons:

Conductive network

Since the aspect ratio of zinc oxide whisker is five, higher than that of spherical zinc oxide, which is only one. Owing to the high aspect ratio of w-ZnO, this material is more efficient than s-ZnO in forming a conductive network, and hence, produces lower surface resistivity.

The charge concentrating effect at a pinpoint¹⁶

According to the results of Zhou et al.,¹⁶ the intensity of surface electrons of a whisker (whose aspect ratio is 10), is 400 times higher than that of general spherical particles. The conspicuous charge concentrating effect may cause the polymer matrix between the tips of the whiskers to break down, and the effect thus results in conductivity.

Tunnel effect¹⁶

Tunnel effect refers to the phenomenon of leakage current within the materials. The following equation regarding the charge-carrier and penetrating probability explains this phenomenon:

$$p = \exp \left[- \frac{2L}{\hbar} \sqrt{2m(V_0 - E)} \right] \quad (1)$$

where p is the penetrating probability, L is the distance between the charge-carrier, m is the mass of charge-carrier, and $\hbar = h/2\pi$; $h = 6.63 \times 10^{-34}$ J s is the

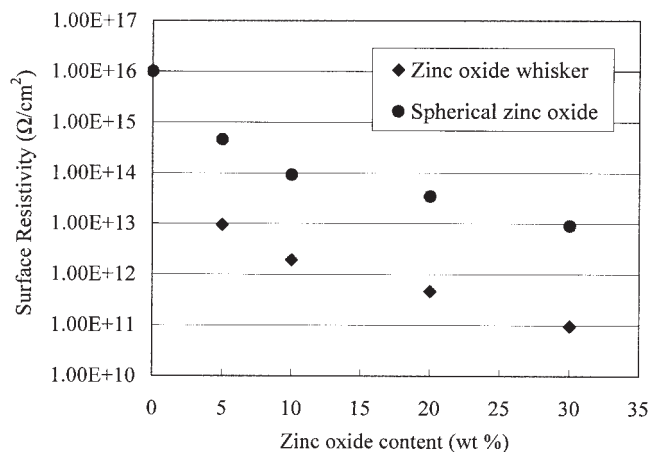


Figure 2 Surface resistivity of zinc oxide/polystyrene nanocomposites, with different types of zinc oxide nanoparticles.

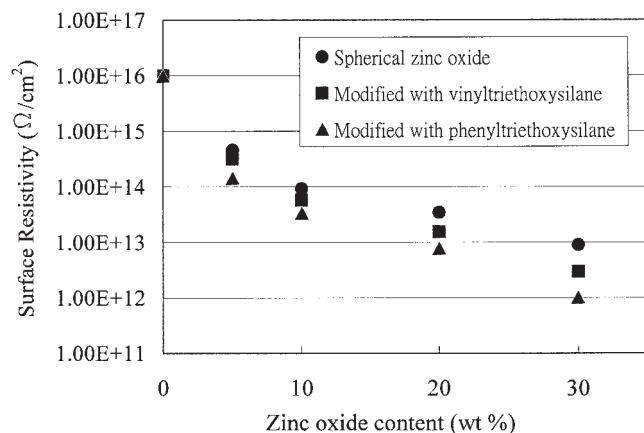


Figure 3 Surface resistivity of spherical zinc oxide/polystyrene nanocomposites with different modifiers.

Planck's constant and $(V_0 - E) = 1.6 \times 10^{-19}$ J in the electronic system.

Equation (1) reveals that penetrating probability is inversely proportional to the distance between charge-carriers. Restated, adding only a small quantity of whisker is not likely to cause the tunnel effect. The tunnel effect clearly affects the conductivity of the material, beyond a certain amount of addition.

Both Figures 3 and 4 indicate that polystyrene nanocomposites with modified spherical zinc oxide or zinc oxide whisker have lower surface resistivity than those to which no modified zinc oxide is added. Adding 30 wt % of spherical zinc oxide and zinc oxide

