

Elastic and plastic properties of epoxy resin syntactic foams filled with hollow glass microspheres and glass fibers

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ABSTRACT: Representative volume elements of syntactic foams with a random filling of short glass fibers and hollow glass microspheres in epoxy resin were established by a random sequential adsorption method. The fiber volume fraction was set at 4%, and the microsphere volume fraction range was from 5 to 30%. This numerical simulation was studied with ANSYS software. The influence on the elastic and plastic mechanical properties of syntactic foams of the microsphere volume fraction and relative wall thickness were investigated, and the plastic strain evolution process in the composites was analyzed. The results show that the compressive yield limit and Young's modulus values of the syntactic foams decreased with increasing microsphere volume fraction when the microsphere relative wall thickness was 0.02, but these properties were enhanced with increasing microsphere volume fraction when the relative wall thickness exceeded 0.04. The specific strength and tangent modulus values of the composites increased with increasing microsphere volume fraction. In addition, we observed that the yield stress, Young's modulus, and tangent modulus values of the syntactic foams were obviously enhanced by the addition of glass fibers. © 2016 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2016**, *133*, 44188.

KEYWORDS: fibers; foams; mechanical properties

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INTRODUCTION

A composite of hollow-particle-filled polymers is called a *syntactic foam*; it has superior characteristics of low weight, low moisture absorption, high specific strength and stiffness, and good impact behavior. In recent years, syntactic foams have attracted more and more attention from scholars, and they have gradually become a research hotspot.^{1–6} Syntactic foams in which epoxy resins, polyurethane, and several other polymers are used as the matrix and hollow glass microspheres, hollow ceramic microspheres, and even metal hollow spheres are used as fillers have been widely used in the fields of machinery, automobiles, sports equipment, aviation, spaceflight, and deep-sea engineering.^{7–9} These experimental studies found that the properties of syntactic foams were related to the volume fraction, wall thickness, and size of hollow particles. Filling with a higher volume fraction of hollow particles can greatly reduce the density of syntactic foams, and this will be conducive to syntactic foams used in aerospace structures and marine structures. However, the strength and stiffness of composites greatly decreases because of the addition of a higher volume fraction of hollow particles.^{10,11} Much of the published literature has shown that the addition of fibers in syntactic foams is one method for improving the mechanical properties.^{12–22} For example,

Wouterson *et al.*¹⁶ experimentally studied the effects of the fiber volume fraction and length on the mechanical properties and thermal properties. They showed that the tensile strength and flexural strength of the composites were enhanced with increasing fiber volume fraction. Ferreira *et al.*¹⁷ prepared composites with hollow glass microspheres and random fiber filling in epoxy resin, in which the volume fraction of the fibers could reach as high as 1.2%. Their results show that the effects on the bending stiffness and fracture toughness of the composites with the addition of glass fibers was not obvious, but its impact properties showed a larger enhancement. Wang *et al.*²¹ investigated the flexural properties of syntactic foams reinforced by fiberglass mesh and short glass fibers, and their experimental results show that the addition of fibers could improve the bending stiffness.

A few theoretical methods have shown the effects of the particle volume fraction and wall thickness on the modulus of syntactic foams.^{23,24} However, research is still lacking in fiber-reinforced syntactic foams. The parameters of particle wall thickness, size and volume fraction, fiber size, and volume fraction have direct effects on the mechanical properties of the composites, and a variety of conditions is difficult to design by experimental methods. However, adopting the method of numerical

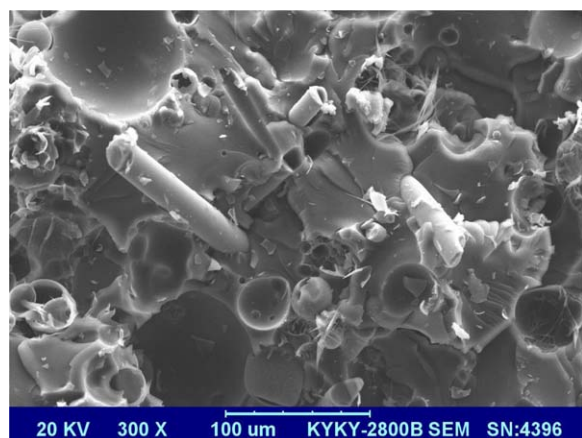


Figure 1. Scanning electron microscopy photograph of the fracture surface of the fiber-reinforced syntactic foams. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

