Fire-Resistant Effect of Nanoclay on Intumescent Nanocomposite Coatings

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ABSTRACT: The aim of the study is the development of an intumescent nanocomposite coating to provide fire protection for the metallic substrate. Acrylic nanocomposites containing nanoclay and relative intumescent nanocoatings are prepared. The effect of nanoclay on the thermal degradation of an intumescent nanocomposite coating is analyzed by using differential thermal analysis, thermogravimetry, and X-ray diffraction. The influence of the added content of nanoclay on fire performance is studied by a fire protection test and measurements of the limiting oxygen index and effective thermal conductivity. The distribution of nanoparticles in the acrylic nanocomposite is characterized by transmission electron microscopy. The flame-retardant efficiency of the intumescent nanocomposite coating is improved by 1.5% well-distributed nanoclay particles. However, 3% nanoclay produces a negative effect on the fire performance of the coating. Fire protection tests and scanning electron microscopy observations reveal that the fire-retardant property of a conventional intumescent coating is destroyed by aging, whereas the nanocomposite coating modified with 1.5% nanoclay demonstrates good aging and fire resistance. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 103: 1681–1689, 2007

Key words: intumescent coatings; nanoclay; nanocomposite coating; fire performance; antiaging

INTRODUCTION

The exposure of steel structural members to high temperatures reduces their strength and rigidity and may lead to structural collapse of the steel when the critical temperature of the structure is reached. The steel's structural safety in a fire is assured when the temperature of the steel members remains less than the critical structural temperature. Fire safety of steel structural members may be achieved by use of intumescent coatings. Conventional intumescent coatings consist of ammonium polyphosphate, pentaerythritol, melamine, acrylic resin, and fillers. These conventional coatings have good expanding effects and fire-retardant properties, so they are widely used to provide fire protection for steel structural members.^{1,2} The char structure formed from the intumescent coating is easily damaged at high temperature, and the intumescent coating is vulnerable to aging. Consequently, it is necessary to improve the fire retardancy and durability of the intumescent coating.

Numerous studies have shown that introducing nanoparticles into polymers or coatings can greatly improve some properties such as the abrasion resistance, scratch resistance, UV resistance, fire resistance, and so forth. Nanoscale fillers added into the

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The focus of this work is to develop nanocomposite coatings modified by nanoclay and to improve the flame-retardant and antiaging properties of the nanocomposite coatings. We examine the thermal degradation and fire performance of three types of flame-retardant coatings in which different contents of nanoclay are incorporated.

EXPERIMENTAL

Materials

Methyl methacrylate (MMA) and styrene (St) were supplied by Shenyang Chemical Reagent Co. Nanoclay



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1.42E containing 105 mequiv quaternary ammonium ions/100 g clay (primary particle size = 20 ± 5 nm, length/thickness ratio = 200-300, specific surface area = $750 \text{ m}^2/\text{g}$) was obtained from Nanocor Inc. The structure of quaternary ammonium ions is N⁺(CH₂CH₂OH)₂(CH₃)HT, where HT is composed of 65% C₁₈H₃₇, 30% C₁₆H₃₃, and 5% C₁₄H₂₉. Ammonium polyphosphate [APP50, (NH₄PO₃)_n, n = 50, soluble fraction in H₂O < 1 wt %], pentaerythritol, and melamine were purchased from Xingxing Co.

Preparation of acrylic/clay nanocomposites

(Zhejiang, China).

The polymerization of MMA and St (MMA/St ratio = 1) was initiated by 1% 2,2'-azobisisobutyronitrile (AIBN) at 80–90°C. The number-average (M_n) and weight-average (M_w) molecular weights of the obtained acrylic resin were determined by gel permeation chromatography (GPC). The M_n and M_w values of the thermoplastic acrylic resin were 34,132 and 73,680, respectively.

