

## THERMAL DECOMPOSITION AND FLAMMABILITY OF ACRYLONITRILE-BUTADIENE-STYRENE/MULTI-WALLED CARBON NANOTUBES COMPOSITES

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**Abstract** Thermal and flammability properties of acrylonitrile-butadiene-styrene copolymer (ABS) with the addition of multi-walled carbon nanotubes (MWNTs) were studied. ABS/MWNTs composites were prepared via melt blending with the MWNTs content varied from 0.2% to 4.0% by mass. Thermogravimetry results showed that the addition of MWNTs accelerated the degradation of ABS during the whole process under air atmosphere, and both onset and maximum degradation temperature were lower than those of pure ABS. The destabilization effect of MWNTs on the thermal stability of the composites became unobvious under nitrogen, and the addition of MWNTs could improve the maximum degradation temperature. The heat release rate and time of ignition ( $t_{\text{ign}}$ ) for the composites reduced greatly with the addition of MWNTs especially when the concentration of nanotubes was higher than 1.0%. The accumulation of carbon nanotubes with a network structure was observed and the char layer became thicker with increasing nanotubes concentration. Results from Raman spectra showed a higher degree of graphitization for the residues of ABS/MWNTs composites.

**Keywords:** Carbon nanotubes; Thermoplastic resin; Flammability; Thermal properties.

### INTRODUCTION

Since their discovery in 1990s carbon nanotubes (CNTs) have attracted intense attention throughout the academic and industrial world. Due to their exceptional mechanical, electrical, thermal properties and large aspect ratio, CNTs have potential applications in many areas such as biosensors, conducting agents, field-effect transistor, and nanocomposites.

Improvements in thermal stability and flammability properties of polymers have been obtained with nanoscale additives, and these filled systems provide an alternative to conventional flame retardants. The polymer/layered silicate nanocomposite is one of the most common approaches and has been reported in recent years<sup>[1–6]</sup>. Carbon nanotubes, due to their unique structure and properties, offer another candidate as a flame retardant additive<sup>[7–11]</sup>. At present, flammability properties of polymer/carbon nanotube composites with various resins such as polypropylene (PP)<sup>[12–14]</sup>, polymethyl methacrylate (PMMA)<sup>[15–17]</sup>, polyamide 6 (PA6)<sup>[18]</sup>, ethylene vinyl acetate copolymer (EVA)<sup>[19]</sup> have been investigated. It is found that the heat release rate of the polymers can be significantly reduced by carbon nanotubes, which are found to be at least an effective flame retardant as layered silicates<sup>[12]</sup>.

Acrylonitrile-butadiene-styrene copolymer (ABS) is an important engineering plastics widely used in industry due to its superior mechanical properties, chemical resistance, ease of processing and recyclability. One of the main drawbacks of ABS is its inherent flammability during combustion. There is a few reports concerning with the thermal degradation of ABS/single-walled carbon nanotubes composites<sup>[20]</sup>. Little work has been done on the fire performance of ABS/MWNTs composites. In this paper, multi-walled carbon nanotubes (MWNTs) were used to fabricate ABS/MWNTs composites by melt blending. The dispersion of MWNTs in ABS was observed. Effects of MWNTs on the thermal stability under air and nitrogen and flammability properties of the

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composites were investigated. The structures of the residue after combustion were investigated, and the mechanism of flame retardancy was discussed.

## EXPERIMENTAL

### Materials

The ABS resin (HI-121H) was obtained commercially from LG Industries, Korea. MWNTs were provided by the Institute of Material Physics and Microstructure at Zhejiang University (China), which were prepared by catalytic decomposition of acetylene at 700°C for *ca.* 60 min<sup>[21]</sup>. The diameters of MWNTs are not uniform, with a range from 10 to 60 nm, and the lengths are of the order of microns. The MWNTs were purified by soaking in nitric acid for 48 h at room temperature for removing the catalyst residues and impurities.