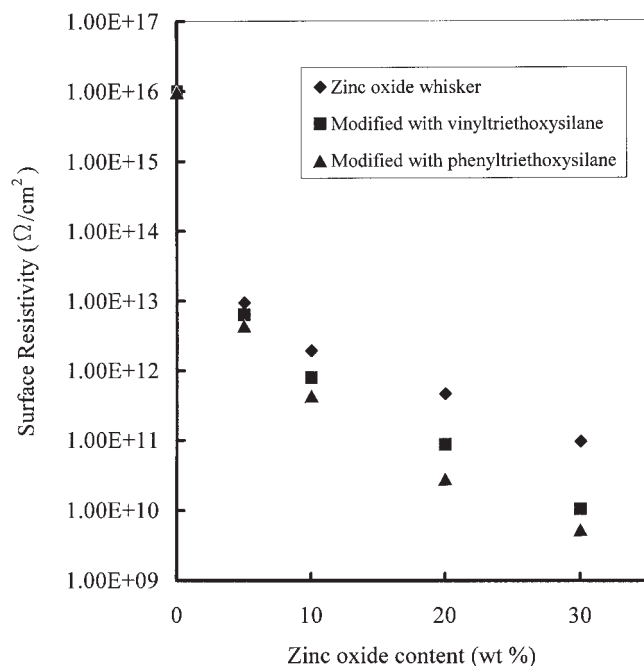


Figure 4 Surface resistivity of zinc oxide whisker/polystyrene nanocomposites with different modifiers.

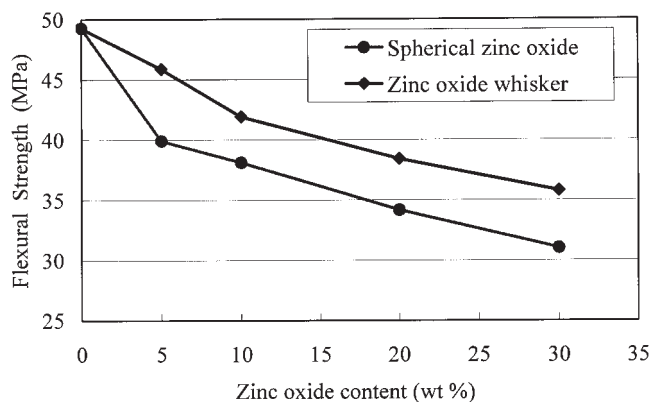


Figure 5 Flexural strength of zinc oxide/polystyrene nanocomposites, with different types of zinc oxide nanoparticles.

whisker, modified with VTES, decreased the surface resistivities of the materials from 1.0×10^{16} to 2.95×10^{12} Ω/cm^2 and 1.05×10^{10} Ω/cm^2 , respectively. The surface resistivities of both materials, with 30 wt % of spherical zinc oxide and zinc oxide whisker (modified with PTES), fell to 9.98×10^{11} and 5.27×10^9 Ω/cm^2 , respectively. The modified zinc oxide can be easily dispersed in resin, and so the compatibility between PTES and polystyrene resin is also considerably improved. The modified zinc oxide is more effective than VTES in reducing the surface resistivity of materials.

Flexural properties of zinc oxide/polystyrene nanocomposite

Figures 5–10 plot the flexural strength and the flexural modulus of various zinc oxide/polystyrene nanocomposite materials. The flexural modulus of materials increases, and the flexural strength of materials declines, as the ZnO content increases. The flexural modulus of materials, with 30 wt % of spherical zinc oxide and zinc oxide whisker, was 17.1 and 27.6%, respectively, higher than that of pure polystyrene resin. The flexural strengths of materials are 37.1 and 27.4%, lower than that of pure polystyrene resin. The dispersion is poor when the rigid nanoscale ZnO is added to the highly viscous polystyrene resin. Additionally, the high surface energy of nanoparticles may cause the nanoparticles to aggregate, and the stress may concentrate within the aggregation. Hence, the strengths of the nanomaterials were reduced, and the modulus was increased.

Figures 5 and 6 reveal that the polystyrene nanocomposite composite, with the 5, 10, 20, or 30 wt % zinc oxide whisker, has higher flexural strength and flexural modulus than do materials with the same proportions of spherical zinc oxide. Zinc oxide whisker has a unique structure, and so a whisker is easily inserted into the polystyrene resin, increasing the con-

