

# Preparation and Characterization of Rare Earth Complex Europium<sup>3+</sup>-acrylate-1,10-phenanthroline Grafted onto Polypropylene

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**ABSTRACT:** Rare earth (RE) complex photoluminescence films were conventionally prepared by blending. The RE complexes were unevenly dispersed in the polymer matrix, thus readily quenching the fluorescence. An improved approach for preparing photoluminescence films was developed, in which the chemical or covalent bonding between the RE complex and polymer was introduced through melt-grafting polymerization. An RE complex (europium<sup>3+</sup>-acrylate-1,10-phenanthroline) [Eu(AA)<sub>3</sub>phen] was synthesized, and then grafted onto polypropylene (PP) in the melt phase to render the resin fluorescently active. The complex and the grafted PP were systematically characterized using various analytical tools including elemental analysis, TGA, IR, DSC,

WAXS, and fluorescence. The results indicated that the complex possessed favorable fluorescence and thermal stability, and the Eu(AA)<sub>3</sub>phen complex was successfully grafted onto the PP backbone. The findings from the DSC and WAXS measurements revealed that the complex acted as a nucleating agent, increasing the crystallization rate effectively and destroying the integrity of the crystallinity as well. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 1547–1552, 2006

**Key words:** rare earth complex; polypropylene; melt grafting; fluorescence; crystallization

## INTRODUCTION

Rare earth (RE) polymers generally refers to polymers with RE ions doped in or bonded on. Wolff and Pressley<sup>1</sup> first studied the fluorescence and laser properties of europium(thenoyltrifluoroacetone)<sub>3</sub> in poly(methyl methacrylate) in 1963 and pioneered the research on RE polymers. Since then, a number of functional RE polymers have been synthesized and the application areas of RE polymers are enormous. For instance, RE polymers have been extensively used as fluorescent, laser, protective, and optic materials.<sup>2</sup> There are two types of RE polymers: one is an RE chemical compound that is uniformly dispersed in monomers and polymers as a doping agent to produce doping-type RE polymers; the other is RE ions that are directly bonded onto the macromolecule chain to obtain bonding-type RE polymers.<sup>3</sup> Compared with bonding types, doping-type RE polymers have disadvantages

in applications because of the poor dispersion of RE complexes in the polymer matrix. Doping-type RE polymers also create a concentration quenching effect among fluorescence molecules, which makes the proportion of effective fluorescence molecules decrease; thus, the fluorescence intensity weakens and life declines.<sup>4</sup> In contrast, the preparation of bonding-type RE polymers with a high RE concentration, high light transmittance, and desirable properties is readily performed. At present, there are two approaches to synthesize bonding-type RE polymers: first, RE ions coordinate with polymers containing coordination groups; second, low molecular RE complexes take part in polymerization as monomers.<sup>5</sup>

Polypropylene (PP) is a widely used material because of its outstanding mechanical properties and low cost. Adding RE complexes to a PP matrix further extends its applications, rendering PP photoluminescent. Previous research<sup>6,7</sup> mainly focused on polymers doped with RE complexes. However, this type of RE polymer has limited applications because of the poor compatibility of RE complexes with polymers, whereas RE complexes combined with polymers by chemical bonds can address such a problem. Li et al.<sup>8</sup> grafted an RE complex containing acrylic acid (AA) onto polyethylene films by UV-induced polymerization and conferred the RE complex, having incorpo-

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rated polymer materials with chemical bonds. Zhang et al.<sup>9</sup> improved the thermal stability and hardness of poly(vinyl chloride) by grafting RE acrylate onto it in THF solution. To date, however, PP modification using the bonding RE complex approach has seldom been reported. This stimulated our interest in developing an effective approach to utilize this method for PP modification. Therefore, the key objectives of the research presented in this article were to synthesize a europium<sup>3+</sup>-AA-1,10-phenanthroline [Eu(AA)<sub>3</sub>phen] RE complex and then to melt graft the complex onto the PP backbone to produce a plastic resin emitting red fluorescence under UV. The modified PP was well characterized in order to establish the structure–property relation for the bonding-type RE polymers.

