

# Study on the Mechanical Properties of Hybrid Reinforced Rigid Polyurethane Composite Foam

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**ABSTRACT:** The mechanical properties of hybrid reinforced rigid polyurethane (PU) foams were investigated with the reinforcing agent SiO<sub>2</sub> and fibers. The effect of content of SiO<sub>2</sub> and fibers and the effect of length of fibers on the properties of the PU composite foam were emphatically analyzed. The experiment results show that the tensile strength of the PU composite foam is optimal when the content of SiO<sub>2</sub> and glass fiber is 20 and 7.8%, respectively.

Furthermore, the reinforcing effect of glass fiber, Nylon-66 fiber, and PAN-matrix carbon fiber were compared and the results show that the tensile strength of the PU composite foam reinforced with 3–5% carbon fiber is optimal. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 92: 1493–1500, 2004

**Key words:** polyurethane; fiber; particle; microstructure; strength

## INTRODUCTION

Rigid polyurethane (PU) foam is one of the most important materials used in the construction industry and the global appliances (freezers, refrigerator, etc.) industry with excellent characteristics as , heat-insulation, sound-absorption, as well as being lightweight and shock-proof. As a structural material, however, it has to possess some strength, hardness, and toughness.<sup>1–2</sup> Common methods to improve the mechanical properties of the rigid PU foam will increase its density and filling reinforcing agent; therefore, the recent study of the effect of reinforcing agent on the mechanical properties of the rigid PU foam has been one of the most attractive points.<sup>3–10</sup>

Hybrid fiber-reinforcing and particle-filling rigid PU foam is a composite material constituted with resin, fibers, particles, and cells. It is a new trend used to prepare the rigid PU foam with high performance.<sup>11</sup> In this study, glass fiber, nylon-66 fiber, and PAN-matrix carbon fiber as well as silicon dioxide powder are filled into the rigid PU foam as reinforcing agent and the physical and mechanical properties of the hybrid fiber-reinforcing and particle-filling rigid PU foams are described in detail.

## EXPERIMENTAL

### Raw materials

The materials used in this study were from commercial sources. The polymethylene polyphenylisocyanate

(PAPI) was supplied by Shanghai Plastics Co. (Shanghai, China). The polyether polyol, synthesized from glycerol and mannitol, Freon-11, used as a physical-chemical blowing agent, 33LV used as an amine catalyst, and 1,3-butane diol used as a toughening agent were also supplied by Shanghai Plastics Co. Glass fiber with a diameter of 13–15  $\mu\text{m}$  was supplied by Yaohua Glass Fiber Co. (Shanghai, China). Nylon-66 fiber with a diameter of 35  $\mu\text{m}$  was supplied by Qiu-long Chemical Engineering Co. (Shanghai, China). PAN-matrix fiber with a diameter of 6  $\mu\text{m}$  was supplied by Shanghai Xinxing Carbon Co. (Shanghai, China). The powder of silicon dioxide with a size of 30–50  $\mu\text{m}$  was supplied by Shanghai Hengxin Chemical Agent Co. (Shanghai, China). A-1100 used as a coupling agent was supplied by Shanghai Huarun Chemical Engineering Co. (Shanghai, China). The polyol and 1,3-butane diol were dehydrated before use at 90°C for 24 h in a vacuum oven. The others were used as received.

### Experimental design and formulations

The effects of the following variables in the foam formulation on the properties of the hybrid reinforced rigid PU foams were studied: (1) content of silicon dioxide powder (parts per hundred polyols): 0, 10, 20, and 30; (2) content of glass fiber (parts per hundred polyols): 0, 3.5, 7.5, 9.0, and 12.5; (3) length of glass fiber: 0, 3, 5, 10, 12, and 15 mm; (4) reinforcing effect of different fiber (glass fiber, nylon-66 fiber, and PAN-matrix fiber). Other factors in the foam formulation, such as catalysts, surfactant, toughening agent, and isocyanate index, were kept constant. The foam formulation for the hybrid reinforced rigid PU foam is

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**TABLE I**  
**Formulation for the Hybrid Reinforced Rigid Polyurethane Foam**

Ingredients	Parts by weight
Component A	
Polyol	100
Silicon dioxide powder	0, 10, 20, and 30
Glass fiber	0, 3.5, 7.5, 9.0, and 12.5
Catalysts	3.0
Surfactant	5.0–6.0
Blowing agent (Freon-11)	1.0
Toughening agent	6.0–7.0
Component B	
PAPI	135, 140, 145

listed in Table I. The amount of isocyanate added in each formulation was based on total hydroxyl content of the polyether polyol. Three replicate foams were produced with each formulation.

