

Dependence of Flame-Retardant Properties on Density of Expandable Graphite Filled Rigid Polyurethane Foam

Xiang-Cheng Bian,¹ Jian-Hua Tang,² Zhong-Ming Li,³ Zhong-Yuan Lu,¹ Ai Lu¹

¹College of Materials Science and Engineering, Southwest University of Science and Technology, Mianyang 621002, Sichuan, People's Republic of China

²College of Chemical Engineering, Sichuan University, Chengdu 610065, People's Republic of China

³College of Polymer Science and Engineering, State Key Laboratory of Polymer Materials Engineering, Sichuan University, Chengdu 610065, People's Republic of China

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ABSTRACT: Different density rigid polyurethane foams (RPUF) filled with various expandable graphite loadings were fabricated by cast molding. The flame retardant properties of these composites were assessed by limiting oxygen index and horizontal and vertical burning tests. The results showed that the flame retardant efficiency got better with increase in the foam density at the fixed EG weight percent or with increase in the EG weight percent at the fixed foam density. After burning, the low density (0.065 g/cm³) pure burned RPUF produced the highly collapsed and carbonized material, while the high density (0.510 g/cm³) pure RPUF had little change in size and had reduced destruction of the material. Moreover, the scanning electronic microscope (SEM) observation showed that

the higher density EG/RPUF composites had a more compact outer layer (burned layer) after burned, in which more wormlike materials composed of expanded graphite particles appeared. In addition, higher foam density led to less plastic deformation in the interface layer between the burned and the inside layers. These results indicated that a weight percent of a flame retardant additive that achieves satisfactory flame retardancy for a certain density foam cannot effectively be applied for another density foam. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 104: 3347–3355, 2007

Key words: polyurethane foam; density; expandable graphite; flame retardant

INTRODUCTION

Polyurethane foam (PUF) is widely used in industries and our daily lives due to its excellent properties, such as mechanical properties, insulated properties, etc. Density is a key feature of PUF materials, which greatly affects their properties. It has been demonstrated that strength and modulus and energy absorption of a material all increase linearly with the foam density.^{1–3} In contrast, the deformation at high temperature and the oil sorption behavior of PUF decrease rapidly with the increase of the foam density.^{2,4} Consequently, the density of PUF materials, to a great degree, determines their applications. The lower density PUFs are usually used as insulating materials, sofa cushion, mattress and reduced energy materials, etc.,^{5,6} while the higher density PUFs are used as structural materials, energy absorption materials, mechanical shock materials, and even used to encapsulate the stockpile of nuclear weapon, etc.⁷

Unfortunately, PUF is flammable and releases toxic gases in the process of burning,^{8,9} which limits their further applications. Nowadays, more and more attention has been paid to improve the flame retardant properties of PUF. The main approach reaching such a target is to incorporate flame retardants into the PUF. The most widely used flame retardants are these compounds, including halogen,^{10–12} phosphorus,^{10,13,14} nitrogen,^{15–18} etc. But the flame retardant ability of phosphate esters and nitrogen compounds are very inapparent.^{19–21} Moreover, the current demand in flame retardants has considerably changed, and environmental protection is a key factor in all stages of foam cycle. Usually, the halogenated additives release corrosive, obscuring, and toxic smoke,²² which pollutes environment, erode instruments, and even damage people's health. As a result, the flame retardant additives which have good flame retardant efficiency and hardly pollute environment are particularly needed. Following this orientation, a new-style flame retardant, i.e., intumescent systems, attracts more and more attention.

Expandable graphite (EG) is typical of the intumescent flame retardant. EG is composed of flake graphite in which sulfuric acid was early inserted.

Correspondence to: Z.-M. Li (zm_li@263.net.cn).

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TABLE I
Specifications of the Materials Used in This Work

Materials	Specifications
Polyether polyol	GR-4110G, prepared by polypropylene oxide and sucrose/glycerin, obtained from GaoQiao Petro. Co. (Shanghai, China). Main properties: density (25°C), 1.1 g/cm ³ ; typical hydroxyl number, 430 mg potassium hydroxide (KOH) equiv/g; viscosity (25°C), 3,283 cps; functionality, 4.1; average molecular weight, 550 g/mol
Isocyanate(PAPI)	N200, obtained from ChangFeng Chemical Co. (Chongqing, China). Main properties: Isocyanate equivalent weight, 126.5 g; -NCO wt %, 31.8%; viscosity (25°C), 215 cps; functionality, 2.2
Triethanolamine	Crosslinked catalyst, available from Shanghai Chemical Reagent Co. (Shanghai, China); density, 1.122 g/cm ³
Dibutyl tin dilaurate	Catalyst for the prolongation of molecular chains. Main properties: density, 1.052 g/mol; Sn content, 18 wt %; provided by Sichuan Chemical Reagent Co. (Chengdu, China)
Silicone glycol copolymer	Surfactant
Distilled water	Blowing agent
Expandable graphite (EG)	Flame retardant, obtained from Haida Graphite Co. (Qingdao, China). Main properties: ash, 1.0%; moisture, 1.0%; volatile, 15%; PH value, 3.0; particle size, 80%; expansion rate, 200 mL/g