## EXPERIMENTAL

### Materials

Eu<sub>2</sub>O<sub>3</sub> (99.9 wt %) was purchased from Shanghai Yue-long Non-Ferrous Metals Limited and used without further purification. The PP homopolymer used in this study was PP Y1600 (Shanghai Petrochemical Co., Ltd., Shanghai, China). AA was obtained from Shanghai Puqiao Chemical Technology Graduate School and purified before use. Chemical grade diisopropylbenzene hydroperoxide (DCP), purchased from Shanghai Gaoqiao Petrochemical Co., Ltd., was used as an initiator. Analytical grade phen was obtained from Shanghai Chemical Reagent Co. Other analytical grade organic solvents were purchased from Shanghai Lingfeng Chemical Reagent Co., Ltd.

### Eu-AA-phen

AA (30 mmol) and phen (10 mmol) were dissolved in 70 mL of 95% ethanol aqueous solution. Then, 50 mL of EuCl<sub>3</sub> ethanol aqueous solution (10 mmol) was slowly added dropwise under constant stirring. The mixture solution was adjusted to pH 6.5 using an ethanol solution of NaOH (2M) and stirred at 70°C for 4 h. Then, it was filtered and washed repeatedly with 20% ethanol aqueous solution and dried under a vacuum at 50°C for 6 h. The complex was slightly soluble in alcohol, but fairly soluble in DMF and DMSO.

### PP-g-Eu(AA)<sub>3</sub>phen

The PP pellets were mixed with Eu(AA)<sub>3</sub>phen, DCP initiator, and other additives and fed into a Haaks mixer (Rheocord System 40) where the grafting reaction was conducted in the melt at 170°C. The grafted PP sample was dissolved in xylene at 140°C and purified by reprecipitating the solution with DMF. The purification was repeated twice. The precipitated sam-

ple was filtrated out and dried at 110°C in a vacuum oven.

### Characterization techniques

The content of Eu<sup>3+</sup> in the complex and the polymer was determined by EDTA titration. The carbon, hydrogen, and nitrogen contents were determined using an elemental analyzer (Elementar Vario EL III). The IR spectra were recorded on a Nicolet Magna 550 FTIR spectrophotometer, and the fluorescence spectra were obtained using a Hitachi F-4 spectrophotometer. TGA was carried out with a TGA-SDTA851E thermal analyzer at a heating rate of 10°C/min in an air atmosphere. DSC measurements were conducted with a PerkinElmer (DSC 7) thermal analyzer (Foster City, CA). The samples were heated to 220°C at a rate of 20°C/min under a nitrogen atmosphere and maintained at 220°C for 5 min. Afterward the samples were cooled to room temperature at a rate of 10°C/min and held at room temperature for 5 min to destroy anisotropy. The samples were measured from room temperature to 220°C at a rate of 20°C/min, held for 5 min, and then cooled to room temperature at the same rate. Both melting and crystallization parameters were obtained from the heating and cooling scans. WAXS was performed using a Rigaku WAXS diffractometer set at 40 kV and 30 mA with Cu K $\alpha$  radiation ( $\lambda = 1.54056 \text{ \AA}$ ).