### Sample preparation

The rigid PU foam samples reinforced with different reinforcing agents were synthesized by the casting method. All chemicals in Component A apart from reinforcing agent were put into the reactor by weight and mixed for 80–180 s with a stirrer and the rotate speed was set at 3000 rpm throughout the mixing; then stirring stopped and the mix was allowed to degas. After the temperature of the system was adjusted at 18°C, PAPI (Component B) was added and then reinforcing agents were rapidly put in, and stirring continued for another 15–30 s. Finally, the reactants were poured immediately into a close aluminum mold (200 × 200 × 100) to produce the hybrid reinforced rigid PU foams and cured for 2 h at 100°C. Considering the great effect of density of the PU composite foams on their mechanical properties, the density of the rigid PU foam is kept constant (0.65 g/cm<sup>3</sup>) so that the reinforcing effect of reinforcing agent can be compared in the same condition. To exert the reinforcing function of reinforcing agent to the highest extent, reinforcing agent has to be treated in advance with silane coupling agent. The detailed processing is listed as follows: first silicon dioxide powders dipped with 1% KH550 of tetrahydrofuran solution were dried in a vacuum oven at 90°C and 50 mmHg overnight; then the dried powders were spread on the surface of fibers treated with the coupling agent and all reinforcing agents were mixed with some materials of component A. Finally, they were filled into the total mixing system.

### Disposal of reinforcing agent

Interface is a very important microstructure of composite materials and excellent performance of reinforcing

agent depends on the resin-matrix and the interface between resin and reinforcing agent. Interface determines stress transmission between resin-matrix and reinforcing agent and integrity of interface determines general performance of composite materials. Disposal to the surface of reinforcing agent is the most effective way to enhance the adhesive strength of interface.

SiO<sub>2</sub> powders would be disposed of by a coupling agent as they could not be compatible with the system. A silicon coupling agent, such as KH550, was optimal as stronger hydroxyl existed on the surface of SiO<sub>2</sub> powders. A layer of film formed after the hydroxyl on the surface of SiO<sub>2</sub> powders reacting with couple agent could be compatible with the system so that the mechanical properties of the rigid PU foam obviously improved.

Before disposal, the surface of the fibers was slippery and chemically inert and could not soak with resin matrix so that the general performance of the composite materials was badly influenced. The process of disposal took place after the following: the fibers were soaked in the acetone and oxidated at 200°C after being dried. Oxygenic functional groups formed on the surface of fibers and the surface got rougher. Finally, the surface of the fibers was disposed of with KH550 and the interface between the resin matrix and fibers improved.

### Measurements

#### Water absorption

Water absorption of the hybrid reinforced rigid PU foam was measured according to GB 1034-86.<sup>12</sup> Prepared specimens were placed in an oven and dried for 1 h at 80°C and then weighted up. The mass of the dried samples was set as *M*. After dipped in water for 24 or 48 h, the samples were weighted up again and the mass of the soaking samples was set as *N*. Then, the water absorption (*W<sub>A</sub>*) of them was calculated as

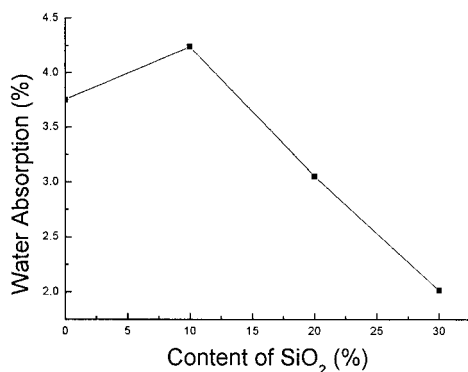
$$W_A = (N - M) / N \times 100\%$$

#### Hardness

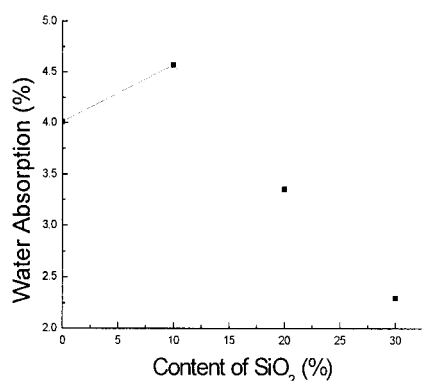
S-D hardness of the hybrid reinforced rigid PU foam was measured by means of XHS Rubber-Plastic Sclerometer (Yingkou, Liaoning, China) according to GB 2411-80.<sup>13</sup>