Nanoclay was dried in a vacuum oven at 80°C for 48 h. Then, 5 and 10% dried nanoclay was mixed with MMA and St (MMA/St ratio = 1) at 75–80°C for 1 h under a nitrogen sweep and then initiated by 1% AIBN. The polymerization reaction temperature quickly increased to 120°C and remained stable at 120°C for 1 h. The polymer–clay reaction was used to prepare the acrylic nanocomposite. The M_n and M_w values of the acrylic/clay nanocomposite containing 5% nanoclay were 38,538 and 88,639, respectively, and the M_n and M_w values of the acrylic/clay nanocomposite containing 10% nanoclay were 42,736 and 99,846, respectively.

Preparation of nanocomposite coating

Thirty percent thermoplastic acrylic resin, 12% pentaerythritol, 24% ammonium polyphosphate, 7% melamine, 8% titanium dioxide, 2% glass fiber, 5% butyl acetate, and 12% dimethyl benzene were ground for 5 h in a ball mill to obtain the number 1 coating. The acrylic nanocomposites containing 5 and 10% nanoclay substituted the acrylic resin of the number 1 coating to obtain the number 2 nanocomposite coating containing 1.5% nanoclay and number 3 nanocomposite coating containing 3% nanoclay, respectively. The coatings were applied by spreading 5-mm thickness square steel plates (100 × 100 mm). The coated plates with a film thickness of 1 mm were dried at 25°C for 3 weeks in a ventilated environment.

Characterization

Differential thermal analysis (DTA) and thermogravimetry (TG) were carried out using a Setaram

Mtb10-8 and a Beijing optics CT-2 instrument, respectively, under air with a scan rate of 10°C/min. X-ray diffraction (XRD) analysis was conducted with a Bruker AXS diffractometer using Cu Ka radiation $(\lambda = 1.50595 \text{ A}, 2\theta 1.5-10)$. Acrylic/clay nanocomposite and acetone were mixed to obtain a nanocomposite solution. Transmission electron microscopy (TEM) samples were prepared by spreading a small amount of nanocomposite solution directly on a TEM grid. TEM images were obtained with a JEM-2000EX at an acceleration voltage of 200 kV. Char samples for scanning electron microscopy (SEM) observation were prepared by heating flame-retardant coatings under air at 600°C for 30 min. The morphology of the char samples was recorded with an SEM XL30 ESEM microscope operated at 20 kV. GPC analysis was performed at a flow rate of 1.00 mL/min and 35°C in THF using a Waters 410 component system. The limiting oxygen index (LOI) was measured according to the standard oxygen index test (ASTM D 2837/77). The apparatus used in the test was a DRK304 oxygen index meter. The dimensions of the specimens used for the test were $140 \times 38 \times 3$ mm.

Fire protection test

The flame temperatures likely to be reached in a practical fire were determined by the standard ISO 834 fire curve (Fig. 1). Three K NiCr thermocouples were used for temperature measurements, and temperature readings were taken every 2 min. Flame-retardant coatings with a thickness of 1 mm were applied over the bare steel surface. The coated plates were exposed to the flame of a gas blow lamp whose temperature was increased in accordance with the standard ISO 834 curve. The temperatures measured in the center of back side of the covered steel plates were plotted versus the time. When the average temperatures of three thermocouples reached 300°C, the time was defined as fire-resistant time. The fire-resistant time and thickness of the protective layer after the test were used to investigate the thermal insulation and fire protection of the intumescent coatings. The test was conducted to study the heat insulation properties of intumescent nanocomposite coatings in the presence of a fire. Changes in the heat insulation properties measured upon exposure to heat and fire give an indication of the resistance that the coatings have against exposure to static heat sources.

Accelerated aging test

This test studied the comparative fire performance of coatings before and after accelerated aging. The test was performed in a SH60CA weatherometer



Figure 1 A standard ISO 834 fire curve.

according to the ASTM D 529 standard. The test procedures were based on cycles of water spraying and drying (water spraying = 18 min/2 h) with exposure to UV radiation (exposure light intensity = 550 W/m K). The test temperature and relative humidity were 50 \pm 5°C and 70 \pm 5%, respectively. Flame-retardant coatings were subjective to accelerated aging tests for 500 h.