When exposed to a heat source, EG expands and generates insulative layer on the surface of the polymeric matrix providing fire retardant performance. Modesti et al.^{19,23} studied the flame retardant properties of EG/PUF foam with a density of 0.035 g/cm³, and concluded that increasing EG content gives a significant improvement in fire reaction of materials. In our previous work, we prepared high-density (0.46–0.50 g/cm³) rigid RPUFs with excellent flame retardant properties,^{22–24} and found that when the EG loading was 20 wt %, the limiting oxygen index (LOI) increased to a high level, 39.5 vol %. As to the origin of the flame retardancy of EG, it has been established that a “worm-like” structure layer can be formed on the surface of the materials due to the expansion of EG during burning, and such a layer of graphite can prevent heat and oxygen entering the bulk.^{20,21,25} The flame retardant efficiency of EG was also influenced by the sizes of EG particles. Lower size EG particles led to lower volume expansion ratio and less flame retardant efficiency.^{26–28}

Usually, different applications require different foam densities, since density dominates the properties, naturally including fire performance, of a foam material. There arises a problem that a well-defined fire retardant formula for a specific density foam cannot, quite possibly, be effective in another density foam. Consequently, it is of significance to thoroughly investigate the correlation between flame properties and density for a foam material. But there is still no literature available about this investigation. In this study, we prepared different density and different EG loading EG/RPUF composites by cast molding, and characterized the flame retardant properties of these foam composites through horizontal-vertical burning test and LOI, etc., and discussed the origin of these different density RPUF composites.

EXPERIMENTAL

Materials

The main materials used in this study include polyester polyol, isocyanate (PAPI), triethanolamine, dibutyl tin dilaurate, distilled water, and expandable graphite (EG), whose details about suppliers, specifications, etc. are listed in Table I. The equilibrium ratio of NCO to OH groups and the weight ratio of polyol to PAPI are listed in Table II.

Foam preparation

Pure RPUF was prepared by cast molding. All components except EG and PAPI were mixed and stirred together with an electric stirrer until a uniform mixture was obtained. PAPI was added into the mixture in a certain weight ratio and then stirred fast for 30 s. PAPI reacts with distilled water to generate polyurea and carbon dioxide. Carbon dioxide inflates the reactants. The mixture was completely cast in a mold with a lid closed quickly. The laden mold was put into an oven and hot treated for 4 h at 100°C to increase the cross-link density and make reaction more sufficient. The foam was taken out from the mold and the hard coat was removed. EG filled RPUF was prepared using the same way, but EG was added to the PAPI before blowing polymerization. In this work, the foams with

TABLE II
Equilibrium Ratio of NCO to OH groups and the Weight Ratio of Polyol to PAPI

Density (g/cm ³)	Equilibrium ratio of NCO to OH groups	Weight ratio of Polyol to PAPI
0.065	1.30	100/140
0.175	1.32	100/150
0.370	1.35	100/165
0.510	1.38	100/180

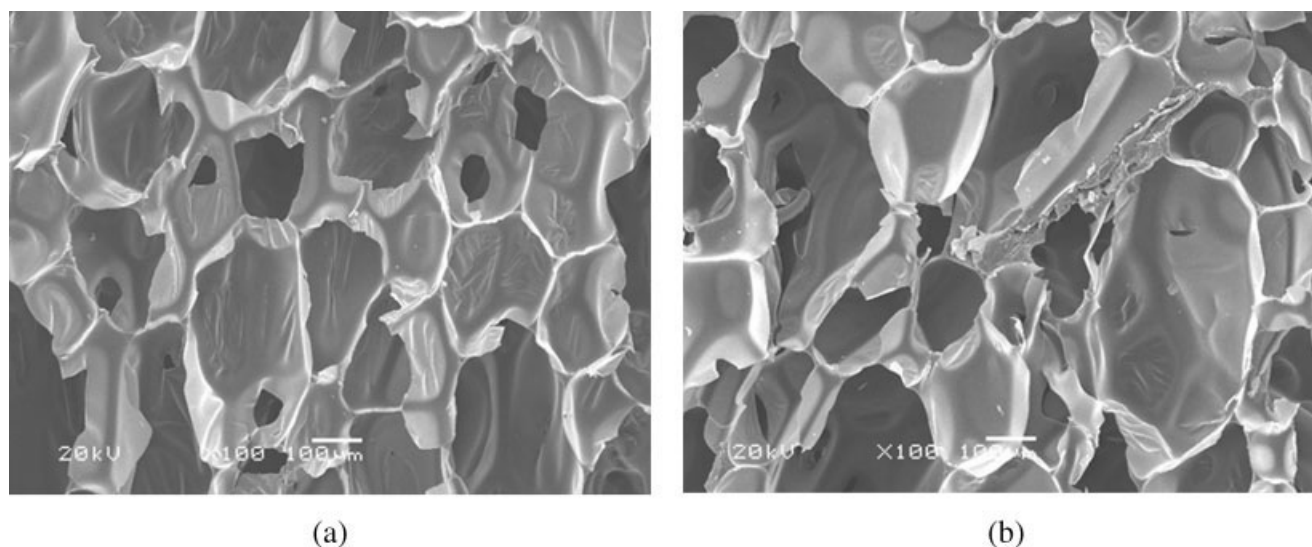


Figure 1 SEM micrographs of the pure RPUF and 10 wt % EG filled RPUF composites with a density of 0.065 g/cm^3 . (a) Pure RPUF with a density of 0.065 g/cm^3 ; (b) 10 wt % EG filled RPUF composites with a density of 0.065 g/cm^3 .

a density of 0.065 ± 0.004 , 0.175 ± 0.01 , 0.370 ± 0.02 , and $0.510 \pm 0.02 \text{ g/cm}^3$ were fabricated. The EG weight loading was from 5 to 20 wt %.