### Preparation of ABS/MWNTs Nanocomposites

ABS/MWNTs nanocomposites containing 0.2, 0.5, 1.0, 2.0 and 4.0 wt% of MWNTs were prepared *via* melt compounding at 190°C in ThermoHaake Rheomix with a rotor speed of 60 r/min, and the mixing time was 10 min for each sample. The mixed samples were transferred to a mold and preheated at 190°C for 3 min, then pressed at 14 MPa, and successively cooled to room temperature while maintaining the pressure to obtain the composite sheets for further measurements.

### Characterization

Thermal decomposition studies of the composites were performed over a temperature range of 50–600°C using a TA instrument SDT Q600 under both nitrogen and air environments at a heating rates of 10 K/min.

Flammability of the nanocomposites was characterized by using cone calorimeter tests performed with an FTT, UK device according to ISO 5660 at an incident flux of 35 kW/m<sup>2</sup> using a cone shape heater. All samples were 10 cm × 10 cm plates and were placed in an aluminum foil and then in a box having the same dimensions in the horizontal position. The thickness of the samples was 1.5 mm. Heat release rate (HRR), mass loss rate (MLR), specific extinction area (SEA), ignition time ( $t_{\text{ign}}$ ), and specific heat of combustion data are reproducible to within 10% when measured at 35 kW/m<sup>2</sup>. The cone data reported here are the averages of replicated experiments.

Morphological studies of dispersion of MWNT in the matrix were performed using a field emission scanning electron microscope (FEI Sirion FESEM) and a transmission electron microscope (TEM, JEM-1200EX). Morphology of the char residues after cone calorimeter tests was investigated by FESEM.

## RESULTS AND DISCUSSION

### Composites Morphology

Multi-wall carbon nanotubes (MWNTs) are commonly used due to their low cost over single wall carbon nanotubes. Compared to clay, carbon nanotubes are organophilic and can be dispersed directly into the polymer. The distribution of the MWNTs in the composites was examined by FESEM and TEM, as shown in Fig. 1. Figure 1(a) indicates that the nanotubes are relatively uniformly distributed in the ABS matrix. A higher magnification image by TEM (Fig. 1b) shows the region of slight aggregated nanotubes which have a curled, entangled structure.

### Thermal Stability

As for the thermal degradation of ABS, researchers obtained various results due to variety of ABS resins<sup>[22–25]</sup>. The differences come from the resin itself including molecular weight, component ratios of acrylonitrile, butadiene, and styrene, *etc.* All of these factors affect the decomposition of ABS. Suzuki *et al.*<sup>[22]</sup> applied thermogravimetry/Fourier transform infrared (TGA/FTIR) method to investigate the degradation of ABS, and they found that the evolution of butadiene commenced at 340°C and styrene at 350°C, while the evolution of acrylonitrile began at 400°C. Dong *et al.* investigated the degradation of ABS with different TGA testing conditions, the evolution of volatile substances was observed throughout the whole process<sup>[23]</sup>.