Effective thermal conductivity

The thermal insulation analysis of the intumescent coatings is not sufficient when only characterized by the temperature on the back side of steel plates. The effective thermal conductivity (λ/d) can provide much more thermal information about the coatings. Therefore, the λ/d is used to study the heat insulation properties of coatings further.

Measurements of the thermal conductivity (λ) were performed on a SWK-20 instrument. The λ of the intumescent materials was calculated using the relationship $\lambda = \alpha \times C_p \times \rho$, where α is the thermal diffusivity, C_p is the specific heat, and ρ is the density of the samples. The thermal diffusivity is determined from the measured temperature rise versus time data.

The coating samples were placed into a furnace for 10 min at 200, 300, 350, 400, 500, 600, 700, 800, 900, and 1000°C. The λ and thickness (*d*) of the intumescent materials at the different temperatures were measured, and the λ/d value was calculated. The λ/d values of the treated samples at different temperatures were plotted versus the time.

RESULTS AND DISCUSSION

Thermal analysis

Thermal degradation of the number 1 coating, number 2 nanocomposite coating, and number 3 nanocomposite coating is analyzed by the DTA curves in Figure 2. Initially, ammonium polyphosphate (acid source) breaks down to yield polyphosphoric acid at a temperature higher than 260°C. Then, polyphosphoric acid takes part in the dehydration of pentaerythritol and acrylic nanocomposite (carbonific agent) to yield the carbonaceous compound, and at the same time melamine (blowing agent) decomposes to yield gaseous products such as NH₃ and CO₂. Those gaseous products cause the fused carbonaceous compound to swell; hence, the process provides an intumescent insulating material for a metallic substrate.⁵ Two endothermic peaks at 191.6 and 264.3°C respond to the transformation of the crystal structure of pentaerythritol and melting of pentaerythritol, respectively.⁶ An endothermic peak at 330-420°C is attributed to char formation initiated by the reaction of ammonium polyphosphate, pentaerythritol, melamine, and acrylic nanocomposite.

MMT is an aluminosilicate mineral with a sheettype structure. Its formula is $M_y^+(Al_{2-y}Mg_y)(Si_4)$ $O_{10}(OH)_2 \cdot n(H_2O)$. The decomposition of MMT as a precursor of carbonization catalyst leaves a strong acid catalytic site that can further favor the esterification between ammonium polyphosphate and pentaerythritol, and it increases the char yield in the charring process. MMT can decompose to form a ceramic-like inorganic structure at a higher temperature range, and the structure creates a protective



Figure 2 DTA curves of (a) number 2 nanocomposite coating, (b) number 1 coating, and (c) number 3 nanocomposite coating.

physical barrier on the surface of the insulating materials. The inorganic protective barrier can enhance the resistance of fire protective materials to flame erosion and lead to good fire performance.⁷ Compared with the endothermic peak of the number 1 coating at 330–430°C, the endothermic peak of the number 2 nanocomposite coating is stronger than that of the number 1 coating, and the endothermic

peak of the number 3 nanocomposite coating is weaker than that of the number 1 coating. This demonstrates that the nanocomposite containing 5% nanoclay is favorable to the endothermic reaction of a flame-retardant coating. A splitting of the exothermic peak of the number 2 nanocomposite coating at 420–540°C denotes the reduction in the exothermic process and relative enhancement in fire retardancy.



Figure 3 XRD spectra of the residue inorganic materials obtained from the number 2 nanocoating at 800°C.



Figure 4 TG curves of (a) number 1 coating and (b) number 2 nanocomposite coating.

These positive effects can limit the flame and heat diffusion to the substrate and act as an efficient protective barrier for the metallic substrate.