Characterization

Horizontal and vertical burning test

The horizontal and vertical burning tests were measured with a CTF-2 horizontal and vertical burning instrument (made in Jiangning Courty, China) according to the standard horizontal burning test, ASTM D 635-98, and the standard vertical burning test, ASTM D 3801-96. The specimens for measurement were machined into sheets $127 \times 13 \times 10 \text{ mm}^3$. According to the standard vertical burning test, ASTM D 3801-96, the procedures include (1) Clamping a specimen from the upper 6 mm of its length, with the longitudinal axis vertical; (2) Placing the test flame centrally under the lower end of the test specimen and maintaining a flame impingement time of $10.0 \pm 0.5 \text{ s}$; (3) Withdrawing the test flame and measuring the first after-flame time, t_1 , then inflicting the test flame again and measuring the second after-flame time, t_2 , with the same method. If $t_1 + t_2 \leq 10 \text{ s}$, the rating of the materials classifications is defined V-0. If $t_1 + t_2 \leq 30 \text{ s}$, the rating of the materials classifications is defined V-1. If the flame of the specimens burning reaches to the clamp, the specimens cannot be classified by the vertical burning test, and should be classified by the horizontal burning test. According to the standard horizontal burning test, ASTM D 635-98, the procedures include (1) Preparing fifteen bar specimens and marking each specimen with two lines perpendicular to the longitudinal axis of the bar, 25 ± 1

and $100 \pm 1 \text{ mm}$ from the end that is to be ignited; (2) Applying the test flame for $30 \pm 1 \text{ s}$ without changing its position. If the flame front does not pass the 25 mm reference mark and quenches after the ignition source is removed, the rating of the materials classifications is defined HB-1. Others can be classified through the rate of linear burning.

LOI test

The LOI test was performed using an HC-2 oxygen index test instrument (made in Jiangning Courty, China) in terms of the standard LOI test, ASTM D 2863-97. The specimens for measurement were sheets $127 \times 10 \times 10 \text{ mm}^3$.

SEM observation

The morphology of the samples including original and burned samples was observed by a JSM-9600 (JEOL, Japan) scanning electron microscopy (SEM) with an accelerating voltage 20 kV. All the samples were obtained by impact fracture at room temperature and the surfaces were coated with a layer of a conductive material.

RESULTS AND DISCUSSION

Morphology of EG/RPUF composites with different densities

Figure 1 shows SEM micrographs of the pure RPUF and 10 wt % EG filled RPUF with a density of 0.065 g/cm^3 . For the pure RPUF [Fig. 1(a)], the shape of cells is approximately polyhedron. Most cells are

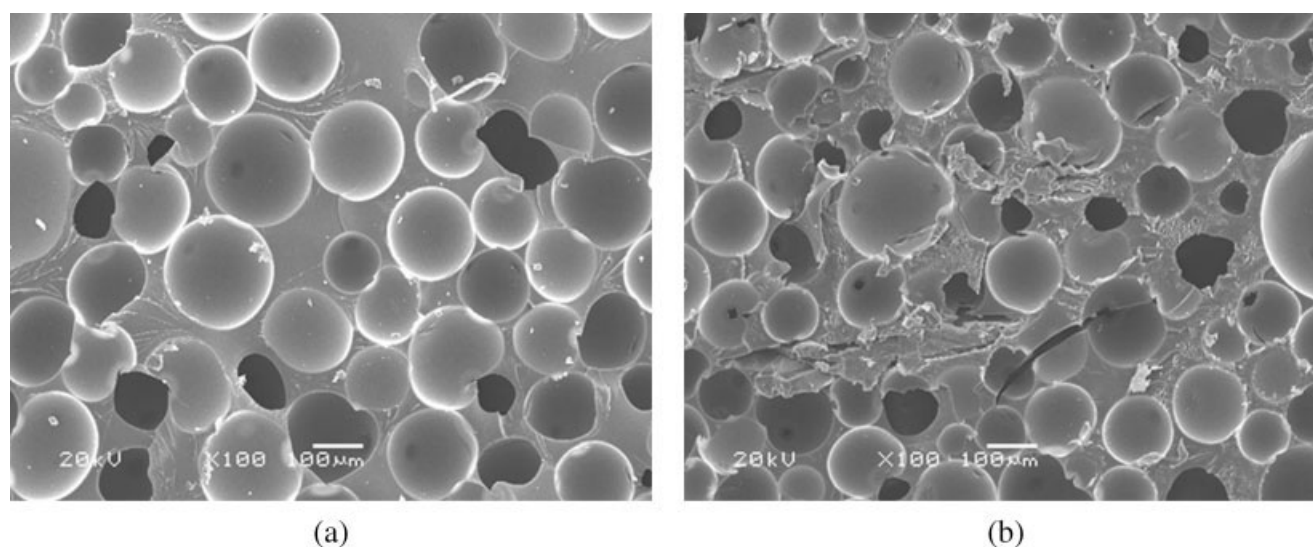


Figure 2 SEM micrographs of the pure RPUF and 10 wt % EG filled RPUF with a density of 0.510 g/cm^3 . (a) Pure RPUF with a density of 0.510 g/cm^3 ; (b) 10 wt % EG filled RPUF composites with a density of 0.510 g/cm^3 .