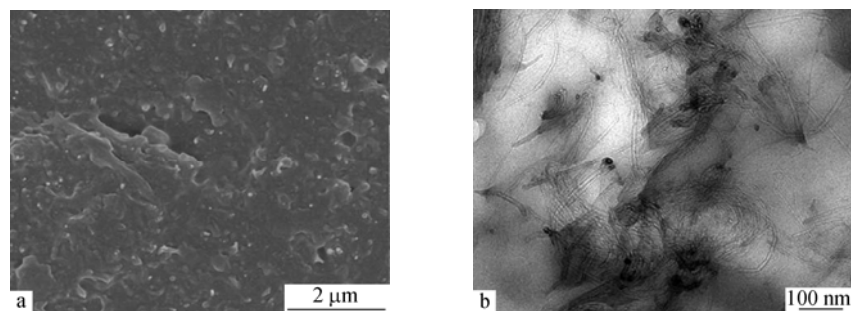


**Fig. 1** MWNTs dispersion in ABS matrix with (a) FESEM image and (b) TEM image

Figure 2 shows the TGA curve obtained in nitrogen atmosphere for the MWNTs. The results show that only 5% weight loss is found for MWNTs at 600°C, indicating the high thermal stability. The degradation of pure ABS resin and ABS/MWNTs composites was analyzed, and the TGA and DTG curves under air and nitrogen are presented in Fig. 3 and Fig. 4, respectively. Figure 3 shows that two steps of degradation, one around 300–500°C and the other upper than 500°C, exist for pure ABS under air, which is consistent with the results obtained by Hu *et al.*<sup>[26]</sup>. However, only one step decomposition around 350–500°C is found for ABS under nitrogen (Fig. 4).