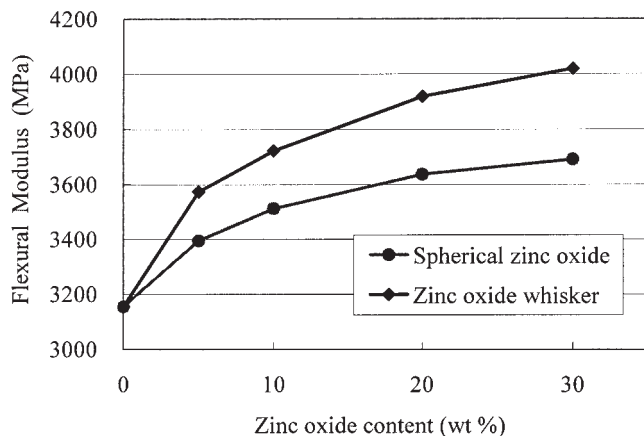


Figure 6 Flexural modulus of zinc oxide/polystyrene nanocomposites, with different types of zinc oxide nanoparticles.

tact area between the zinc oxide whisker and polystyrene resin. Moreover, from the perspective of reinforcement of the composite, zinc oxide whisker will increase the flexural strength and flexural modulus, because it has a higher aspect ratio.

Figures 7–10 demonstrate that adding only a small amount of the two modifiers can increase the flexural strength and flexural modulus of nanomaterials. However, the influence of the modifiers on the flexural strength and flexural modulus of materials may fall, as the quantity of modifier increases. The flexural moduli of both materials, with 30 wt % spherical zinc oxide and zinc oxide whisker (modified with VTES), are 19.7 and 30.5% higher, respectively, than those of pure polystyrene resin. The flexural strengths of both materials are 33.9 and 26.0% lower, respectively, than that of pure polystyrene resin. The flexural moduli of both materials, modified with PTES, are 20.6 and 32.7% higher, respectively, than those of pure polystyrene resin. The flexural strengths of both materials are 32.9

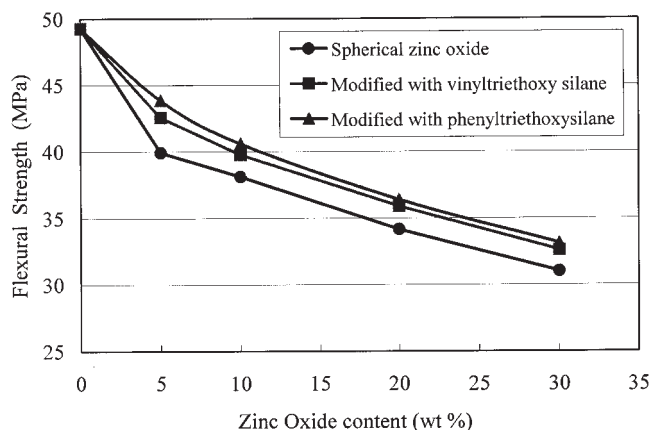


Figure 7 Flexural strength of spherical zinc oxide/polystyrene nanocomposites with different modifiers.

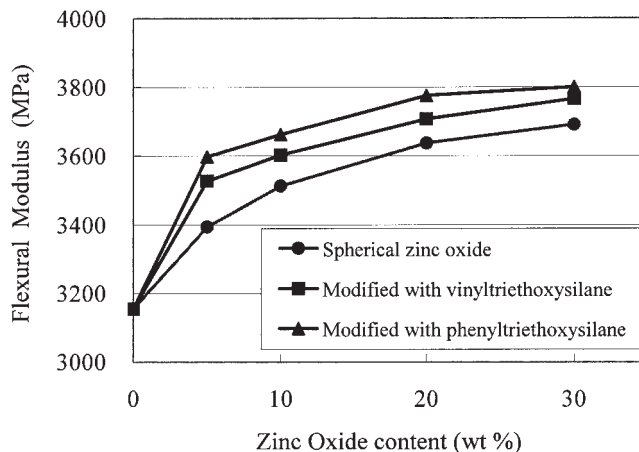


Figure 8 Flexural modulus of spherical zinc oxide/polystyrene nanocomposite with different modifiers.

and 25.4% lower, respectively, than that of pure polystyrene resin. These figures show that PTES more strongly affects the flexural strength and flexural modulus of the materials than VTES does. The chemical structure of PTES makes it more compatible with polystyrene resin. Finally, the modified nanoparticles exhibit strong molecular interaction with polystyrene polymer chains.

Morphology of zinc oxide/polystyrene nanocomposite

Figures 11 and 12 show the morphology of zinc oxide/polystyrene nanocomposites. The analyses of morphological characteristics reveal that the particle size of unmodified spherical zinc oxide (s-ZnO in Fig. 11) and unmodified zinc oxide whisker (w-ZnO in Fig. 12) are 200–300 nm, when the filler content reaches 10 wt %. If silane coupling agent is added, the particle size of zinc oxide (s-ZnO and w-ZnO) is smaller than 100 nm.