closed. By analyzing the images, the average thickness of cell wall and the average equivalent diameter can be obtained (from the high magnification image that was not shown here). The average thickness of cell walls is only $1.38 \mu\text{m}$, which is very low because of the excessively low density. There are some holes in the thin cell wall formed due to local internal stress or unbalanced foam growth. When the RPUF contains 10 wt % of EG [Fig. 1(b)], the shape and the wall thickness of the cells seem almost unchanged. However, the cell size became large compared to the pure RPUF. To compare the size of the cells in these two foams, the average equivalent diameter was estimated, which is the diameter of the circle whose area equals to the real cells. The average equivalent diameter of pure RPUF is $208.5 \mu\text{m}$, while $261.9 \mu\text{m}$ for the 10 wt % EG filled RPUF. This is a reasonable result because, at a low foam density, the addition of EG means the reduction of the PU resin content. Furthermore, this indicated that in the very low density foam, the blowing nucleation of EG particles is very weak. In the observed surface, we can find few EG particles, because there is too low EG loading in the ultra low density foam.

Figure 2 shows SEM micrographs of the pure RPUF and 10 wt % EG filled RPUF with a density of 0.510 g/cm^3 . Compared to the low density foam (Fig. 1), the cell shape of the high density foam is an approximate spheroid. There is a thick layer of matrix between cells, whose thickness is about $50.0 \mu\text{m}$. In addition, all the cells in Figure 2(a) were closed. According to Figure 2(b), it could be seen that the presence of EG caused more or less collapse and collision of the cells. Because there are more EG particles per volume in the high density foam than in the low density one, some particles can be observed.

It is interesting that the exfoliation of carbon flakes exists for some EG particles in the foam. EG is composed of some carbon flakes, as it is well known in the literature.^{20–22} In the process of foaming, the force generated by the rising matrix and gas led to the exfoliation of carbon flakes. The average cell diameter of pure RPUF with a density of 0.510 g/cm^3 [Fig. 2(a)] is $177.1 \mu\text{m}$, while $154.4 \mu\text{m}$ for the 10 wt % EG/RPUF composites at the same density. This indicates that the addition of EG particles results in a decrease of the cell size at a high density. Obviously, in the high density foam, the smaller size particles act as a blowing nucleation agent, which increased the concentration of cells and decreased the cell size.²²

Flame properties of EG/RPUF composites with different densities

To evaluate the flame retardant properties of EG/RPUF composites with different densities, horizontal and vertical burning tests were performed, and the results obtained are summarized in Table II and III. In the vertical burning test, the pure RPUF burned fast, resulting in failure of classification. Although the horizontal burning tests were implemented, most 10 wt % EG filled RPUF composites burned quite slowly and extinguished fast. As a result, these com-

TABLE III
Rate of Linear Burning of Different Density Pure RPUFs

Density (g/cm^3)	Rate of linear burning (mm/min)
0.065	405.6
0.175	108.0
0.370	49.3
0.510	23.4

TABLE IV
Classification of Different Density EG/RPUF Composites by Vertical Burning Test

Density (g/cm ³)	After-flame time after the first flame impingement, <i>t</i> ₁ (s)	After-flame time after the second flame impingement, <i>t</i> ₂ (s)	Rating
0.065	187.0	–	–
0.175	24.5	1.8	v-1
0.370	2.7	1.7	v-0
0.510	2.0	1.3	v-0

posites could also not be classified except the specimens with a low density of 0.065 g/cm³.

Table III shows that the burning rate of pure RPUF increases with decrease in the foam density. When the density of pure RPUF increases from 0.065 to 0.510 g/cm³, the rate of linear burning decreases from 405.6 to 23.4 mm/min. From Table IV, it was found that the low density 10 wt % EG/RPUF composite (0.065 g/cm³) could not be classified by the vertical burning test, but the flame retardant properties of 10 wt % EG/RPUF composites are greatly improved with the foam density. As the value is above 0.37 g/cm³, the addition of 10 wt % of EG brought out excellent fire properties. Its after-flame time after the first and second flame impingement is less than 10 s, and so the burning test achieves V-0 rating.

As shown in Table V, the flame retardant properties of EG/RPUF composites was improved with increase in EG loading. But these composites could not be classified by the vertical burning test. As the content of EG is below 10 wt %, the flame retardant ability of these composites can be distinguished merely by the rate of linear burning. Moreover, the rate of linear burning decreased to 91.7 mm/min from 405.6 mm/min, while the content of EG increased from 0 to 5 wt %. As the content of EG is above 10 wt %, the composites could comply with or exceed the test rating of the horizontal burning tests. For example, 10, 15, and 20 wt % EG/RPUF composites all passed HB-1 rating test. The flame retardant properties of EG/RPUF composites with a high density increase with increase in EG loading.²²

TABLE V
Classification of 0.065 g/cm³ EG/RPUF Composites Filled with Various Contents of EG Particles by the Horizontal Test