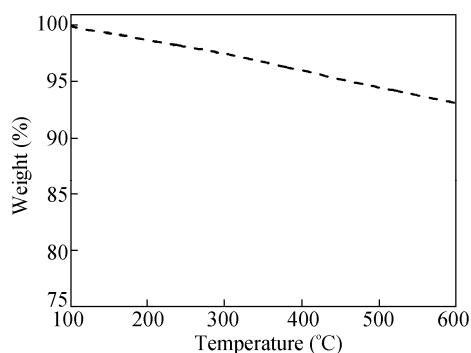


**Fig. 2** TGA curve of MWNTs under nitrogen

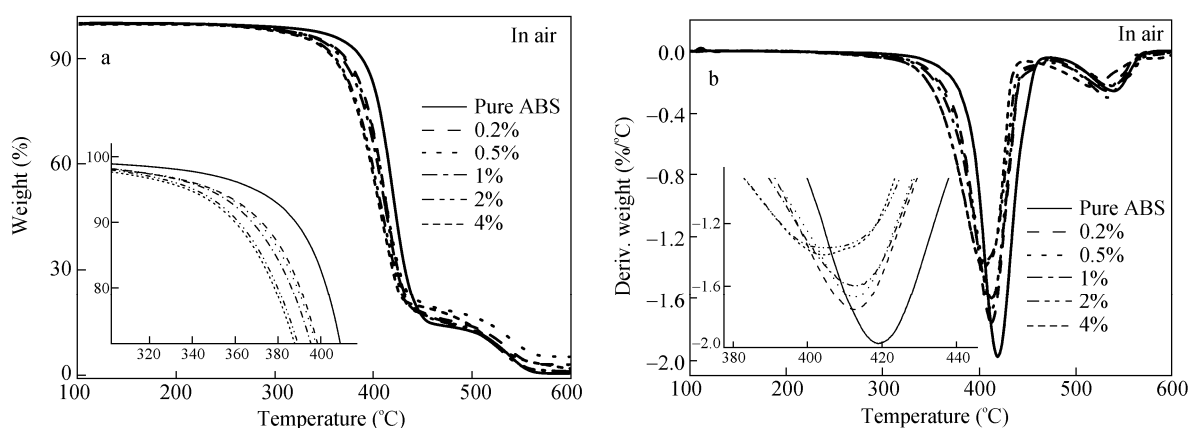


**Fig. 3** TGA (a) and DTG (b) curves of pure ABS and ABS/MWNTs nanocomposites with different MWNTs contents under air

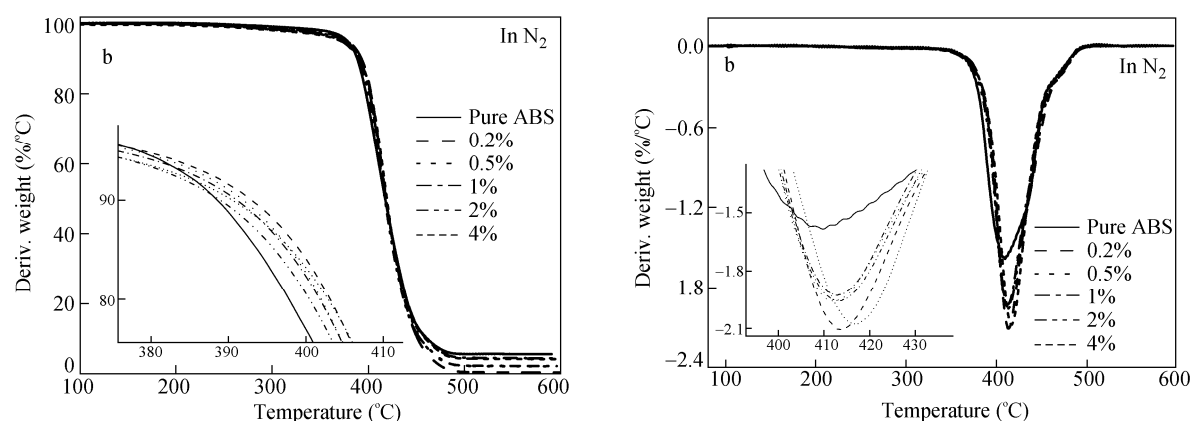


**Fig. 4** TGA (a) and DTG (b) curves of pure ABS and ABS/MWNTs nanocomposites with different MWNTs contents under nitrogen

The summary of the TGA and DTG results for the various composites in air and nitrogen are presented in Table 1. Under air conditions, all the composite samples degraded in two major steps, similar to the pure ABS resin. From careful analyses it is obvious the composites start to degrade at a lower temperature than that of ABS. The detailed TGA data, for example, the onset decomposition temperature ( $T_{\text{onset}}$ ) and the maximum decomposition temperature ( $T_{\text{max}}$ ) for ABS and its composites are summarized in Table 1. It is found that the addition of MWNTs accelerates the degradation of ABS/MWNTs composites during the whole process. The results show that with just 0.2 wt% MWNTs, the  $T_{\text{onset}}$  is 19.2 K lower than that of neat ABS. With the concentration of the MWNTs increasing from 0.2% to 1.0%, the degradation temperatures of ABS/MWNTs composites continue to decrease slowly. But when the concentration of the MWNTs is higher than 1.0%,  $T_{\text{onset}}$  is reduced significantly again and keep constant with the MWNTs content upper than 2.0%. As for higher temperatures, the addition of MWNTs also destabilized the second step degradation. When the MWNTs concentration is lower than 1%,  $T_{\text{max2}}$  is slightly lower than that of pure ABS resin. However, at higher MWNTs loadings,  $T_{\text{max2}}$  is significantly reduced where the maximum lowering is 21.3 K with 4% MWNTs. The results above are not consistent with the studies of Yang *et al.*<sup>[20]</sup> in which the addition of SWNTs destabilized the first degradation step but reinforce it at the second step degradation. The final residue for all the samples is small, generally below 3% as shown in Table 1, except for the sample with 0.5% MWNTs that has a residue of 5.2%.

**Table 1.** Results of TGA analysis of ABS and ABS/MWNTs nanocomposites with different MWNTs concentration under different atmosphere environment

| MWNTs content<br>(wt%) | ABS/MWNTs (in air)      |                        |                        |                      | ABS/MWNTs (in N <sub>2</sub> ) |                       |                      |
|------------------------|-------------------------|------------------------|------------------------|----------------------|--------------------------------|-----------------------|----------------------|
|                        | $T_{\text{onset}}$ (°C) | $T_{\text{max1}}$ (°C) | $T_{\text{max2}}$ (°C) | Residue at 600°C (%) | $T_{\text{onset}}$ (°C)        | $T_{\text{max}}$ (°C) | Residue at 600°C (%) |
| 0                      | 369.2                   | 419.3                  | 539.1                  | 0.4                  | 410.3                          | 409.8                 | 5.6                  |
| 0.2                    | 350.1                   | 412.7                  | 536.3                  | 2.8                  | 411.2                          | 413.8                 | 0.4                  |
| 0.5                    | 349.6                   | 412.5                  | 538.7                  | 5.2                  | 409.2                          | 412.5                 | 2.2                  |
| 1.0                    | 347.3                   | 413.2                  | 531.5                  | 1.1                  | 405.7                          | 413.1                 | 4.5                  |
| 2.0                    | 338.1                   | 403.6                  | 531.6                  | 1.2                  | 400.0                          | 413.4                 | 2.2                  |
| 4.0                    | 338.0                   | 404.3                  | 517.8                  | 1.9                  | 398.7                          | 417.0                 | 4.2                  |