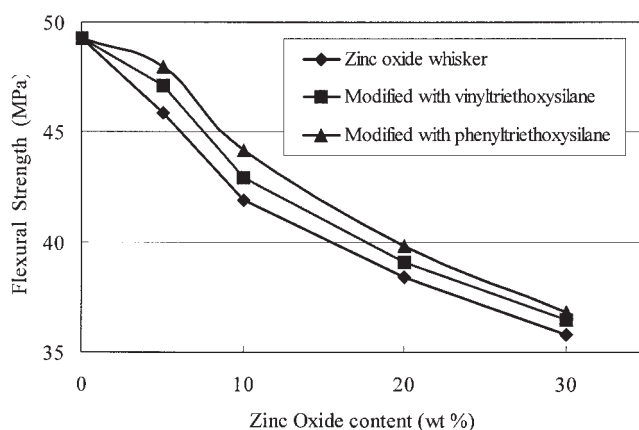


Figure 9 Flexural strength of zinc oxide whisker/polystyrene nanocomposite with different modifiers.

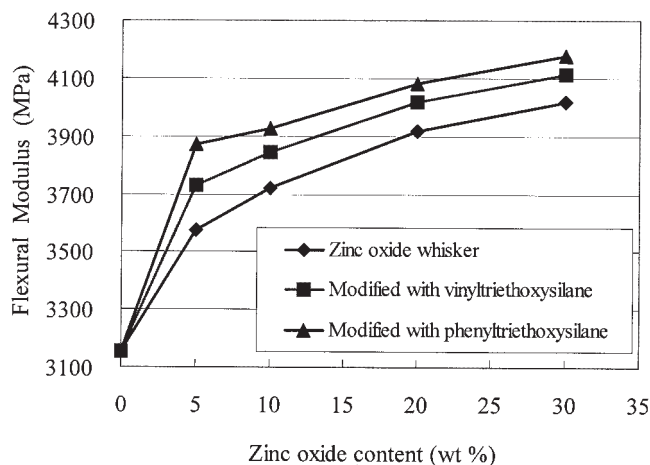


Figure 10 Flexural modulus of zinc oxide whisker/polystyrene nanocomposites, with different contents and types of modifiers.

When the ZnO content is 30 wt %, the particle size of modified ZnO (s-ZnO and w-ZnO) is around 200–300 nm. The results indicate that silane coupling agents are effective dispersion agents for ZnO.

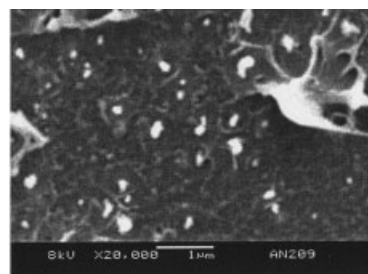
The glass transition temperature (t_g) of zinc oxide/polystyrene nanocomposite

Tables I–III summarize the DSC analyses of zinc oxide/polystyrene nanocomposites. The glass transition temperature of zinc oxide/polystyrene nanocomposite increased with ZnO content, because ZnO can restrict the segmental motion of polystyrene molecules and reduce the free volume of polymer chain folding. For instance, the glass transition temperature of zinc oxide/polystyrene nanocomposite increased from 87.6 to 93.6°C (spherical zinc oxide) and 94.7°C (zinc oxide whisker). Zinc oxide whisker has a larger aspect ratio than spherical zinc oxide, and so the restriction of molecular chain mobility exceeds that of spherical zinc oxide. Nanocomposites treated with VTES and PTES treatment exhibit higher glass transition temperatures. The T_g s of 30 wt % spherical zinc oxide with VTES and PTES treatment are 94.8 and 95.6°C, respectively. The T_g s of 30 wt % zinc oxide whisker with VTES and PTES treatment are 95.7 and 97.3°C, respectively. Zinc oxide is effectively dispersed in the polystyrene matrix treated with silane, and so the glass transition temperature of nanocomposite increases, as the silane is added.