Content of EG (wt %)	Rating
0	405.6 mm/min
5	91.7 mm/min
10	HB-1
15	HB-1
20	HB-1

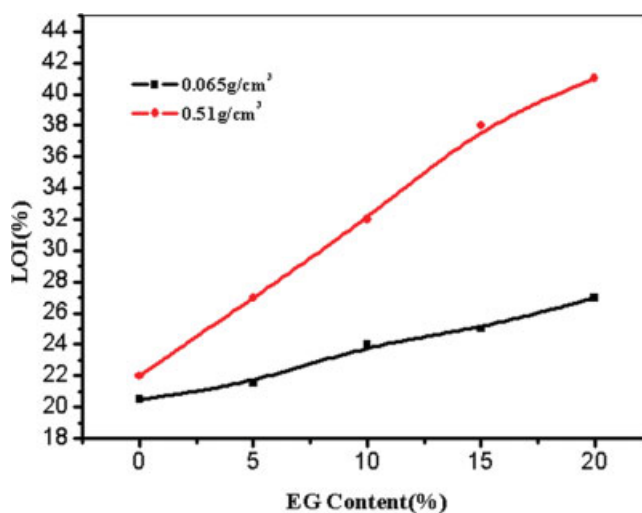


Figure 3 LOI of EG/RPUF composites as a function of EG loading at a fixed density. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

LOI test was also employed to determine the fire-retardant properties of the RPUF. The LOI of the EG/RPUF composites as a function of the EG content was shown in Figure 3. At the same density, the LOI considerably rises with increase in the EG content, being in accordance with our previous results.²² Moreover, the trend of the LOI increase is more obvious for the composite with a higher density. When the content of EG in the 0.065 g/cm³ composite increased from 5 to 20 wt %, the LOI increased from 21.5 to 27 vol %. In contrast, when the composite was 0.510 g/cm³, under the same conditions, the LOI rose from 27 to 41 vol %.

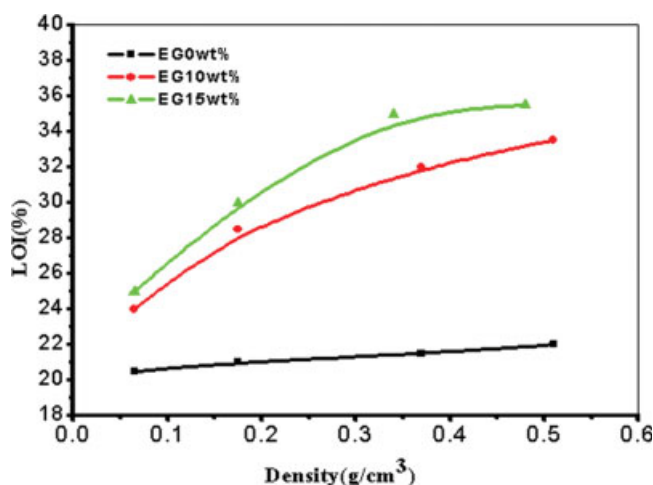


Figure 4 LOI of EG/RPUF composites as a function of the density at the same content of EG. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