simulation is effective in the consideration of various parameter changes.^{25–29} Nguyen and Gupta²⁵ studied the elastic constant of syntactic foams reinforced by fibers with the method of numerical simulation, and the distribution of particles and fibers were assumed to be uniform in the model. Also, the effects of the hollow microsphere volume fraction and wall thickness on the elastic modulus of the composites and stress distribution in the matrix were investigated. Liang *et al.*²⁹ studied the plastic properties of composites in hollow, particle-filled epoxy resin, and factors such as microsphere volume fraction and thickness ratio were taken into account; this affected the mechanical properties. The representative volume elements (RVEs) are usually used as models, especially three-dimensional, microstructure-based models.^{30–33} Jin *et al.*³⁴ established a three-dimensional cell model of Ti matrix composites reinforced by both short-fiber-like TiB and particle-like TiC and investigated its stress–strain relation though with the method of numerical simulation.

As shown in previous literature, the experimental study of syntactic foams reinforced by fibers have been adopted by many scholars. Adopting the method of numerical simulation, which can be used to consider the various parameter changes, is conducive to the optimization design of materials, but this study of numerical simulation of fibers and hollow, particle-filled polymers together is still lacking. In this study, the RVEs of syntactic foams with short glass fibers and hollow glass microsphere random filling in epoxy resin were established through the random sequential adsorption method. The influence of the elastic and plastic properties of syntactic foams by microsphere volume fraction and relative wall thickness was investigated. The influence on the mechanical properties of syntactic foams of the addition of fibers was discussed.

EXPERIMENTAL

Microstructure of the Syntactic Foams

The syntactic foam of short fiber-reinforced polymers is a kind of effective method for enhancing the mechanical properties of composites. The microstructure of syntactic foams with short glass fibers and hollow glass microsphere random filling in

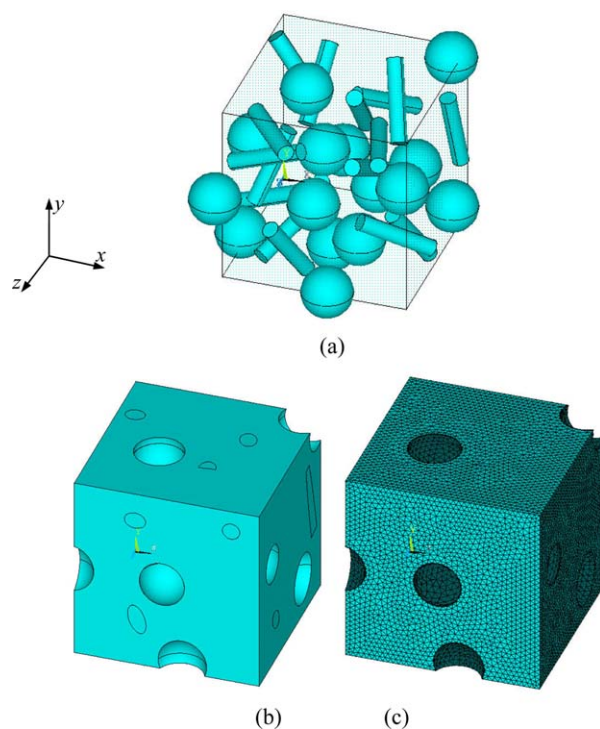


Figure 2. Model and meshed figure. (a) distribution of fillers; (b) RVE model; (c) the meshing. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

epoxy resin is presented in Figure 1. It is a fracture scanning electron microscopy photograph of the flexural specimen. In this figure, glass fibers with an average length of 106 μm and microspheres with an average diameter of 60 μm randomly distribute in the matrix, and a few fibers are exposed. Many literatures cover such composites.^{15–17,21} Although the model of fibers and microsphere uniform distribution is simple, it is not consistent with the actual situation. In particular, the loading direction of the fiber is into a bevel. The situation of fibers and microspheres randomly distributed in the matrix will be closer to the real situation, so it was used to generate numerical simulation models in this study. The microsphere–epoxy and fiber–epoxy were assumed to be perfect adhesives.