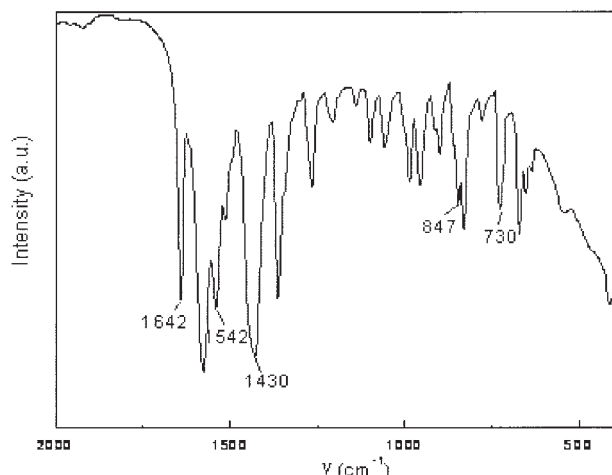
## RESULTS AND DISCUSSION

### Characterization of Eu(AA)<sub>3</sub>phen

#### Structure of Eu(AA)<sub>3</sub>phen

The C, H, and N contents of the complex determined by elemental analysis were 46.00, 3.04, and 4.96%, respectively; the Eu content determined by EDTA titration was 27.96%. The calculated values for the complex were 46.23% C, 3.127% H, 5.14% N, and 27.89% Eu. The IR spectrum of the Eu(AA)<sub>3</sub>phen complex (Fig. 1) shows the stretching vibration of phen at 1650 cm<sup>-1</sup> and the bent vibrations at 853 and 738 cm<sup>-1</sup> were shifted to a lower frequency at 1642, 847, and 730 cm<sup>-1</sup>, respectively, indicating that phen has coordinated with the Eu ion. The C=O and O—H stretching bands of free AA at 1702 and 930 cm<sup>-1</sup> are essentially absent in the complex. The strong bands at 1542 and 1430 cm<sup>-1</sup> correspond to the antisymmetric and symmetric stretching vibration of COO<sup>-1</sup> for AA,<sup>10</sup> which confirms AA has coordinated with the Eu ion. The experimental results above suggest that Eu(AA)<sub>3</sub>phen should be the reasonable formula for the complex. The reaction is inferred as follows:

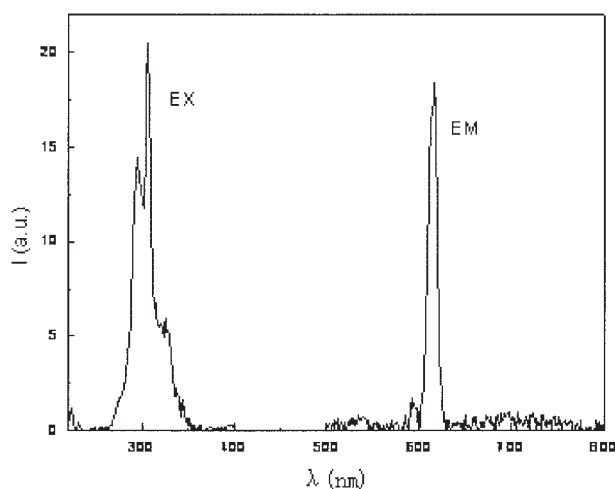




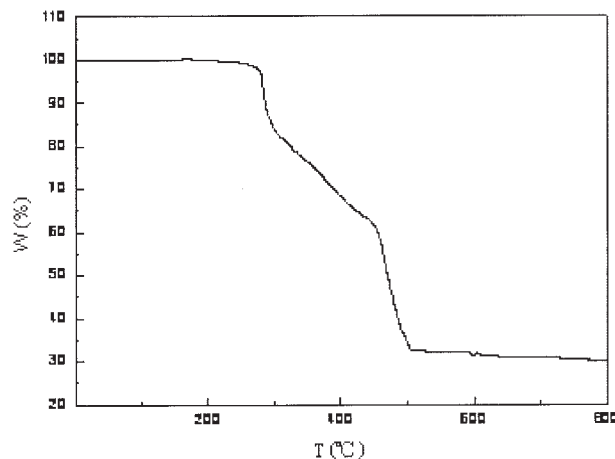
**Figure 1** The IR spectra of  $\text{Eu}(\text{AA})_3\text{phen}$  complexes.