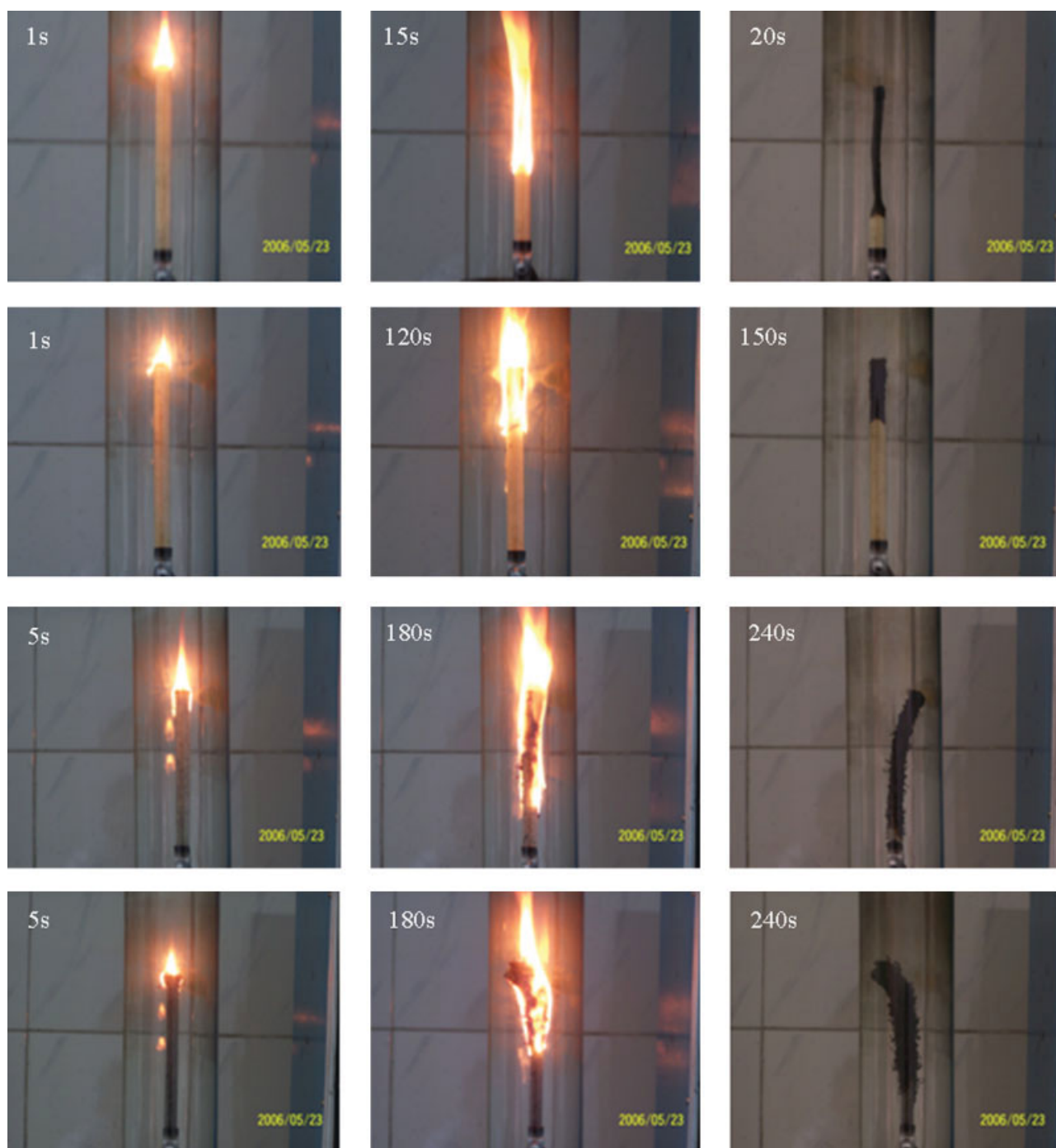


Figure 5 Optical images taken during burning of pure RPUF and EG/RPUF composites at different oxygen concentrations. (a) Pure RPUF with a density of 0.065 g/cm^3 ; oxygen concentration, 22.5 vol %; (b) pure RPUF with a density of 0.510 g/cm^3 ; oxygen concentration, 24 vol %; (c) 10 wt % EG/RPUF with a density of 0.065 g/cm^3 ; oxygen concentration, 26 vol %; (d) 10 wt % EG/RPUF with a density of 0.510 g/cm^3 ; oxygen concentration, 34 vol %. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Figure 4 shows LOI curve of EG/RPUF with different densities at the same EG content. When the density of pure RPUF increases from 0.065 to 0.510 g/cm^3 , the LOI has only a little increase, from 20.5 to 22.0 vol %. However, the great difference of LOI exists for the RPUF composites with

more than 10 wt % of EG, as shown in Figure 4. It is interesting that the increased density results in better fire-retardant properties, especially for the foam with a higher EG loading. For instance, when the density is $\sim 0.5100 \text{ g/cm}^3$, the LOI is as high as 33.5 vol %.