Finite Element Models

The RVE models were established in a specific area with ANSYS software, as shown in Figure 2(a). The fibers and microspheres were randomly generated in the RVE by means of a random sequential adsorption method. In this study, to make the model closer to reality, this method was a kind of commonly used method.^{31–36} In this study, the dimensions of matrix were 180

Table I. Material Properties

	Elastic modulus (GPa)	Poisson's ratio	Density (g/cm^3)
Epoxy resin	3	0.35	1.16
Hollow glass microsphere	70	0.20	2.18
Glass fiber	73	0.20	2.18

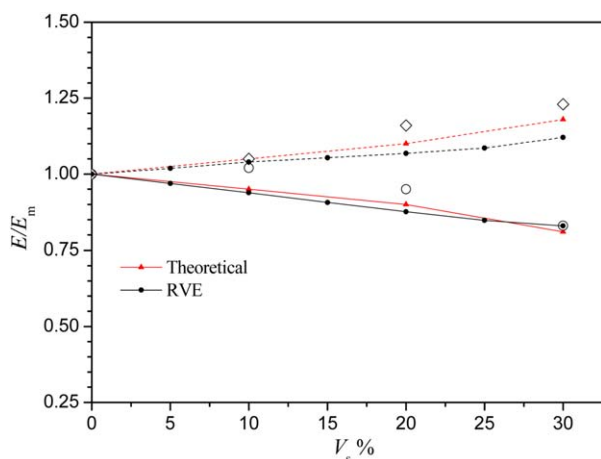


Figure 3. Relative modulus (E/E_m) of the syntactic foams: relative wall thickness (η) = (—) 0.02 and (- - -) 0.06. The open dots and open diamonds indicate the experimental results. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

$\times 180 \times 180 \mu\text{m}^3$. The glass microsphere had a diameter of 50 μm . The fiber had a diameter and length of 16 and 80 μm , respectively. The volume fraction of glass fibers had two degrees of 0 and 4%. The volume fraction of glass microspheres varied from 5 to 30% (5, 10, 15, 20, and 30%). The wall thickness of the glass microspheres had four degrees. Commercial software ANSYS 14.0 was used for the analysis. The material properties were selected on the basis of widely studied epoxy resin syntactic foams in many studies. The elastic constants of the constituents used in the analysis are given in Table I.²⁴ The three-

dimensional SOLID185 element was used to mesh the matrix and glass fibers to calculate the elastoplastic problem. The SHELL181 element was used to mesh the hollow glass microspheres because of the thin shell structure of the microspheres. The resin matrix was assumed to be elastic/perfectly plastic and isotropic; the fiber and microsphere were assumed to be linearly elastic and isotropic. The meshed model is shown in Figure 2(c). The meshing of RVE was denser at regions close to the filler and less dense in the matrix far away from the filler. The 10-node tetrahedral isoparameter element was adopted in this study. The 10% compressive strain was applied on the unit cell surface along the y direction, and the coupling processing of other surfaces ensured that each surface remained planar after the process of loading.

For the boundary conditions of the numerical simulation model of particle-filled composites, some researchers have adopted free boundary conditions, and more researchers have adopted periodic boundary conditions. In these models, the matrix contains numerous glass microspheres and glass fibers. RVE is similar to a cube cut from material, so free boundary conditions were selected to save calculation time. Figure 3 compares the relative modulus of syntactic foams containing hollow glass microspheres obtained from RVE in this study and the theoretical results²⁴ and experimental results from the literature.¹⁵ The open dots show the experimental results of K15-type microspheres, whose diameter was 70 μm and relative wall thickness was about 0.02. The open diamonds show the experimental results of the K46-type microspheres, whose diameter was 40 μm and relative wall thickness was about 0.063. In this study, the relative modulus was the ratio of the moduli of the syntactic