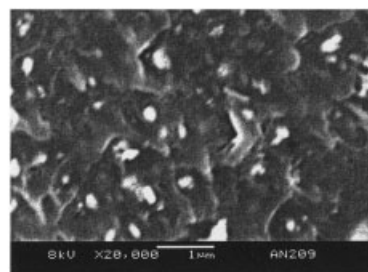
Thermal stability of zinc oxide/polystyrene nanocomposite

Tables IV–VI demonstrate the thermal decomposition behavior of zinc oxide/polystyrene nanocomposite. The results indicate that adding ZnO may increase the

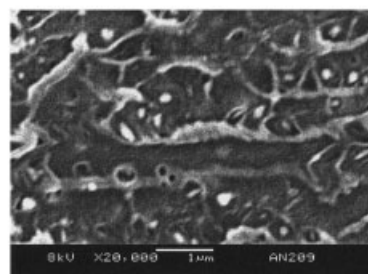
thermal degradation temperature. For example, the thermal degradation temperature associated with a weight loss of 10 wt % (T_{d10}) of the zinc oxide/polystyrene nanocomposite increased from 326 to 362°C (spherical zinc oxide) and 374°C (zinc oxide whisker). ZnO is an inorganic material that exhibits heat-resistance, and so the presence of ZnO can retard thermal degradation or delay the onset of the thermal degradation of the polystyrene. Zinc oxide whisker has a higher specific surface area (for the same addition amount) than that of spherical zinc oxide, and so the former has a greater contact surface with polystyrene, increasing the thermal stability of the nanocomposite. The nanocomposite, treated with VTES and PTES, exhibited higher thermal degradation temperatures. The T_{d10} s of 30 wt % spherical zinc oxide, treated with VTES and PTES, were 372 and 374°C, respectively.



(a) 10wt % s-ZnO / PS nanocomposite

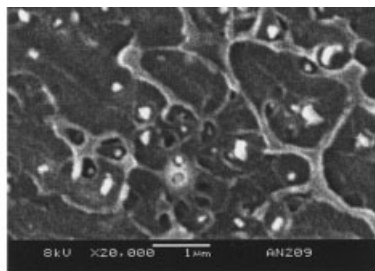


(b) 30wt % VTES modified s-ZnO / PS nanocomposite

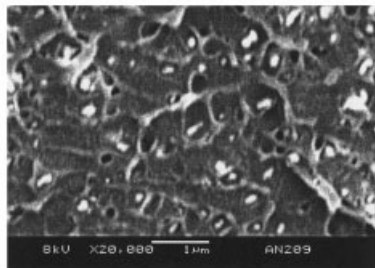


(c) 30wt % PTES modified s-ZnO / PS nanocomposite

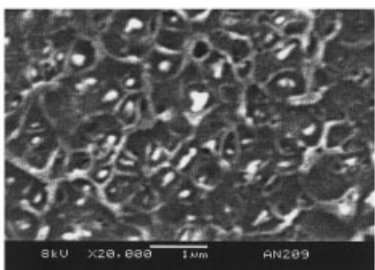
Figure 11 The SEM microphotographs of spherical zinc oxide/polystyrene nanocomposite ($\times 20,000$), with various contents and types of coupling agents.



(a) 10wt % w-ZnO / PS nanocomposite



(b) 30wt % VTES modified w-ZnO / PS nanocomposite



(c) 30wt % PTES modified w-ZnO / PS nanocomposite

Figure 12 The SEM microphotographs of zinc oxide whisker/polystyrene nanocomposite ($\times 20,000$), with various contents and types of coupling agents.

The T_{d10} s of 30 wt % zinc oxide whisker treated with VTES and PTES were 384 and 392°C, respectively. Zinc oxide is effectively dispersed in the polystyrene matrix treated with silane, and so the thermal degra-

TABLE I
Glass Transition Temperature of Zinc Oxide/Polystyrene Nanocomposite with Various Spherical Zinc Oxide Contents and Zinc Oxide Whisker Contents

Spherical zinc oxide content (wt %)	T_g (°C)	Zinc oxide whisker content (wt %)	T_g (°C)
0	87.6	0	87.6
5	92.0	5	93.0
10	92.5	10	93.6
20	93.0	20	94.1
30	93.6	30	94.7

TABLE II
Glass Transition Temperature of Zinc Oxide/Polystyrene Nanocomposite with VTES- and PTES-Modified Spherical Zinc Oxide

VTES-modified spherical zinc oxide content (wt %)	T_g (°C)	PTES-modified spherical zinc oxide content (wt %)	T_g (°C)
0	87.6	0	87.6
5	93.1	5	93.3
10	93.5	10	93.9
20	94.2	20	94.7
30	94.8	30	95.6

ation temperature of nanocomposite increases, as the silane is added.