### Fluorescence properties of $\text{Eu}(\text{AA})_3\text{phen}$

The excitation spectrum of  $\text{Eu}(\text{AA})_3\text{phen}$  (Fig. 2) shows that there were two excitation bands in the UV range at 295 and 330 nm. Being excited at these bands, the complexes all exhibit typical fluorescence of  $\text{Eu}^{3+}$  ions, which is attributed to the  $^5\text{D}_0 \rightarrow ^7\text{F}_j$  ( $j = 0, 1, 2, 3, 4$ ) transitions of the  $\text{Eu}^{3+}$  4f electron. The emission peaks are at 579, 592, 616, 650, and 695 nm successively; of these, the  $^5\text{D}_0 \rightarrow ^7\text{F}_2$  emission (about at 616 nm) is the most intense and emits the typical red fluorescence of  $\text{Eu}^{3+}$ . The f-f transition of free  $\text{Eu}^{3+}$  ions belongs to "parity forbidden." When  $\text{Eu}^{3+}$  ions coordinate with phen to form a complex, however, other parity compositions (such as 5d) are interloaded in it and the complex symmetry decreases, which results in the notable increase of the "induced electric dipole transition" and luminescence intensity. The ligand phen absorption band is in the range of 220–350



**Figure 2** The excitation ( $\lambda_{\text{em}} = 616$  nm) and emission ( $\lambda_{\text{ex}} = 330$  nm) spectra of  $\text{Eu}(\text{AA})_3\text{phen}$ .

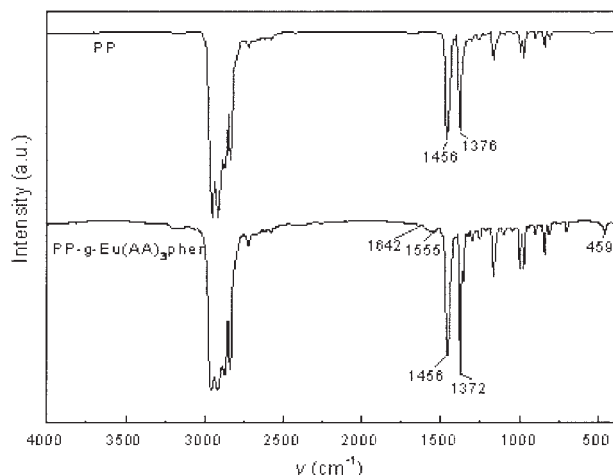


**Figure 3** The TGA curve of the  $\text{Eu}(\text{AA})_3\text{phen}$  complex.

nm whereas  $\text{Eu}^{3+}$  has weak absorption in this range. Accordingly, when excited at 295 or 330 nm, the complex obtaining intense fluorescence of  $\text{Eu}^{3+}$  shows that the energy of the excitation ray was absorbed by phen and was transferred to the excitation state energy level of central  $\text{Eu}^{3+}$  ions in complex molecules, and then was reduced to the lowest excitation state energy level ( $^5\text{D}_0$ ) through a nonradiative transition. Afterward, the  $^5\text{D}_0 \rightarrow ^7\text{F}_j$  transition occurred and generated fluorescence. Individual fluorescence and phosphorescence of ligands cannot be observed in the emission spectrum of the complex at room temperature, and the fluorescent intensity of  $\text{Eu}^{3+}$  is higher. This demonstrates the lowest triplet state energy level of ligands has better matching with the emission one ( $^5\text{D}_0$ ) of  $\text{Eu}^{3+}$ .<sup>11</sup> The emission spectrum of  $\text{Eu}(\text{AA})_3\text{phen}$  differs from that of  $\text{Eu}^{3+}$ - $\beta$ -diketone chelates.<sup>12</sup> Moreover, its emission peak at 616 nm is narrow and intense compared with that of  $\text{Eu}^{3+}$ - $\beta$ -diketone chelates, which shows that the color purity of  $\text{Eu}(\text{AA})_3\text{phen}$  is higher.