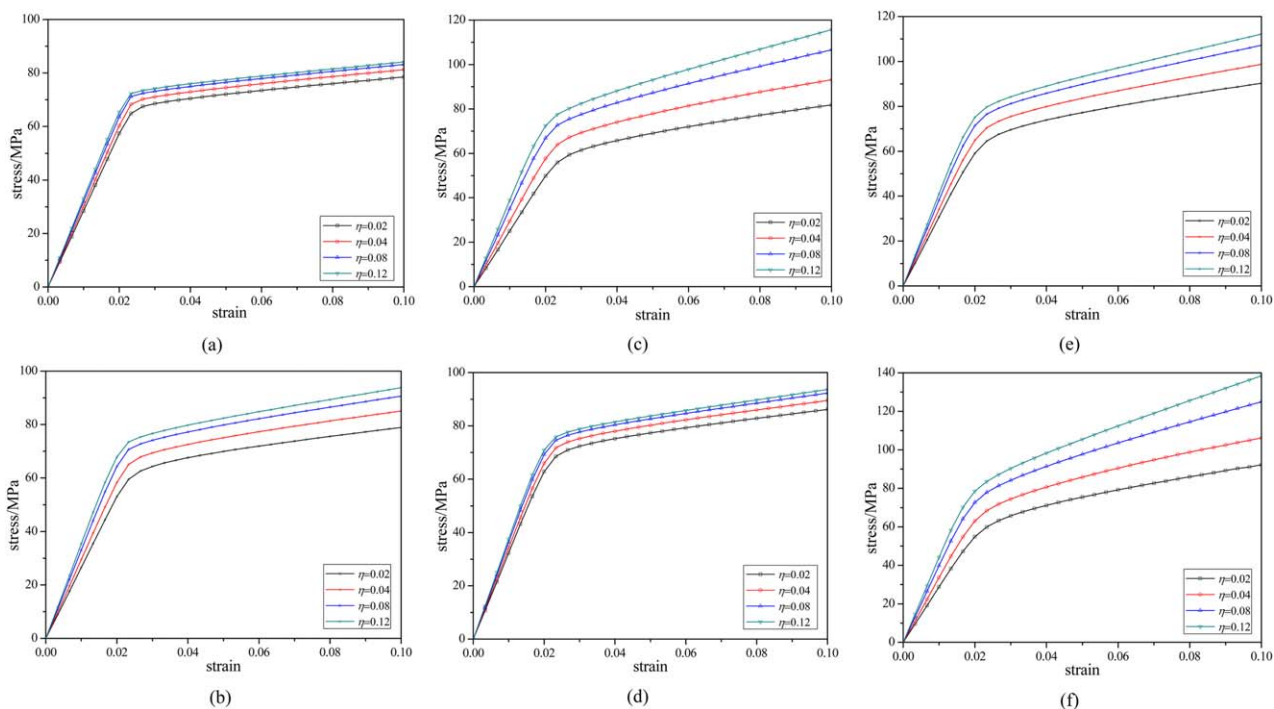


Figure 4. Compressive stress–strain curves for each RVE: (a) $V_s = 10\%$ and $V_f = 0\%$, (b) $V_s = 20\%$ and $V_f = 0\%$, (c) $V_s = 30\%$ and $V_f = 0\%$, (d) $V_s = 10\%$ and $V_f = 4\%$, (e) $V_s = 20\%$ and $V_f = 4\%$, and (f) $V_s = 30\%$ and $V_f = 4\%$. η = relative wall thickness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

foams and the epoxy resin matrix. The relative wall thickness was defined as the ratio of the wall thickness to the radius of the hollow glass microspheres. V_s is the volume fraction of the hollow glass microspheres, and V_f is the volume fraction of the glass fibers. As shown in Figure 3, the theoretical results and RVEs were close, and the change trends were consistent with the experimental results. There was a difference in microsphere diameter compared with the experimental microsphere diameter. The diameter of the microsphere in RVE was constant at 50 μm , and the relative wall thickness was the variable value that accounted for the same scheme. Therefore, at lower relative thickness values, the RVEs used in this article were valid.