#### Impact strength

Impact strength of the hybrid reinforced rigid PU foam was measured on a XJJ-5 Impacting Test Machine (Chengde, Hebei, China) according to GB 1843-80.<sup>14</sup>



a. 24h



b. 48h

**Figure 1** Effect of SiO<sub>2</sub> content on water absorption of the PU composite foam.

#### Tensile intensity and rate of elongation

Tensile strength and rate of elongation of the hybrid reinforced rigid PU foam were measured on a DL-1000B Electronic Pulling Test Machine (Changchun, Jilin, China) according to GB/T 9641-88.<sup>15</sup>

#### Analysis of microstructure

Photographs of fracture of the hybrid reinforced rigid PU foam were observed on an S-515 Philips SEM (Netherlands). Fiber distribution morphologies were observed on an N-625 Philips cinematography microscope.

## RESULTS AND DISCUSSION

### Effect of SiO<sub>2</sub> content on water absorption of the PU composite foam

To study the effect of SiO<sub>2</sub> content on physical and mechanical properties of the PU composite foam, the hybrid reinforced rigid PU foams reinforced with both

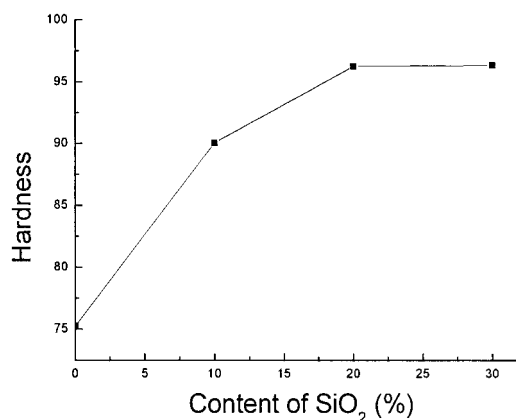
7.8% fiber and SiO<sub>2</sub>, whose content ranges from 0 to 30%, were prepared and their water absorption, hardness, impact strength, and tensile strength were measured.

The rigid PU foams are close systems with some perforation and the netting structure of the resin matrix has the quality of water absorption, which is relevant to the content of inorganic filler. The effect of SiO<sub>2</sub> content on water absorption of the PU composite foam (24 and 48 h) is shown in Figure 1(a, b).

As shown in Figure 1(a), water absorption of the PU composite foam increased from 3.75 to 4.25% as the content of SiO<sub>2</sub> increased from 0 to 10% and water absorption then decreased from 4.25 to 2.0% as the content of SiO<sub>2</sub> increased from 10 to 30%. It means that water absorption gets the maximal value when the content of SiO<sub>2</sub> is 10%. What caused this result is that SiO<sub>2</sub> is a polar material and there are many hydroxyl groups and carboxyl groups on its surface; moreover, its specific surface area is huge. Although water is also a polar material, water absorption of the PU composite foam will be sure to increase as SiO<sub>2</sub> is filled into them according to the principle that organic components with similar solubility are mutually soluble. However, a protective layer, which prevented the PU composite foam from absorbing water, was formed on the surface of cells as SiO<sub>2</sub> is filled into voids formed after cells were stacked close; therefore, water absorption of the PU composite foam decreased as the content of SiO<sub>2</sub> increased again after the content of SiO<sub>2</sub> is 10%.

### Effect of SiO<sub>2</sub> content on hardness of the PU composite foam

Hardness of the hybrid reinforced rigid PU foam can be increased by mixing inorganic fillers. The effect of SiO<sub>2</sub> content on the hardness of the PU composite foam is shown in Figure 2.



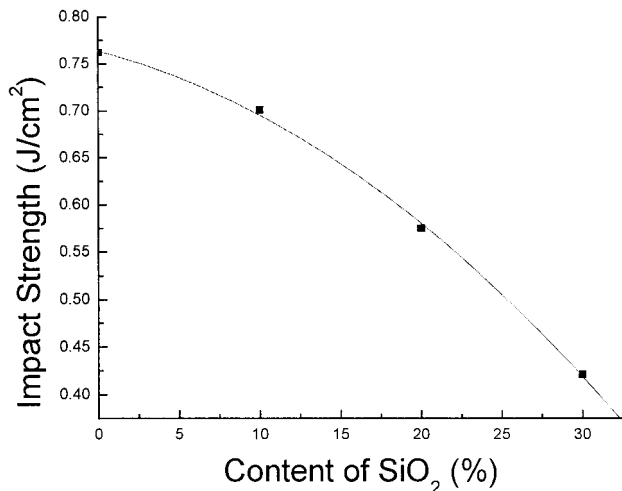
**Figure 2** Effect of SiO<sub>2</sub> content on hardness of the PU composite foam.