From the results of Table 1, it can be seen that there is a mutation for  $T_{\text{onset}}$  and  $T_{\text{max}}$  at 1% loading of MWNTs. There is little difference for  $T_{\text{onset}}$  and  $T_{\text{max}}$  at lower MWNTs loadings but a significant reduction when the MWNTs loading is higher than 1%. A further investigation suggested that network structure for MWNTs formed in ABS resin when the MWNTs concentration reached 1%. The correlation between MWNTs network and flammability properties is being prepared for further publication.

Compared to the degradation under air, the degradation behavior of the composites is basically a one-step

process under nitrogen (Fig. 4). The results show that the addition of MWNTs does not deteriorate the thermal stability significantly for ABS/MWNTs composites which can be contributed to the depletion of oxygen. The TGA and DTG data under nitrogen are summarized in Table 1. Compared to the results in air,  $T_{\text{onset}}$  does not show any change when MWNTs concentration is lower than 1.0%. The destabilizing effect of MWNTs is more obvious when MWNTs concentration is higher than 1.0%: with 4% of MWNTs, the  $T_{\text{onset}}$  is 11.6 K lower than that of pure ABS. But the addition of MWNTs improves the maximum degradation temperature: with 0.2% of MWNTs, the  $T_{\text{max}}$  is 4 K higher than that of pure ABS and the maximum increase is 7.2 K. The final residues of all the composites are lower than that of pure ABS. Considering the residues formed by MWNTs itself, the ABS matrix resin is almost entirely decomposed.

The above results indicate that the MWNTs may reduce the thermal stability of ABS under air and nitrogen atmosphere. The destabilization effect of MWNTs on the thermal stability of composites becomes unobvious under nitrogen. It indicates the catalytic effect of MWNTs is associated with the participation of oxygen. It is well known that carbon nanotubes have very high thermal stability as reported by Pang *et al.*<sup>[27]</sup> and Li *et al.*<sup>[28]</sup>. Since the degradation of ABS is known as a radical process<sup>[23]</sup>, and the rubber domains are the most vulnerable sites for degradation<sup>[29]</sup>, MWNTs may have participated in the radical initiation process<sup>[20]</sup>. MWNTs take part in the initiation of the degradation of ABS and degrade themselves along with ABS. This may explain why the final residues are lower than that of pure ABS during TGA tests.

### Flammability Properties

Cone calorimetry is widely used to evaluate fire performance. Various parameters are obtained from cone measurements, including the time to ignition ( $t_{\text{ign}}$ ), heat release rate (HRR), peak release rate (PHRR), time to peak heat release rate ( $t_{\text{PHRR}}$ ), total heat release (THR), average mass loss rate (AMLR) and average specific extinction area (ASEA), a measure of smoke. HRR and PHRR are important terms to evaluate fire safety. HRR determines the speed of fire development during the combustion tests (Table 2 and Fig. 5). It has been noted that a microcomposite gives a minimal reduction in PHRR while nanocomposite formation brings about a larger reduction<sup>[30]</sup>.