RESULTS AND DISCUSSION

Stress–Strain Relations

Figure 4 gives the compression stress–strain curves of the RVE models of the syntactic foams. Figure 4(a–c) shows the curves of syntactic foams without fibers, and the curves of different wall thicknesses when the fiber had a 4% volume fraction are shown in Figure 4(d–f). As shown in the figures, the stress–strain curve of syntactic foam with a thicker microsphere wall thickness was higher than that with a thin microsphere wall thickness. This trend indicated better mechanical properties in the composites. When the microsphere volume fraction was small ($\leq 10\%$), the difference in this curve was not significant because of the small microsphere content [see Figure 4(a,d)]. However, when the microsphere content increased, the effect on the mechanical properties of the composites was gradually enhanced, and the difference between each curve was obvious when the microspheres were at 30% volume fraction [see Figure 4(c,f)]. Therefore, the wall thickness of the microsphere had a great effect on the mechanical properties of the composites when the microsphere volume fraction attained a certain value. Additionally, each curve showed bilinear regularity, and its slope increased with increasing microsphere content [cf. Figure 4(a) and 4(c)]. The material with fibers also presented the same rule [see Figure 4(d–f)]. This suggested that the microsphere volume fraction had a great influence on the mechanical properties of these syntactic foams.

Figure 5 shows the compression stress–strain curves of the RVE models of the syntactic foams at different microsphere volume fractions (V_s) when the wall thickness of the microsphere was constant. As shown in Figure 5(a), the curve crossover occurred at a strain around 6% when the microsphere wall thickness was small (relative wall thickness = 0.02); that is, with increasing microsphere volume fraction, the mechanical properties and yield limit of syntactic foams gradually decreased at small strain. After the plastic stage, the difference between these curves decreased with increasing strain, and curve crossover occurred until the strain was around 6%. Then, as the strain continued to increase, the mechanical properties of the composites with a high microsphere volume fraction were stronger. The reason was that the microsphere wall thickness was smaller, and the use of microspheres to fill the composites reduced the mechanical properties of the composites. The higher the microsphere volume fraction was, the lower the mechanical properties of the composites were. So, the mechanical properties of the composites were lower within a certain range of strain after the elastic and yield stage. There

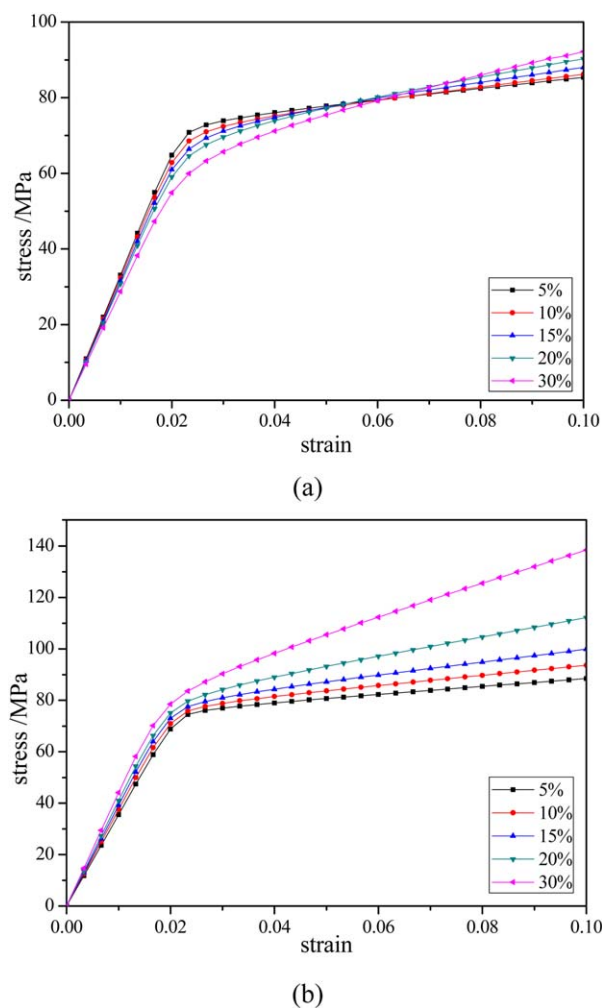


Figure 5. Compressive stress–strain curves for each RVE with different V_s values: (a) $\eta = 0.02$ and $V_f = 4\%$ and (b) $\eta = 0.12$ and $V_f = 4\%$. η = relative wall thickness. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