As shown in Figure 2, the hardness increased from 75 to 96% as the content of  $\text{SiO}_2$  increased from 0 to 30%. The hardness increased faster when the content of  $\text{SiO}_2$  increased from 0 to 10%. Afterward, the extent of increment got slower, and the hardness of the PU composite foam approached a constant value after the content of  $\text{SiO}_2$  was 30%. This result can be explained by the fact that hardness of the rigid  $\text{SiO}_2$  particle is larger than that of the matrix of the PU composite foam. Hardness of the PU composite foam will be sure to increase after  $\text{SiO}_2$  is filled in them. However, as shown in Figure 2, the hardness of the system increased less when the content of  $\text{SiO}_2$  is greater than 20%.

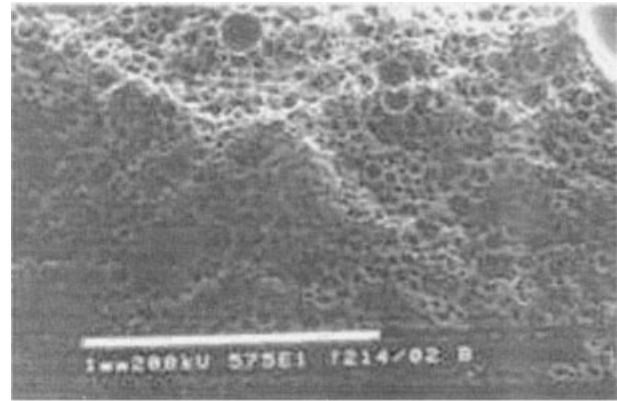
#### Effect of $\text{SiO}_2$ content on impact strength of the PU composite foam

Inorganic filler has a large effect on impact strength of the hybrid reinforced rigid PU foam, and the effect of  $\text{SiO}_2$  content on impact strength of the PU composite foam is shown in Figure 3.

As shown in Figure 3, the impact strength decreased from 0.77 to 0.44  $\text{J}/\text{cm}^2$  as the content of  $\text{SiO}_2$  increased from 0 to 30%. This result can be explained by the weak interface between  $\text{SiO}_2$  powders and resin matrix and uneven dispersal of  $\text{SiO}_2$  powders. Polarity between  $\text{SiO}_2$  powders and surface of matrix differed largely and were not mutually soluble so that  $\text{SiO}_2$  powders could not be dispersed evenly. The fractured morphology of the PU composite foam with 30%  $\text{SiO}_2$  content is shown in Figure 4. It can be seen from Figure 4 that there was an obvious conglomeration of  $\text{SiO}_2$  powders in the PU composite foam. The conglomeration of  $\text{SiO}_2$  powders could form a stress-concentrating area, which causes the system to weaken.



**Figure 3** Effect of  $\text{SiO}_2$  content on impact strength of the PU composite foam.



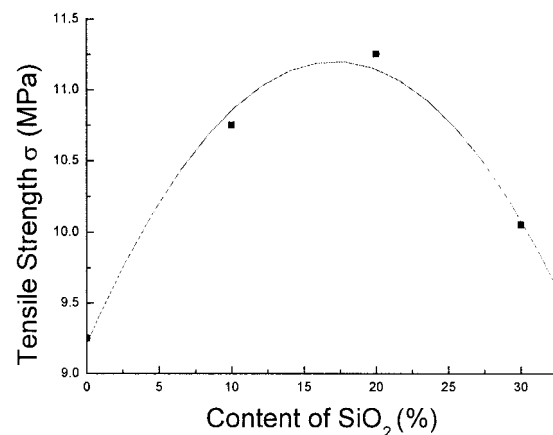
60×

**Figure 4** The SEM photograph of fracture of the PU composite foam with 30%  $\text{SiO}_2$  content.

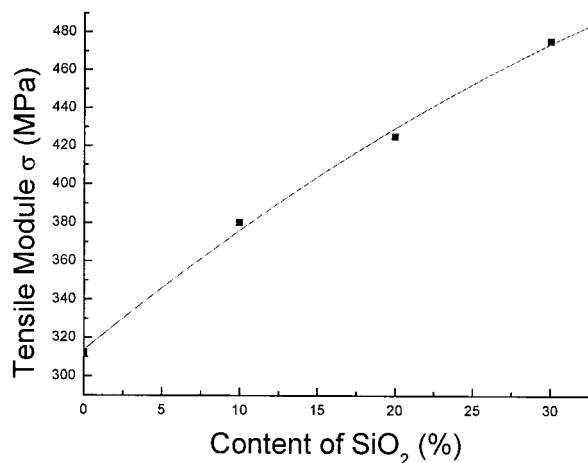
#### Effect of $\text{SiO}_2$ content on tensile strength of the PU composite foam