### Thermal property of RE complex $\text{Eu}(\text{AA})_3\text{phen}$

The TGA results for  $\text{Eu}(\text{AA})_3\text{phen}$  are shown in Figure 3. Note that the decomposition of the complex starts at 230°C and terminates at 590°C. When the temperature rises to near 285, 375, and 468°C, the weight-loss curve of the complex has obvious break points that prove that the decomposition is proceeding step by step. In the first decomposition temperature interval of 230–300°C, the mass loss is 13.2%, amounting to losing 1 AA molecule (theoretical mass loss = 12.4%). In the second decomposition temperature interval of 300–445°C, the mass loss is 24.3%, amounting to losing 2 AA molecules (theoretical mass loss = 24.8%). In the third decomposition temperature interval of 445–590°C, the mass loss is 32.1%, amounting to losing 1 phen molecule (theoretical mass loss = 32.0%). At



**Figure 4** The IR spectrum of PP-g-Eu(AA)<sub>3</sub>phen and PP.

the end of the reaction, Eu<sub>2</sub>O<sub>3</sub> was obtained and the total mass loss was 69.6% (theoretical value = 69.2%). This analysis further proves Eu(AA)<sub>3</sub>phen as the complex formula. The starting decomposition temperature of the complex at 230°C is higher than that of the melt reaction temperature at 170°C. As a result, the complex will no longer decompose in the course of melt grafting and facilitate the melt grafting reaction with PP.

### Characterization of grafting PP

#### Structure of PP-g-Eu(AA)<sub>3</sub>phen

Figure 4 shows the FTIR spectra of the original PP and the grafted PP. The typical absorption peak of methylene occurs at 1455 cm<sup>-1</sup>, whereas the methyl absorption peak for PP-g-Eu(AA)<sub>3</sub>phen was found to be shifted to 1372 cm<sup>-1</sup> and its intensity was higher than that of methylene. This shows that the partial methylene of PP had incorporated with Eu(AA)<sub>3</sub>phen. Meanwhile, compared to original PP, the C=O stretch vibration peak of the carboxylate in the grafted PP appears at 1555 cm<sup>-1</sup>. Moreover, the absorption peaks at 459 and 1642 cm<sup>-1</sup> represent Eu—O and C=N, respectively, which originate from the Eu(AA)<sub>3</sub>phen complex. These results provide evidence of the PP being grafted with the Eu(AA)<sub>3</sub>phen complex.

#### Fluorescence properties of PP-g-Eu(AA)<sub>3</sub>phen

Figure 5 denotes the fluorescence spectra of the grafted PP with grafting rates at 1.35, 2.22, 2.90, and 3.54%, which were excited at 270 nm. At this excitation wavelength the grafted PP emits visible and intense red fluorescence whereas the original PP cannot emit any color fluorescence. The modified PPs with different amounts of grafting all exhibit typical Eu<sup>3+</sup> fluo-

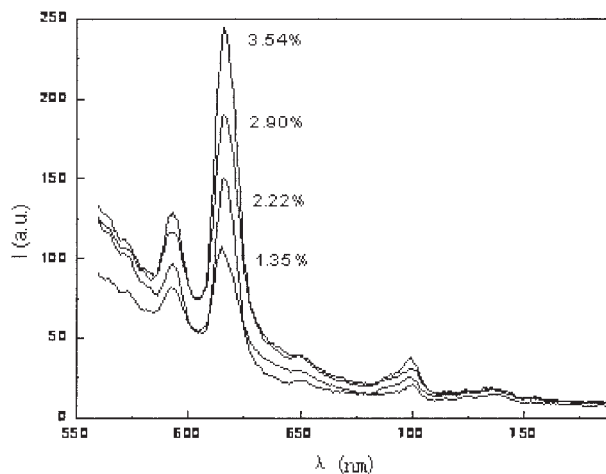
rescence that corresponds to the <sup>5</sup>D<sub>0</sub> → <sup>7</sup>F<sub>*j*</sub> (*j* = 0, 1, 2, 3, 4) transitions of the Eu<sup>3+</sup> 4f electron. Moreover, their emission peaks at 579, 592, 616, 650, and 695 nm successively, for which the <sup>5</sup>D<sub>0</sub> → <sup>7</sup>F<sub>2</sub> emission (about at 616 nm) is the most intense and emits the typical red fluorescence of Eu<sup>3+</sup>.<sup>13</sup>