Ceramic-like inorganic materials play an important protective role at the later stages of a fire. The XRD spectra of the inorganic materials are represented in Figure 3. The three strongest lines at 2.4521, 2.1028, and 1.4240 and at 3.8420, 1.4910, and 3.3325 in Figure 3 are assigned to MgAl₂O₄ and TiP₂O₇, respectively. Therefore, the decomposed products derived from nanoclay, titanium dioxide and ammonium polyphosphate, can lead to the formation of a ceramic-like material on the surface of the char structure that may enhance the char structure and improve the fire performance.⁸

The TG analysis was used to explore the applicability of nanoclay as a modification additive to improve the char yield and antioxidation simultaneously. TG curves of the number 1 coating and number 2 nanocomposite coating are shown in Figure 4. The weight loss at 330–420°C corresponds to thermal degradation and gaseous product release of ammonium polyphosphate, pentaerythritol, melamine, and acrylic nanocomposite. The reactions at this temperature range are a key to char formation. The weight losses of the number 1 coating and number 2 nanocomposite coating at 330–420°C are 17.53 and 17.02%, respectively, which indicates that 1.5% nanoclay in the number 2 nanocomposite coating does not produce a negative effect on the release of gaseous products and intumescence of the flame-retardant system. The residue weight of the number 2 nanocomposite coating at 1000°C is 8.78% higher than that of the number 1 coating at 1000°C. Because the ceramic-like inorganic materials decomposed from nanoclay can provide good protection for the char structure at high temperature, the number 2 nano-composite coating had the high residue weight and good fire retardancy.⁹

Fire performance

The unmodified nanoscale MMT particles agglomerate easily because of the surface attraction of one nanoparticle to another. As a result, the specific flame retardancy of nanoscale MMT is difficult to fulfill. TEM analysis was used to examine the distribution of MMT nanoparticles in the acrylic nanocomposite solution. In the nanocomposite, the functional groups (carbonyl) anchor in the sheets of MMT



Figure 5 A TEM image of the modified nanoclay particles distributed in the acrylic nanocomposite solution.

TABLE I LOI Values of Acrylic Resin and Nanocomposites

Components	LOI (%)
Acrylic resin	17.8
5% Nanoclay + acrylic resin	18.1
10% Nanoclay + acrylic resin	18.5
Acrylic resin + 30% IFR	31.0
5% Nanoclay $+$ acrylic resin $+$ 30% IFR	35.0
10% Nanoclay + acrylic resin + 30% IFR	37.0

and the nonreactive blocks attempt to gain entropy by pushing the sheets apart; hence, this reduces the free energy in the system and promotes miscibility between nanoscale MMT and acrylic resin. When the attractive force is minimized using surface treatment, each MMT particle can be dispersed to a nanoscale size. The TEM image of the modified nanoclay particles distributed in the acrylic nanocomposite solution is provided in Figure 5. The TEM observation shows an excellent distribution of the nanoclay particles with dimensions of 20–80 nm. Acrylic polymers enclose nanoclay particles to form spherical nanoparticles, as shown in Figure 5. Consequently, the well-distributed nanoclay can fully provide the specific function of flame retardancy.^{10,11}

The LOI values of various acrylic resins and nanocomposites are given in Table I. They were used to analyze the effect of nanoclay and the intumescent flame-retardant (IFR) system on the fire retardancy of acrylic resin. The IFR system consists of 65% ammonium polyphosphate and 35% pentaerythritol. A comparison of the samples suggests no significant modification for the nanoclay when it is used alone, but its combination with the IFR system shows some improvement in fire retardancy. The LOI value of the acrylic nanocomposite containing 5% nanoclay and 30% IFR increases from 17.8% acrylic resin to 35.0%. The significant increase in the LOI value indicates that the nanoclay and IFR system (ammonium polyphosphate and pentaerythritol) has some synergistic effect that is advantageous to fire retardancy. The LOI value of the acrylic nanocomposite containing 10% nanoclay and 30% IFR is only 2% higher than that of the acrylic nanocomposite containing 5% nanoclay and 30% IFR. This indicates that the high content of nanoclay cannot produce further significant modification for the LOI value of the acrylic nanocomposite.

The fire protection test is used to analyze the heat insulation properties of fire-resistant coatings in a fire. The thermal properties derived from the test are studied by fire protection curves. Figure 6 and Table II show a comparison of three fire-resistant coatings. At the beginning of each measurement the temperature increase of the three coatings is similar. With the increase of the temperature, three fire protection curves show some differences. The temperature difference among steel plates covered by intumescent coatings depends on the performance of the protective char layer. The intumescent protective char formed from the reaction of ammonium polyphosphate, pentaerythritol, and acrylic nanocomposite at 320-420°C provides an important fire protection for the metallic substrate. The thicknesses of the protective layers of the number 1 coating, number 2



Figure 6 Fire protection curves of (a) number 1 coating, (b) number 2 nanocomposite coating, and (c) number 3 nano-composite coating.