Filler could extrude cells in the hybrid reinforced rigid PU foam so that it could affect tensile property of the materials. The effect of  $\text{SiO}_2$  content on the tensile strength and tensile module of the PU composite foam is shown in Figures 5 and 6.

As shown in Figure 5, the tensile strength of the hybrid reinforced rigid PU foam increased as the content of  $\text{SiO}_2$  is less than 20%; however, the reinforcing effect is not obvious. The tensile strength of the PU composite foam got the maximal value of 11.3 MPa when the content of  $\text{SiO}_2$  is 20%; afterward, this value decreased as the content of  $\text{SiO}_2$  increased. This is due to the filling of  $\text{SiO}_2$  powders.  $\text{SiO}_2$  powders extruded cells continually as they filled into voids formed after the cells were stacked close. Thus, cell size got less and less and the trend of the cells springing back was more



**Figure 5** Effect of  $\text{SiO}_2$  content on tensile strength of the PU composite foam.



**Figure 6** Effect of SiO<sub>2</sub> content on tensile module of the PU composite foam.

and more obvious so that tensile strength of the system would be sure to increase. In addition, when SiO<sub>2</sub> powders with small size were filled into the PU matrix, they acted as nucleating agents to some extent in the nucleation process. Densities of bubbles increased and aperture of cells decreased. Density of the system increased and cell size decreased to a very small value when the content of SiO<sub>2</sub> increased more; thereby, those voids got smaller and smaller so that the SiO<sub>2</sub> powders that could fill in also got smaller and smaller. However, particles within a conglomerated group will only be loosely bound to each other, which will weaken the composite when under tensile stress. Thus, the tensile strength of the PU composite foam would decrease after the content of SiO<sub>2</sub> was more than a critical value. However, change of tensile module is not the same as that of tensile strength. As shown in Figure 6, the tensile module of the hybrid reinforced rigid PU foams increased obviously as the content of SiO<sub>2</sub> increased, which is caused by the finding that the module of SiO<sub>2</sub> powders are much larger than that of matrix. One study showed that the module of the system reinforced with rigid particles would increase even if there was no strong interaction between inorganic particles and resin matrix.<sup>16</sup>

#### Effect of fiber length on the tensile strength of the PU composite foam

To compare the effect of fiber on the tensile strength of the PU composite foam, a series of samples of the hybrid reinforced PU composite foam with 20% SiO<sub>2</sub> and various contents of fibers were prepared and their tensile strength was measured.

The fiber length has shown great influence on the tensile strength of the hybrid reinforced rigid PU foam. If fibers are too short, they are not easy to be adhered by the outside force, whereas if they are too

long, they are rather difficult to disperse evenly and they may conglomerate and bend easily in the PU composite foam. Thus, local stress-concentrating areas are formed and a perfect reinforcing effect cannot be achieved. As early as the 1970s, Cotgreave considered that when fibers are used as reinforcing agent of the rigid polyurethane foam (RPUF), there is an optimal value for fiber length. The reinforcing effect will obviously not be improved even if the fiber length surpasses the optimal value. The critical fiber length value can be expressed as<sup>17</sup>:

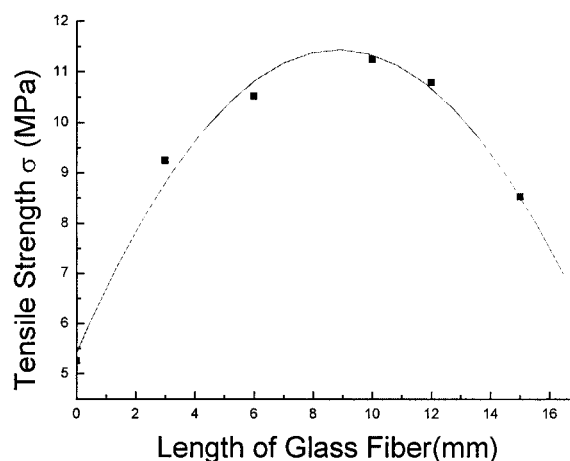
$$l_c = 2\sigma_f \times r_f^2 / \tau_m \times d$$