**Table 2.** Cone calorimetry data for ABS and ABS/MWNTs nanocomposites at 35 kW/m<sup>2</sup>

| MWNT content (%) | $t_{\text{ign}}^a$ (s) | PHRR <sup>a</sup> (kW/m <sup>2</sup> ) | THR <sup>a</sup> (MJ/m <sup>2</sup> ) | ASEA <sup>a</sup> (m <sup>2</sup> /kg) | AMLR <sup>a</sup> (g/s) |
|------------------|------------------------|----------------------------------------|---------------------------------------|----------------------------------------|-------------------------|
| 0                | 28                     | 930.0                                  | 34.0                                  | 1475.6                                 | 0.095                   |
| 0.2              | 27                     | 523.3                                  | 27.3                                  | 1195.6                                 | 0.083                   |
| 0.5              | 28                     | 517.5                                  | 27.4                                  | 999.3                                  | 0.078                   |
| 1                | 34                     | 425.0                                  | 26.6                                  | 957.0                                  | 0.053                   |
| 2                | 35                     | 436.6                                  | 25.7                                  | 1018.6                                 | 0.050                   |
| 4                | 36                     | 438.7                                  | 24.3                                  | 1005.4                                 | 0.048                   |

<sup>a</sup>  $t_{\text{ign}}$ : time to ignition; PHRR: peak release rate; THR: total heat release; ASEA: average specific extinction area; AMLR: average mass loss rate

During the cone experiments, visual observations reveal different fire behavior for pure ABS and ABS/MWNTs composites. As expected, the pure ABS resin melt and behave like liquid accompanied by numerous bubbles bursting on the sample surface. At the end of the test, no residue is left, confirming poor carbonization effect of ABS. On the other hand, ABS/MWNTs composites display thin char layer at the beginning of the thermal exposure. For the composites with 0.2 wt% and 0.5 wt% concentrations of MWNTs black discrete islands form, and thin chars grow with a tendency for cracking in the surface during combustion, and many small bubbles burst at the surface. The char layers become thick and uniform when MWNTs' concentration is higher than 1 wt%.

The cone data for ABS and ABS/MWNTs composites are collected in Table 2 and the heat release rate curves for ABS and its nanocomposites are shown in Fig. 5. Two stages for the parameters of  $t_{\text{ign}}$ , PHRR and AMLR are found. When the MWNTs content is lower than 1%, PHRR displays a 43% reduction, and  $t_{\text{ign}}$ ,

AMLR exhibits little change comparatively to pure ABS resin. However, parameters of all samples change significantly when the MWNTs concentration is higher than 1 wt% compared to the base polymer: PHRR and AMLR display 55 % and 48 % reduction respectively;  $t_{\text{ign}}$  is 7–8 s longer than that of ABS resin. It is worth noting that  $t_{\text{ign}}$ , AMLR and PHRR display no obvious change with the variation of MWNTs concentration when the MWNTs content is higher than 1%. THR is significantly reduced for ABS/MWNTs nanocomposites even with 0.2% MWNTs loading and then gradually reduced with the increase of MWNTs loading.

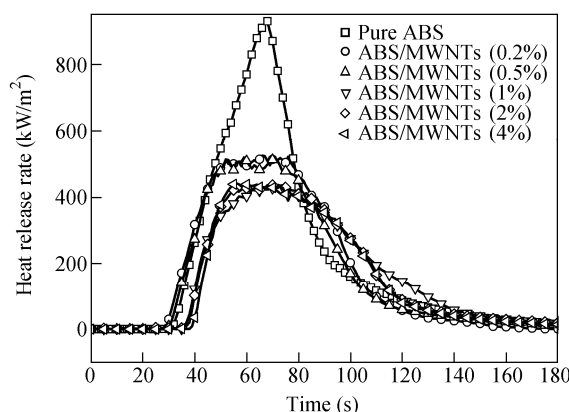


**Fig. 5** Heat release rate curves for ABS and ABS/MWNTs nanocomposites with different MWNTs content after cone calorimeter tests

Previous reports found that formation of the network-structured protective layer<sup>[17]</sup> and the degree of graphitization<sup>[31]</sup> during burning is crucial for the large reduction in heat release rate for nanocomposites based on carbon nanotubes. From the results above, it is found PHRR can be reduced significantly even with 0.2% nanotubes concentration which indicates nanotubes are uniformly distributed and network structured layer forms after combustion. PHRR reaches a minimum value at the mass fraction of 1.0% nanotubes and increases at higher MWNTs concentration, which suggests aggregation exists in the composites with higher concentration of MWNTs. The lowest heat release rate is observed with the ABS/MWNTs (1.0) sample due to the balance between MWNTs content and their distribution in the sample, therefore, the best protective effect can be obtained. A more detailed discussion of residues is given in the following section.