Figure 5 also shows that the fluorescence intensity of grafting PP improves as the grafting rate increases and reaches a maximum at 3.54% without typical emission concentration quenching. The possible reasons for this are that Eu(AA)<sub>3</sub>phen is uniformly distributed along the PP chains and the distance between Eu<sup>3+</sup> is relatively large, leading to the weak concentration quenching.

Moreover, compared to the Eu(AA)<sub>3</sub>phen complex, the excitation wavelength of grafting PP was found to be shifted from 330 to 270 nm, which could be caused by the change of the coordination environments of the RE ions. This phenomenon further confirms the fluorescent RE complexes have incorporated with PP by chemical bonds instead of simply doping with PP.

#### DSC analysis of PP-g-Eu(AA)<sub>3</sub>phen

The results of the DSC heating and cooling scans for PP and PP-g-Eu(AA)<sub>3</sub>phen are shown in Figure 6. The crystallization parameters of the samples obtained from the heating and cooling scans are given in Table I. The  $\Delta H_f$  and  $\Delta H_c$  values are the melting and crystallization enthalpies per gram of samples, respectively. The crystallization peak temperature  $T_c$  represents the temperature at the maximum crystallization rate. The  $T_c$  of PP-g-Eu(AA)<sub>3</sub>phen is about 7°C higher than that of the PP homopolymer. The observed crystallization behavior of PP-g-Eu(AA)<sub>3</sub>phen can be explained in terms of heterogeneous nucleation. In a certain range of the grafting process, Eu(AA)<sub>3</sub>phen



**Figure 5** The emission ( $\lambda_{\text{ex}} = 270$  nm) spectrum of PP-g-Eu(AA)<sub>3</sub>phen.



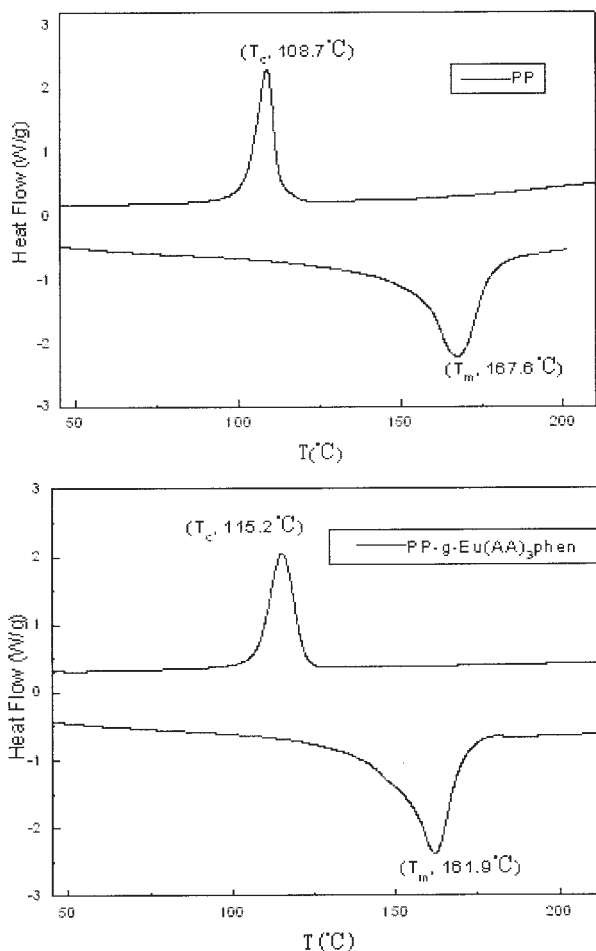


Figure 6 The DSC curves of PP and PP-g-Eu(AA)<sub>3</sub>phen.