where  $\sigma_f$  is fiber strength,  $r_f$  is the ratio of length-to-diameter of fiber,  $\tau_m$  is shear strength of the fiber surface, and  $d$  is the diameter of shearing strip. In Figure 7, the tensile strength of the PU composite foam reinforced with fibers varying in length is shown. As a result, the critical length of glass fiber was about 10 mm in such experimental situations where the tensile strength of the PU composite foam was 11.4 MPa. The optimal rate of length-to-diameter of fiber is about 140.

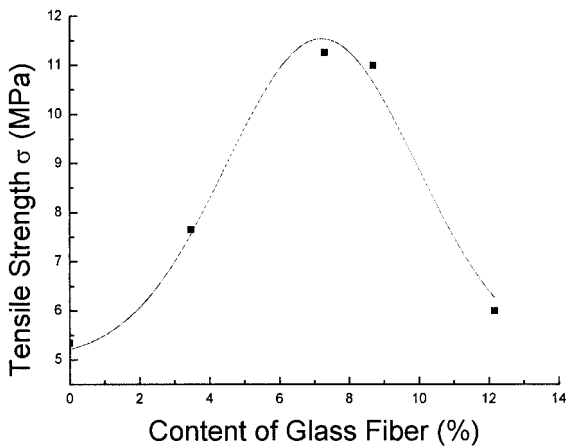
#### Effect of fiber content on the tensile strength of the PU composite foam

The fiber content has shown obvious influence on the tensile strength of the hybrid reinforced rigid PU foam. The relationship between tensile strength and the content of fiber is shown in Figure 8.

As shown in Figure 7, the tensile strength of the hybrid reinforced rigid PU composite foam increased when the content of fibers is less than 7.8%. The tensile strength got the maximal value of 11.3 MPa when the content of fibers is 7.8%. Afterward, this value decreased as the content of fibers increased. This is due to the fact that tensile strength of glass fiber is greater



**Figure 7** Effect of length of glass fiber on tensile strength of the PU composite foam.

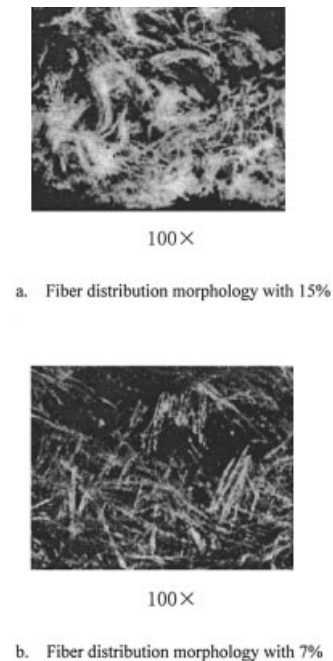


**Figure 8** Effect of the content of glass fiber on tensile strength of the PU composite foam.

than that of the resin matrix. When glass fibers are filled into the un-reinforced rigid PU foam, the tensile strength of the system will be sure to increase at first. With the continual increase of fibers, a tangle of fibers gets more and more serious so that the fibers are conglomerated in part of the area, which means that content of resin is excessively small in this area. In addition, conglomeration of resin forms local stress-concentrating area where density is correspondingly smaller so that the tensile strength of the PU composite foam decreases instead. In Figure 9, fiber distribution morphology in the PU composite foam with different fiber content is shown. As shown in Figure 9, fiber distribution is even in the PU composite foam when the content of fibers is 7%, whereas fibers distribution is tangled in the PU composite foam when the content of fibers is 15%.

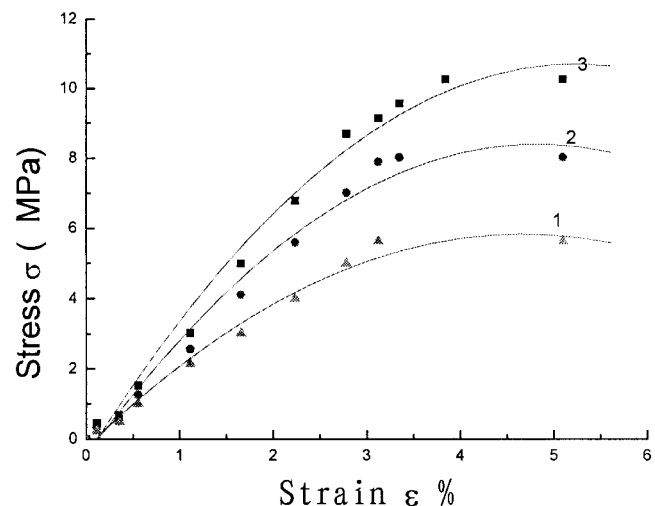
#### Effect of various fibers on the tensile strength of the PU composite foam