### Residues Investigation

Just as the conclusions mentioned above, the formation of a protective network layer and the degree of graphitization are critical for reducing the heat release rate for the nanocomposites based on carbon nanotubes. A better understanding of the char structure of polymer/nanotubes nanocomposites is a key issue towards understanding the mechanism of flame retardancy<sup>[31]</sup>. Raman spectroscopy was used to further characterize the graphitization of the MWNTs and residues of ABS/MWNTs composites (Fig. 6). The peak at  $1598\text{ cm}^{-1}$  (G-band) corresponds to an  $E_{2g}$  mode of hexagonal graphite and is related to the vibration of  $sp^2$ -hybridized carbon atoms in a graphite layer. The D-band at about  $1359\text{ cm}^{-1}$  is associated with the vibration of carbon atoms with dangling bonds in the plane terminations of disordered graphite or glassy carbon<sup>[32]</sup>. The value of G/D can represent the degree of graphitization. The results of Fig. 6 show a higher degree of graphitization of the residue ( $G/D = 1.13$ ) than that of MWNTs ( $G/D = 1$ ) which indicate the nanotubes tend to give better protection to char from oxidation.

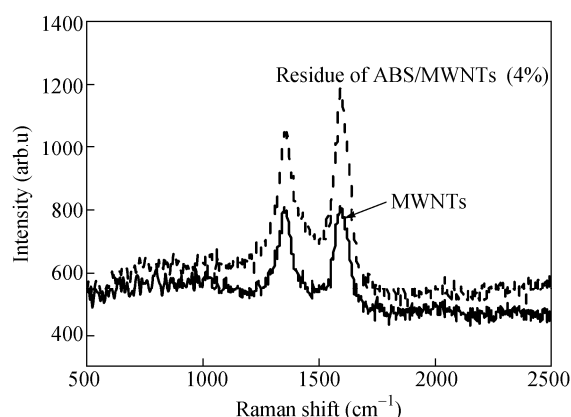


**Fig. 6** Raman spectra of MWNTs and the residue of ABS/MWNTs (4.0%) composites

Figure 7 shows the macroscopic morphology of the chars of all samples produced by cone calorimeter tests. The results show most of the pure resin is almost burnt out and no residues left after cone calorimeter tests as expected. A thin, fluffy char layer with few cracks remains and sectional network-structured char layers form for ABS/MWNTs composites with a low nanotubes concentration. The char layer becomes thicker with increasing nanotubes concentration. An FESEM picture of the residue of ABS/MWNTs (4 wt%) composites is presented in Fig. 8. A network layer is observed, and the accumulation of nanotubes with such structure tends to act as a thermal insulation layer during combustion. In contrast to the layered silicate nanocomposites reported<sup>[33]</sup>, the combustion residue was not a closed layer.