CONCLUSIONS

1. The surface resistivity study reveals that adding spherical zinc oxide and zinc oxide whisker improved the antistatic characteristic of materials. Adding 30 wt % of spherical zinc oxide and zinc oxide whisker reduced the surface resistivities of materials from 1.0×10^{16} to $8.98 \times 10^{12} \Omega/\text{cm}^2$ and $9.57 \times 10^{10} \Omega/\text{cm}^2$, respectively. Adding the same quantities of spherical zinc oxide and zinc oxide whisker reduced the surface resistivities of the materials modified with VTES to 2.95×10^{12} and $1.05 \times 10^{10} \Omega/\text{cm}^2$, respectively. The surface resistivities of both materials, modified with PTES, were lower at 9.98×10^{11} and $5.27 \times 10^9 \Omega/\text{cm}^2$, respectively.
2. The flexural modulus of 30 wt % s-ZnO and w-ZnO nanocomposite was 17.1 and 27.6% higher, respectively, than those of pure polystyrene resin. The flexural strengths of 30 wt % s-ZnO and w-ZnO nanocomposite were 37.1 and 27.4% lower, respectively. The flexural modulus of 30 wt % s-ZnO and w-ZnO nanocomposite, modified with VTES, was 19.7 and 30.5% higher, respectively. The flexural strengths of 30 wt % s-ZnO and w-ZnO nanocomposite were 33.9 and 26.0% lower, respectively. The

TABLE III
Glass Transition Temperature of Zinc Oxide/Polystyrene Nanocomposite with VTES- and PTES-Modified Zinc Oxide Whisker

VTES-modified zinc oxide whisker content (wt %)	T_g (°C)	PTES-modified zinc oxide whisker content (wt %)	T_g (°C)
0	87.6	0	87.6
5	93.4	5	93.9
10	94.0	10	94.6
20	94.7	20	96.1
30	95.7	30	97.3

TABLE IV
Weight Loss (10 %) Temperature of Spherical ZnO and Zinc Oxide Whisker/Polystyrene Nanocomposite with Various Zinc Oxide Contents

Spherical zinc oxide content (wt %)	T_{d10} (°C)	Zinc oxide whisker content (wt %)	T_{d10} (°C)
0	326	0	326
5	335	5	338
10	345	10	353
20	356	20	361
30	362	30	374

flexural modulus of 30 wt % s-ZnO and w-ZnO nanocomposite, modified with PTES, was 20.6 and 32.7% higher, respectively. The flexural strengths of 30 wt % s-ZnO and w-ZnO nanocomposite were 32.9 and 25.4% lower, respectively.

- The morphology of zinc oxide/polystyrene nanocomposite showed that the particle size was 200–300 nm, when the filler content exceeded 10 wt % in the unmodified case. Adding the silane coupling agent reduced the particle size of zinc oxide powder, until the zinc oxide content of nanocomposite exceeded 20 wt %.
- The glass transition temperature of zinc oxide/polystyrene nanocomposite increased with ZnO content. Composite treated with the silane exhib-

TABLE V
Weight Loss (10 %) Temperature of Zinc Oxide/Polystyrene Nanocomposite with VTES- and PTES-Modified Spherical Zinc Oxide

VTES-modified spherical zinc oxide content (wt %)	T_{d10} (°C)	PTES-modified spherical zinc oxide content (wt %)	T_{d10} (°C)
0	326	0	326
5	343	5	352
10	353	10	362
20	361	20	368
30	372	30	374

TABLE VI
Weight Loss (10%) Temperature of Zinc Oxide/Polystyrene Nanocomposite with VTES- and PTES-Modified Zinc Oxide Whisker

VTES-modified zinc oxide whisker content (wt %)	T_{d10} (°C)	PTES-modified zinc oxide whisker content (wt %)	T_{d10} (°C)
0	326	0	326
5	365	5	370
10	371	10	375
20	377	20	382
30	384	30	392

ited a higher glass transition temperature than the composite not so treated. The thermal degradation temperature of zinc oxide/polystyrene nanocomposite increased with ZnO content. Silane treatment increases the thermal degradation temperature of composite.

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