TABLE II Data of Fire Protection Test of Coatings

Sample	Protective layer thickness (mm)	Fire-resistant time (min)
No. 1 coating	29.6	77
No. 2 nanocomposite coating	32.5	87
No. 3 nanocomposite coating	19.3	58

nanocomposite coating, and number 3 nanocomposite coating are 29.6, 32.5, and 19.3 mm, respectively; the relative fire-resistant times of the three coatings are 77, 87, and 58 min, respectively. The fire-resistant time and expanding effect of the number 2 nanocomposite coating containing 1.5% nanoclay are better than those of the number 1 coating without nanoclay. This is because the catalytic effect of the nanoclay and formation of the ceramic-like protective materials initiated by the nanoclay are favorable to the fire protection properties of the number 2 nanocomposite coating.

The inorganic materials deriving from nanoclay can limit the oxygen diffusion to the metallic substrate, which produces a positive effect on the fire protection properties. At the same time, the high content of nanoscale inorganic materials decomposed from nanoclay can hinder the fused carbonaceous structure from swelling through a "lamp wick" effect, and this leads to a negative effect on the fire protection properties. When the mass fraction of nanoclay increases, the negative effect may exceed the positive effect on the fire protection properties. Three percent nanoclay in the number 3 nanocomposite coating produced a much more negative effect, and the side effect caused a decline in the fire resistance of the number 3 nanocomposite coating. Therefore, the fire protection properties of the number 3 nanocomposite coating with 3% nanoclay were even worse than those of the number 1 coating without nanoclay.^{12,13}

Effective thermal conductivity

The heat insulating effect of the reactive intumescent systems depends on the variation of the *d* and λ of the intumescent protective layer. As a result, the λ/d is used as a reference of the heat insulation of intumescent coatings. The λ/d of the intumescent char obtained from coatings at different temperatures is represented in Figure 7. The λ/d value of the number 1 coating continually decreases from the beginning temperature to 400°C, and the λ/d reaches its lowest value of 3.9 at 400°C. This is because a quick swelling of the multicellular char takes place in this temperature range. A cooperation effect of the different reaction processes at 200-400°C, such as char formation, endothermic reactions, and release of nonflammable gases, leads to an effective intumescence and decrease in the λ/d value. At temperatures higher than 400°C, the thermal degradation of the intumescent char layer leads to an increase in the λ/d value and a relative decline in heat insulation properties.

A ceramic-like protective material deriving from the thermal degradation of a specific content (1.5%)



Figure 7 The effective thermal conductivity of intumescent char obtained from (a) number 1 coating, (b) number 2 nanocomposite coating, and (c) number 3 nanocomposite coating at different temperatures.



Figure 8 Fire protection curves of (a) number 1 coating after 500-h accelerated aging and (b) number 2 nanocomposite coating after 500-h accelerated aging.

of nanoclay enhances anti-oxidation and fire retardancy of nanocomposite coatings at high temperature. The enhanced effect of 1.5% nanoclay results in good heat insulation and a low λ/d value of intumescent char at the middle and later stages of a fire. As depicted above, a high content (3%) of nanoclay in the number 3 nanocomposite coating is harmful to the intumescence and fire protection properties, so its λ/d value is the highest. Note from Figure 7 that the λ/d value of the number 1 coating is higher than that of the number 2 nanocomposite coating and lower than that of the number 3 nanocomposite coating. Therefore, 1.5% nanoclay improves the fireretardant and heat insulation properties of intumescent nanocomposite coatings. The λ/d analysis corresponds well with the results of the fire protection test and thermal analysis.¹⁴