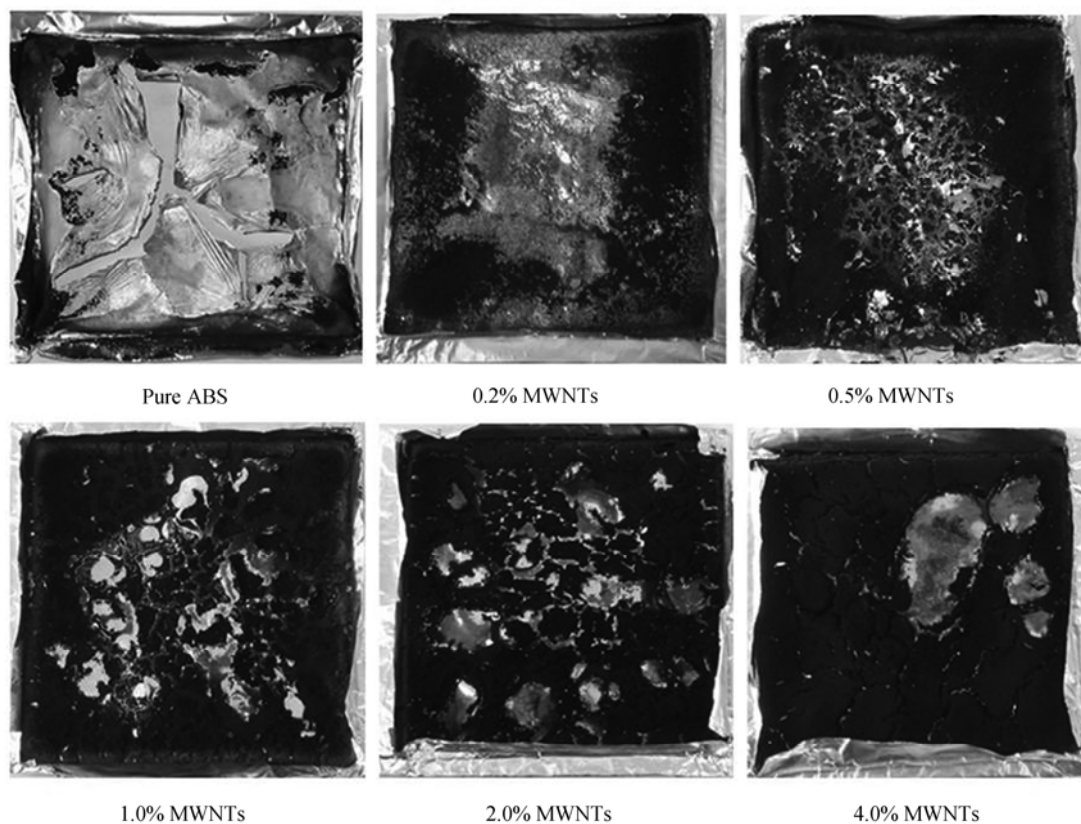


**Fig. 7** Morphologies of the chars for ABS and ABS/MWNTs nanocomposites with different MWNTs content after cone calorimeter tests

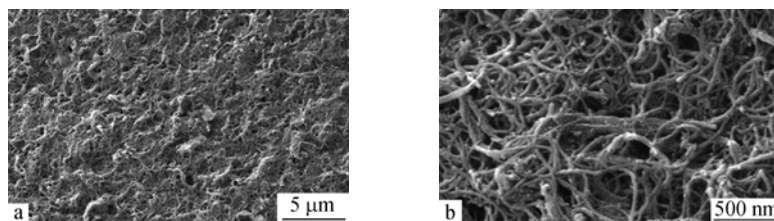


**Fig. 8** FESEM image for the residue of the ABS/MWNTs (4%) nanocomposites at low (a) and high (b) magnification

## CONCLUSIONS

ABS/MWNTs nanocomposites were prepared *via* melt blending with the MWNTs content varied from 0.2 wt% to 4.0 wt% by mass. Dispersion of MWNTs in these nanocomposites was characterized by FESEM and TEM. Thermal properties were measured with thermogravimetry analysis, and flammability properties were measured with a cone calorimeter. A relative good dispersion of MWNTs was found even without using any organic treatment. TGA and DTG results showed that the addition of MWNTs accelerates the degradation of ABS/MWNTs composites during the whole process, and both onset and maximum degradation temperature are lower than those of pure ABS under air atmosphere. The destabilization effect of MWNTs on the thermal stability of composites becomes unobvious, and the addition of MWNTs can improve the maximum degradation temperature under nitrogen. The heat release rate and time to ignition ( $t_{\text{ign}}$ ) for the composites reduce greatly with the addition of MWNTs especially when the concentration of nanotubes is higher than 1.0%. The accumulation of carbon nanotubes with a network structure was observed, and the char layer became thicker with increasing nanotubes concentration. Results from Raman spectra showed a higher degree graphitization for the residues of ABS/MWNTs composites.

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