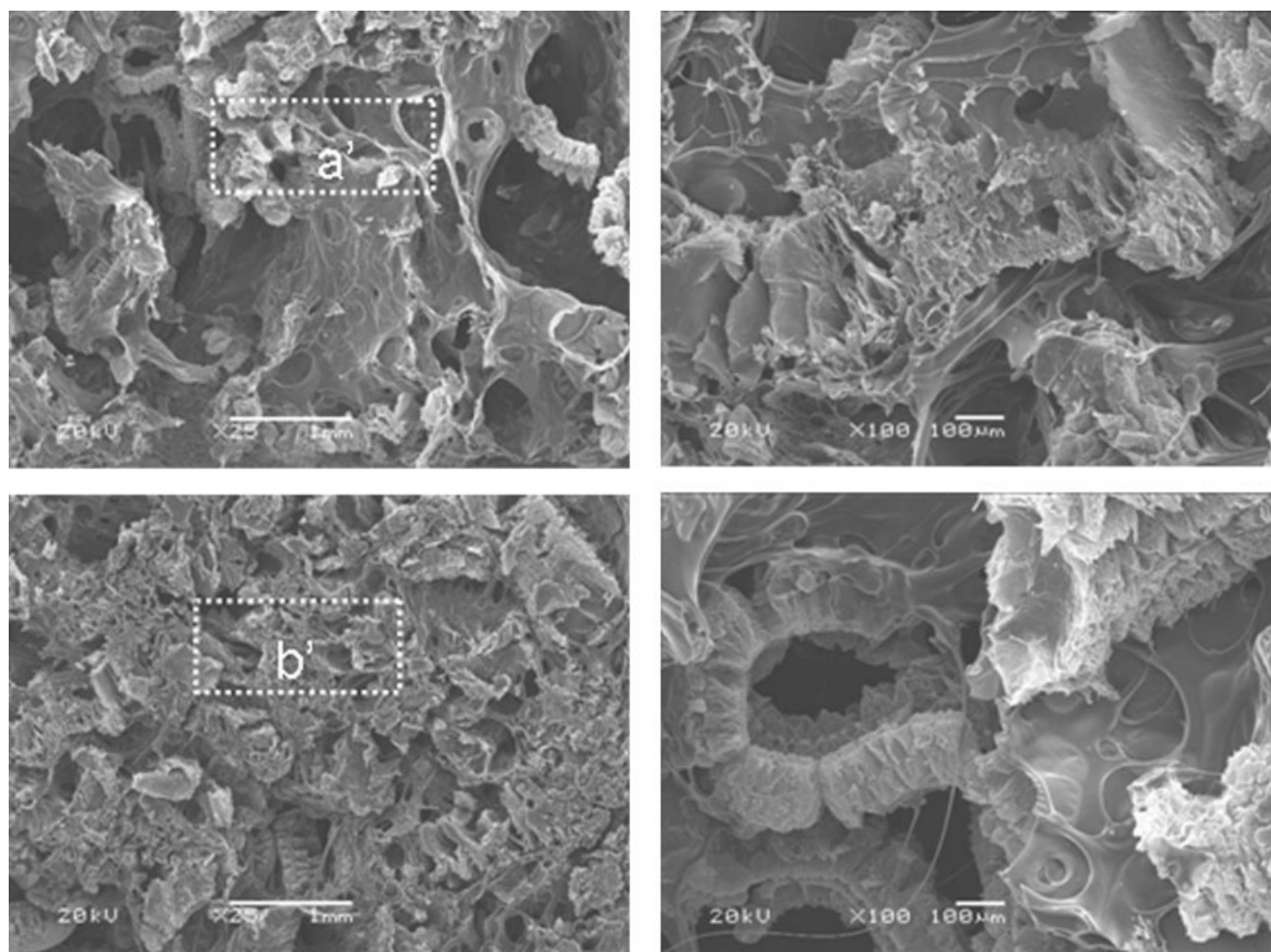


Figure 6 SEM micrographs of the burned 10 wt % EG/RPUF: (a) burned 0.065 g/cm³ 10 wt % EG/RPUF; (b) burned 0.510 g/cm³ 10 wt % EG/RPUF; (a') high magnification of burned 0.065 g/cm³ 10 wt % EG/RPUF; (b') high magnification of burned 0.510 g/cm³ 10 wt % EG/RPUF.

The optical images shown in Figure 5 depict the burning process of the RPUF without and with EG particles. Note that because of different flame retardant properties, different oxygen concentrations were applied so as to ignite the samples. Figure 5(a,b) shows the burning of 0.065 and 0.510 g/cm³ pure RPUF, respectively. The low density (0.065 g/cm³) pure RPUF burned fast, and after quenching the highly collapsed and carbonized material was left. For the high density (0.510 g/cm³) pure RPUF, at the oxygen concentration of 22.5 vol %, the burning velocity decreased obviously compared with the 0.065 g/cm³ one, and the burned sample had little change in size, implying reduced destruction of the material by flame. Figure 5(c,d) shows the burning of 10 wt % EG filled 0.065 and 0.510 g/cm³ RPUF, respectively. After burning, both samples exhibited a layer of non cohesive black char. Comparing Figure 5(c,d), a large volume of charred materials were generated on the surface of the high density sample.

To further reveal the mechanism of the density affecting the flame retardant properties of EG filled RPUF, the fracture surfaces around the burned char were observed. Generally, the EG filled sample can be divided into three sections from the exterior toward the center after burning, including burned, interface and the inner layers.²² The exterior zone characterized by “wormlike” structure is the burned layer; the cell layer that exhibits plastic deformation due to thermal treatment is the interface; and the inner layer is the virgin material that is not affected by the flame or the heat.

Figure 6(a,b) shows the burned layer of the 10 wt % EG/RPUF composites with a density of 0.065 and 0.510 g/cm³, respectively. Both images show that the cells are severely destroyed, and there are only some wormlike expanded graphite agglomerates and charred materials in the burned layer. The high magnification SEM micrographs [Fig. 6(a',b')] indicate that EG particles in the exterior of the burned com-