Effect of aging on fire protection properties

Accelerating factors in the accelerated aging test are moisture/water, increased temperature, and UV radiation. Hydrophilic flame-retardant additives such as ammonium polyphosphate and melamine are very vulnerable to moisture. Ammonium polyphosphate and melamine can migrate to the surface of coatings when exposed to the accelerated aging condition, which destroys their chemical action and interaction in thermal degradation. Fire protection curves and data are provided in Figure 8 and Table III, respectively. The respective fire-resistant and expanding times of the number 1 coating decrease from 77 min and 29.6 mm before accelerated aging to 54 min and 18.9 mm after 500-h accelerated aging. This indicates that the fire performance of the number 1 coating declined after the accelerated aging test. Because some flame-retardant coatings used for steel structural members face outdoor exposure continuously throughout their service life, it is necessary to improve the antiaging properties of flame-retardant coatings to resist the damage of aging to fire-resistant properties.¹⁵

For the number 2 nanocomposite coating before and after 500-h accelerated aging, the differences in the fire-resistant time and thickness of the protective layer are only 7 min and 4.7 mm, respectively. The well-distributed nanoclay particles can form a nanoscale interpenetrating network in the number 2 nanocomposite coating. The nanoparticle network can produce a "labyrinth" effect that may effectively resist moisture permeation and UV damage, so the number 2 nanocomposite coating can retain good fire performance during the aging process.

The SEM micrographs of chars obtained from the number 1 coating and the number 2 nanocomposite

TABLE III Data of Fire Protection Test of Coatings after 500-h Accelerated Aging

	0 0	
Sample	Protective layer thickness	Fire-resistant time
No. 1 coating No. 2 nanocomposite coating	18.9 27.8	54 80



Figure 9 SEM micrographs of intumescent chars obtained from (a) number 1 coating after 500-h accelerated aging and (b) number 2 nanocomposite coating after 500-h accelerated aging.

coating after 500-h accelerated aging are given in Figure 9. When exposed to fire temperatures, the intumescent coating undergoes expansion to form a protective foamed char layer. The intumescent char structure formed from flame-retardant coatings protects the metallic substrate in a fire. The flameretardant efficiency of intumescent coatings depends on the formation of a char structure, so the expanding effect and the formation of a foamed char structure are very important to the general fire-resistant properties of intumescent coatings.

Although the number 1 coating can still expand to form a char structure after 500-h accelerated aging, the aging process has damaged its expanding effect. There is no good "honeycomb" intumescent char structure in Figure 9(a). This is because accelerated aging damages the chemical action of the hydrophilic flame retardants in the number 1 coating. The damaged ammonium polyphosphate and melamine cannot send out amine and phosphoric acid efficiently in thermal degradation, which leads to a great loss in fire-resistant properties. A good honeycomb intumescent char structure is presented in Figure 9(b). The nanoscale network structure formed by nanoclay particles improves the antiaging of the number 2 nanocomposite coating, and thus the chemical action of the flame retardants is retained. In addition, the ceramic-like materials formed from the nanoclay at high temperature enhance the char structure and reduce its cracking. Therefore, the number 2 nanocomposite coating in thermal degradation can still demonstrate a good intumescent char structure and fire retardancy after accelerated aging.¹⁶

CONCLUSIONS

The chemical interaction of ammonium polyphosphate, pentaerythritol, melamine, and acrylic nanocomposite at 330–420°C formed an intumescent char that provided fire protection for the metallic substrate. Nanoclay enhanced the fire performance of intumescent char because nanoscale inorganic materials decomposed from nanoclay created a ceramiclike protective barrier on the surface of the insulating materials. The fire performance of the intumescent coating was improved by 1.5% nanoclay. Three percent nanoclay in the nanocomposite coating produced a much more negative effect than positive effect, and this led to a decline in fire performance. Accelerated aging damaged the chemical action of the flame-retardant additives in thermal degradation, which resulted in poor fire performance of the intumescent coating. One and one-half percent welldistributed nanoclay particles formed a good nanoparticle interpenetrating network in the structure of the nanocomposite coating, and the nanoscale network greatly improved the antiaging and fireretardant properties of the nanocomposite coating.

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