Different fibers have different tensile strengths and will tangle with the rigid PU foam with different modes; thus, they exhibit different reinforcing effects on the rigid PU foam. In the experiment, the tensile strength of three kinds of hybrid reinforced rigid PU foam reinforced with glass fiber, nylon-66 fiber, and PAN-matrix carbon fiber, respectively, were compared. Here fiber length is kept to 3 mm and fiber content is kept to 3% of mass. In Figure 10, the stretching stress-strain curves of three samples of the PU composite foam whose densities are all  $0.45 \text{ g/cm}^3$ , reinforced with three kinds of fibers. As shown in Figure 10, tensile strength of the PU composite foam reinforced with PAN-matrix carbon fiber was the greatest; that of the PU composite foam reinforced with nylon-66 fiber took second place and that of the PU composite foam reinforced with glass fiber was the



**Figure 9** Fiber distribution morphology in the PU composite foam with different fiber contents.

poorest. In Figure 11, the SEM photographs of stretching fracture of the PU composite foam reinforced with three different kinds of fibers were shown. As shown in Figure 11(a), which is the SEM photograph tensile fracture of the PU composite foam reinforced with glass fibers parallel to fracture of the PU composite foam, on account of the fact that the orientation of transverse fibers is vertical to orientation of load,



**Figure 10** Stretching stress-strain curves of three kinds of fiber-reinforced PU composite foams: 1. glass fiber; 2. nylon-66 fiber; 3. PAN-matrix carbon fiber.