functions as a heterogeneous nucleating agent for the PP homopolymer. The half-crystallization and crystallization induction period of the grafted product were all shortened, which accelerated the nucleation rate and allowed the nucleation to occur at a higher temperature. The change in the crystallization peak width ( $\Delta T_c$ ) represents the variation in the homogeneity of the crystallization, and a narrower value of  $\Delta T_c$  means a faster nucleation rate. The  $\Delta T_c$  value for PP-g-Eu(AA)<sub>3</sub>phen is about 5°C narrower than that of the PP homopolymer (46.2°C), which further proves Eu(AA)<sub>3</sub>phen acts as a nucleating agent in the crystallization process and effectively improves the crystallization. The  $\Delta H_c$  of PP-g-Eu(AA)<sub>3</sub>phen almost re-

mains the same as that of PP (97.3 J/g), suggesting that the crystallinity does not change significantly.

The melting temperature ( $T_m$ ) of PP-g-Eu(AA)<sub>3</sub>phen is lower than that of PP (167.6°C), which is due to the fact that the melt grafting destroyed the regularity of the PP crystal an chain degradation occurred in the PP in the process of melt grafting. Moreover, the  $\Delta H_f$  and the melting peak width ( $\Delta T_m$ ) of PP-g-Eu(AA)<sub>3</sub>phen are slightly higher than that of the PP homopolymer, which is likely attributable to the heterogeneous nucleation of Eu(AA)<sub>3</sub>phen for PP crystallization having the advantages of accelerating crystallization and increasing the amount of spherulites per volume of the sample. These results indicate that the crystallites in the PP-g-Eu(AA)<sub>3</sub>phen are less perfect than those in the PP homopolymer.<sup>14</sup>

#### WAXS analysis of PP-g-Eu(AA)<sub>3</sub>phen

The crystallite size ( $D$ ) vertical to the lattice plane ( $hkl$ ) can be obtained by the Scherrer equation<sup>15</sup>:

$$D = k\lambda / \beta \cos \theta \quad (1)$$

where  $k$  is the factor of the crystal figure, assuming  $k = 0.89$ ;  $\lambda$  is the wavelength of the X-ray ( $\lambda = 1.54056 \text{ \AA}$ , assuming  $\text{\AA} = 57.3$ );  $\theta$  is the diffraction angle; and  $\beta$  is equal to  $(B^2 - b_0^2)^{1/2}$ , where  $B$  is the width at half-height of the diffraction peak and  $b_0$  is the broadening factor of the instrument. If we take no account of the lattice distortion, the equation can be simplified as follows:

$$D = K\lambda / \beta \cos \theta = 0.89 \times 1.54056 \times 57.3 / B \cos \theta \quad (2)$$

The WAXS results for PP and PP-g-Eu(AA)<sub>3</sub>phen are shown in Table II and Figure 7. In Table II,  $I$  is the relative intensity of the crystalline peaks,  $B$  is the peak width at half-height, and  $d_{hkl}$  is the space between lattice planes ( $hkl$ ). As shown in Table II and Figure 7, both curves exhibit scattering angles at  $2\theta = 13.920$ ,  $16.660$ , and  $18.400$ , indicating an  $\alpha$ -phase monoclinic structure.<sup>14</sup> The  $d$  of the grafted PP has a slight change whereas  $D$  decreases. The results can be explained by the heterogeneous nucleation of Eu(AA)<sub>3</sub>phen for PP crystallization. This process accelerates the crystalliza-

TABLE I  
Crystallization and Melting Parameters of PP and PP-g-Eu(AA)<sub>3</sub>phen Obtained from DSC

Eu(AA) <sub>3</sub> phen content (%)	$T_c$ (°C)	$\Delta T_c$ (°C)	$\Delta H_c$ (J/g)	$T_m$ (°C)	$\Delta T_m$ (°C)	$\Delta H_f$ (J/g)
0	108.7	46.2	97.3	167.6	69.0	92.3
2.9	115.2	41.2	98.3	161.9	71.0	97.6