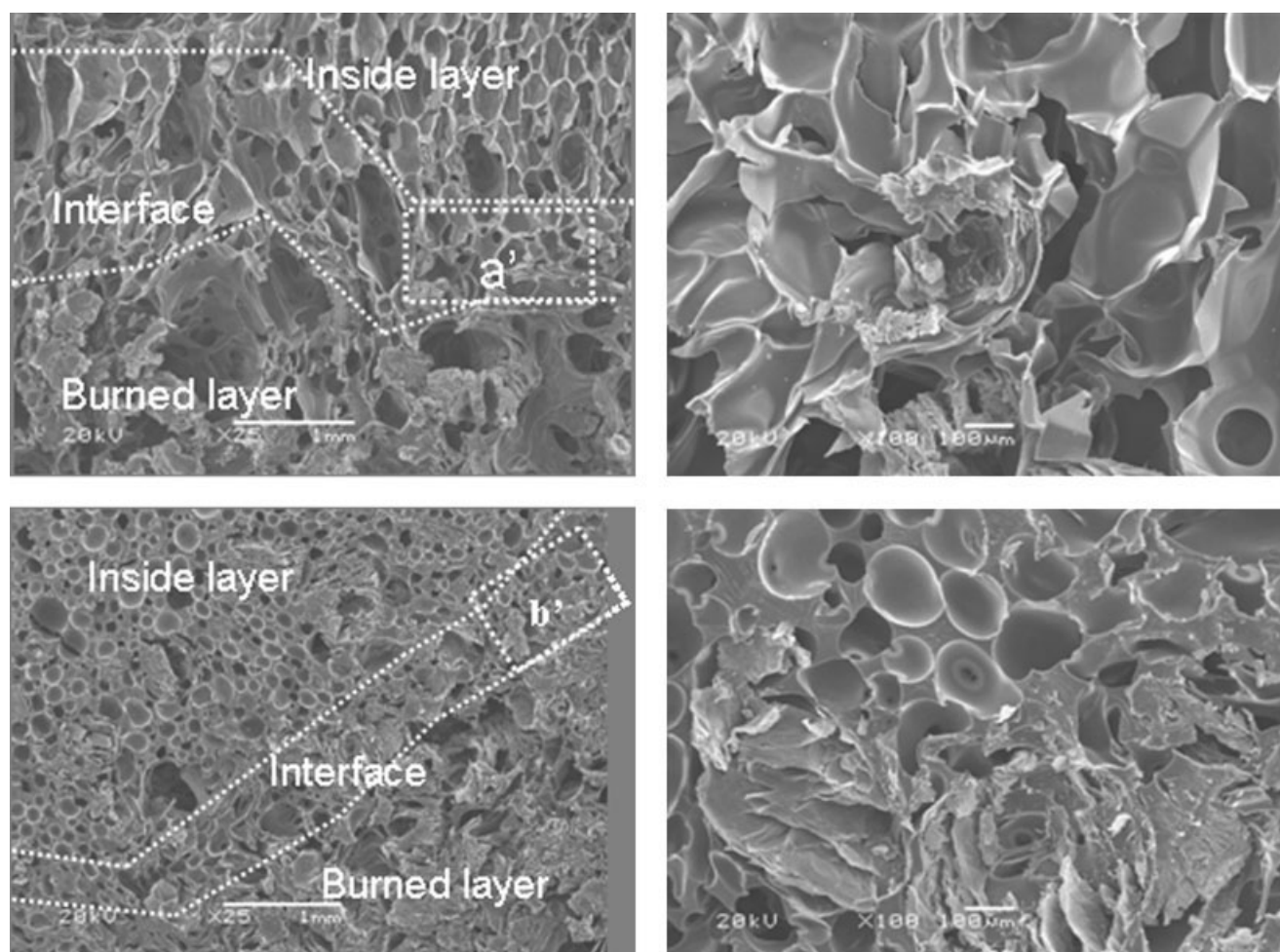


Figure 7 SEM micrographs of the fractured surfaces of the burned 10 wt % EG/RPUF composites: (a) 0.065 g/cm³ 10 wt % EG/RPUF; (b) 0.510 g/cm³ 10 wt % EG/RPUF; (a') high magnification of (a); (b') high magnification of (b).

posite have expanded many times and the residue was mainly composed of expanded graphite. Comparing different density foams, one can find that the high density foam tended to produce dense residue. In other words, in the low density foam, there are more holes appearing in the charred layer.

Figure 7(a,b) shows the overall morphology of the three sections for 0.065 and 0.510 g/cm³ RPUF composites with 10 wt % EG, respectively. From Figure 7(a,b), it can be found that the interface layer becomes thinner with the increase in density, indicating less plastic deformation of the foams at higher density. Figure 7(a',b') exhibit the high magnification of the interface layer. There are still some expanded graphite and burned residue as observed in Figure 7(a'), and the cell structure was destroyed heavily. However, the interface layer of the 0.510 g/cm³ EG/RPUF composite was mainly composed of unexpanded graphite and anamorphic cell. The cell structure had only plastic deformation at the high temperature near the fire spots. So less destruction took place in the high density foam than the low density foam.

As mentioned earlier, the density greatly affects the flame retardant properties of pure RPUF and EG filled RPUF. With the increase of the density, the polyurethane resin matrix per volume increases. That is, the lower density foam becomes less cohesive, and its cell wall thinner when burning, There is a larger contact area between the resin matrix and the air (oxygen), thus resulting in a higher burning rate and a slight lower LOI values.

EG is a graphite compound intercalated by sulfuric acid between the carbon layers of graphite. When exposed to a heat source, carbon and sulfuric acid reacts and generates gases that lead to the voluminous expansion of EG. At the same time, the expansion of EG forms worm-like structures to insulate heat and flame, which provide fire performance to the flammable materials.^{20,21} More worm-like structures means better flame retardant efficiency. For the EG-filled RPUF composites, although the weight ratio of the EG to the resin matrix was fixed at a constant level, the real EG loading per volume increases with increase in the density. At a lower density,

less EG loading cannot sufficiently prevent heat diffusion and insulate the flame, and hence the heat leads to the decomposition of matrix. As the density rises, the EG loading per volume increases. When exposed to the heat source, EG particles expand and cover the overall surface to produce fire retardancy. This observation implies that a weight percent of a flame retardant additive that achieves satisfactory flame retardancy for a certain density foam cannot effectively be applied for another density foam.

CONCLUSIONS

The cell shape of high density RPUF and EG/RPUF composites is approximately spheroid, while the cell shape of low density RPUF or EG/RPUF composites is polyhedron, i.e., density affects the geometry of RPUF or EG/RPUF. In the higher density foam, the smaller size EG particles acted as a blowing nucleation agent, thus resulting in an increment of the amount of cells and reduction of the cell size, but for the lower density foam, EG particles had little such effect.

EG can effectively improve the flame retardant of RPUF, but at the fixed EG weight ratio, higher density foam had more considerable flame retardancy. The level of the LOI and the horizontal and vertical burning tests of RPUF or EG/RPUF composites was enhanced with the increase of the foam density at the same EG weight ratio.

After burning, the low density (0.065 g/cm^3) pure burned RPUF produced the highly collapsed and carbonized material, while the high density (0.510 g/cm^3) pure RPUF had little change in size and reduced destruction of the material. Though all EG-filled RPUF samples exhibited a layer of noncohesive black char after burning, a large volume of charred materials were generated on the surface of the high density one.

The higher density EG/RPUF composites had a more compact outer layer (burned layer) after burning, in which more wormlike materials composed of expanded graphite particles appeared. In addition, higher foam density led to less plastic deformation

in the interface layer between the burned and the inside layers.

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