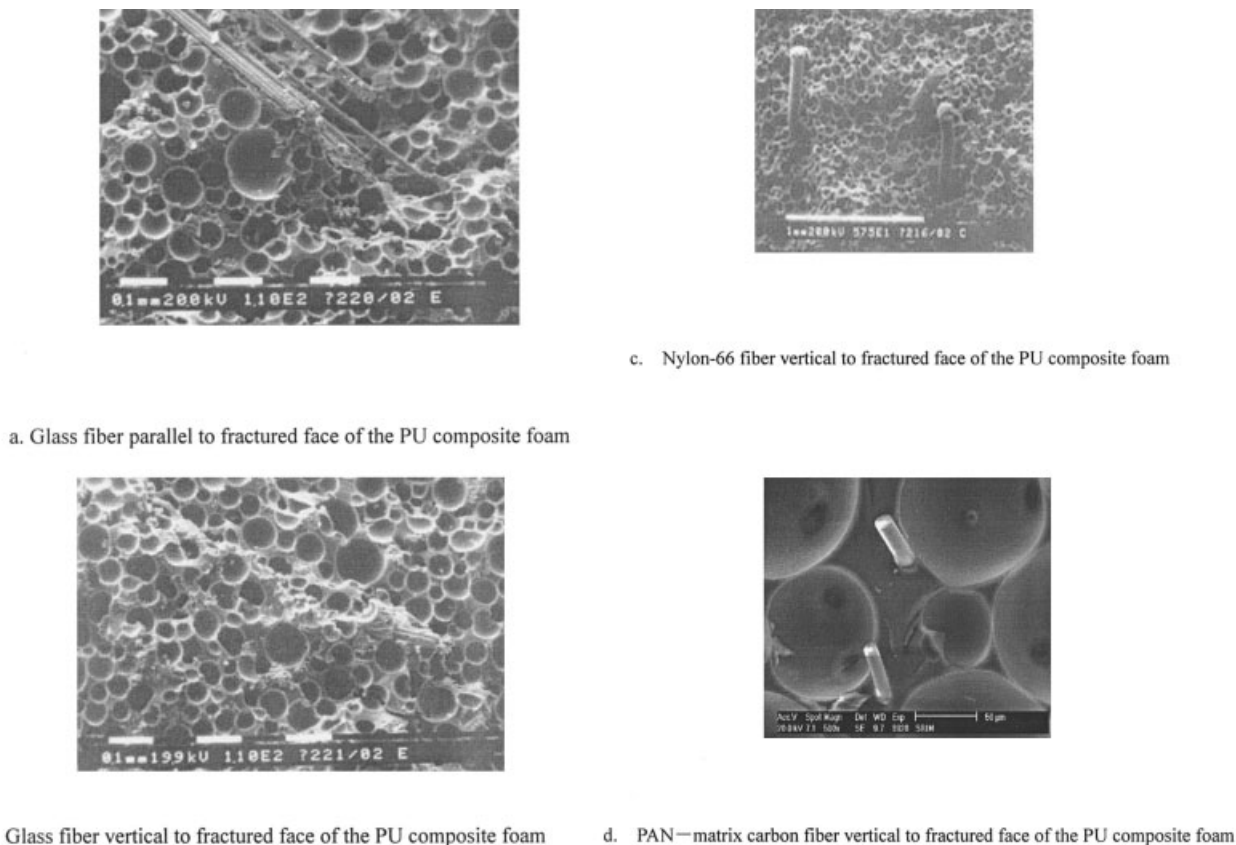


Figure 11 The SEM photographs of fracture of the PU composite foam reinforced with different kinds of fiber.

cracks would spread by getting across the interface between fibers and matrix; thus, a contribution of strength of transverse fibers to reinforcement of tensile strength is correspondingly small. In Figure 11(b–d), the SEM photographs show the tensile fracture morphology of the PU composite foam reinforced with glass fibers, nylon-66 fibers, and PAN-matrix carbon fibers, respectively, which are vertical to the fracture of the PU composite foam. As shown in these figures, the forced trails of longitudinal fibers were very obvious. These fibers broke down or extruded because of the effect of larger tensile stress. From these figures, holes that formed after some fibers were pulled out could be seen on the fracture, which showed that abruption indeed occurred between fibers and resin matrix. As a result, the mechanical properties of fibers could play an important role in reinforcing the tensile strength of the PU composite foam. Tensile performance of three kinds of fibers is listed in Table II. As shown in Table II, the tensile strength of PAN-matrix carbon fibers was the largest; that of nylon-66 fibers took second place and that of glass fibers was the smallest, which ranked in the same order as tensile strength of the PU composite foam showed in Figure 10.

CONCLUSION

(1) Hybrid reinforced rigid PU composite foam was prepared with PAPI, polyester polyol, 1,3-butane diol, silicone surfactant, 33LV as a catalyst, Freon-11 as a blowing agent, and fibers as well as SiO<sub>2</sub> powders. By comparison of reinforcing effect of various contents of SiO<sub>2</sub> powders and fibers on the mechanical properties of the PU composite foam, it showed that the integrated mechanical properties of the hybrid reinforced rigid PU composite foam could get an optimal value. In view of the fact that carbon fibers are expensive, the mechanical properties of the PU composite foam also could be a better value when they were reinforced with 7–8% of glass fibers or nylon fibers.

TABLE II Performance of Three Kinds of Fibers

Technical index	Fiber type		
	PAN-matrix carbon fiber (3 mm)	Nylon-66 fiber (3 mm)	42s glass fiber (3 mm)
Diameter (μm)	6	35	13–15
Density (g/cm <sup>3</sup> )	1.14–1.17	1.74–1.76	1.98–2.01
Tensile strength (MPa)	2000–3000	1500–2000	1000–1500
Tensile module (GPa)	175–215	130–155	65–115

(2) Based on the experimental results of the physical and mechanical properties of the hybrid reinforced rigid PU foam, the content of SiO<sub>2</sub> powders has an obvious effect on water absorption, hardness, tensile strength, and impact strength of the PU composite foam. Water absorption was largest when the content of SiO<sub>2</sub> powders was 10% and water absorption decreased with an increase in the content of SiO<sub>2</sub> powders when the content of SiO<sub>2</sub> powders was more than 10%. Hardness and tensile module increased as the content of SiO<sub>2</sub> powders increased, whereas the change in the trend of the tensile strength was opposite. For high-density hybrid reinforced rigid PU foam, its tensile strength was largest when the content of SiO<sub>2</sub> powders was 20%.

(3) The tensile strength of the hybrid reinforced rigid PU foams got an optimal value when the content of glass fibers was 7–8% and the ratio of length-to-diameter was 140. The reinforcing effect of three different kinds of fibers was compared, and it showed that in such experimental situation the tensile strength of the PU composite foam reinforced with PAN-matrix carbon fiber was the greatest. The PU composite foam reinforced with nylon-66 fiber took second place and the PU composite foam reinforced with glass fiber was the worst, which ranked in the same order as self-tensile strength of fibers.

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