TABLE II  
Crystal Parameters of PP and PP-g-Eu(AA)<sub>3</sub>phen from WAXS

Eu(AA) <sub>3</sub> phen content (%)	<i>hkl</i>	2θ	<i>d</i> <sub><i>hkl</i></sub> (Å)	<i>I</i> (%)	<i>B</i> (°)	<i>D</i> (Å)
0	110	14.10	6.28	100.0	0.43	181.7
	040	16.90	5.24	44.1	0.37	209.0
	130	18.56	4.78	31.9	0.39	203.7
2.90	110	14.04	6.30	42.0	0.63	124.8
	040	16.80	5.27	100.0	0.56	142.1
	130	18.50	4.79	17.4	0.57	139.2

tion in which the growth of grains is restricted because they crush each other in the course of growing. That indicates the crystallization of PP-g-Eu(AA)<sub>3</sub>phen is less perfect than that of the PP homopolymer. As mentioned previously, the overdosing of Eu(AA)<sub>3</sub>phen onto the backbones of PP tends to decrease the crystallinity as well as the perfectibility of spherulites. In addition, the WAXS diffractogram of grafted PP changed with respect to that of PP. This could be because the acting force is not used uniformly during the process of preparing films and it changed the orientation peaks of the molecules.

## CONCLUSIONS

An Eu(AA)<sub>3</sub>phen RE complex was synthesized that possessed favorable fluorescence and heat stability. The complex was successfully grafted onto the backbones of PP in the presence of the radical initiator DCP through melt-grafting polymerization. As a result, a novel bonding-type RE polymer material, emitting red fluorescence under UV and containing favorable workability, was created. This material possesses desirable fluorescence intensity, color purity, low cost, and facile preparation. Thus, it has great potential as an anti-foggy film, photoluminescence display material, lighting transformation agricultural film, and so forth. The DSC and WAXS characterization results demonstrated that the functional groups introduced into PP also acted as nucleating agents that promoted the crystallization of PP and destroyed the integrity of the crystalline phase simultaneously.

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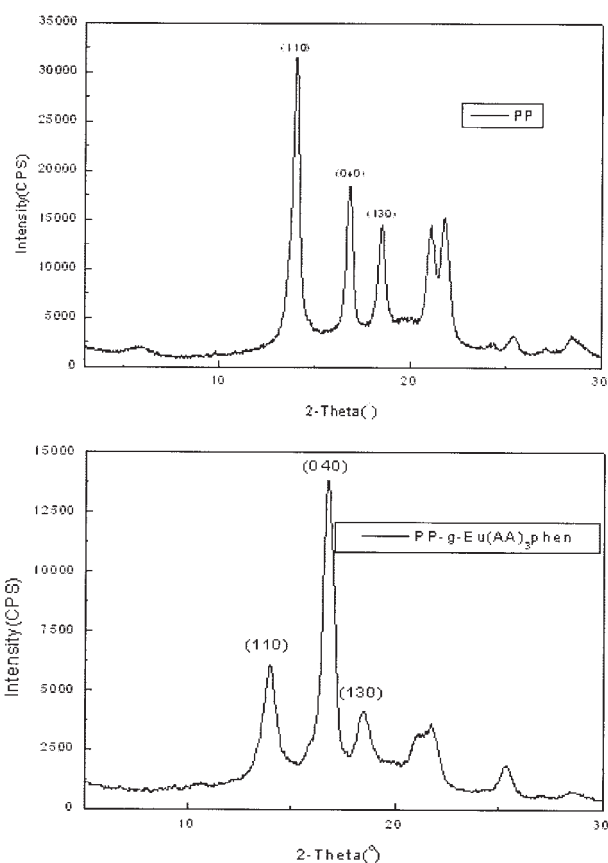


Figure 7 A WAXS diffractogram of PP and PP-g-Eu(AA)<sub>3</sub>phen.