were many regions where fibers close to the microspheres appeared in the matrix, and the number of such regions increased with increasing the microsphere volume fraction. These regions easily reached the plastic flow state because of stress interactions between the microsphere and fiber when the composite was under a larger strain condition. That was the reason that the mechanical properties of composites with larger microsphere volume fractions were better when the composite was under a larger strain condition. As shown in Figure 5(b), the addition of microspheres enhanced the mechanical properties of the composites with a larger microsphere wall thickness; therefore, the higher the microsphere volume fraction was, the better the mechanical properties of the composites were. Particularly, after the plastic stage, the enhancement of the mechanical properties of the composites was especially obvious because of the interactions of stress distribution between the microspheres and fibers.

Analysis of the Mechanical Properties

Figure 6 shows the curves of the relative modulus of the RVE model of the fiber-reinforced syntactic foams. The relative

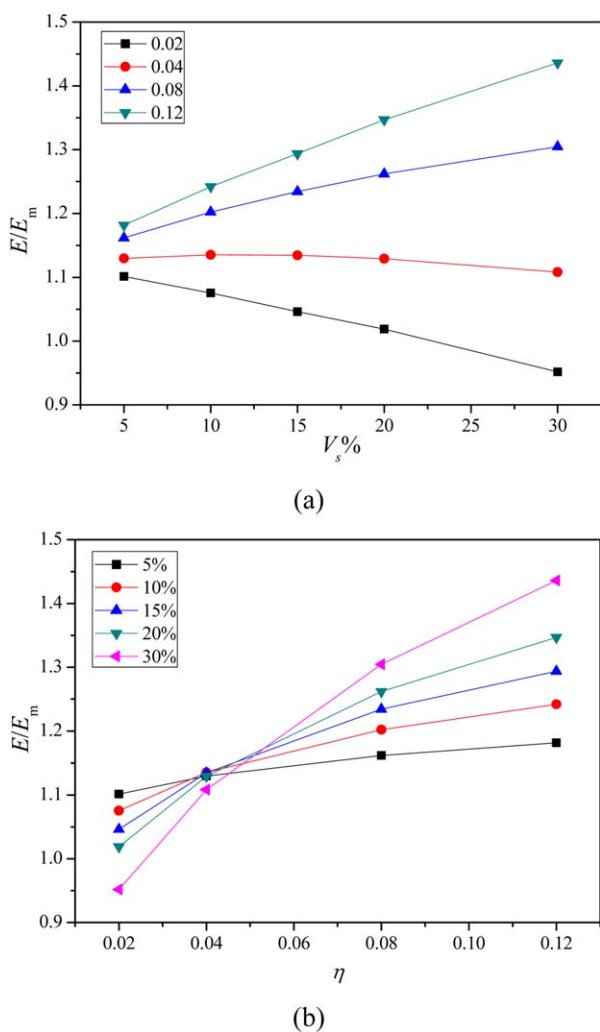


Figure 6. Relative elastic modulus (E/E_m) of the fiber-reinforced syntactic foams. (a) microsphere volume fraction changes; (b) relative wall thickness changes. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

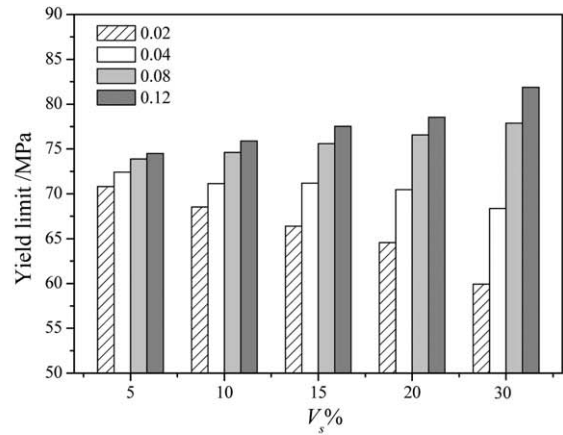
modulus was the ratio of the moduli between the syntactic foams and the epoxy resin matrix. Figure 6(a) gives the curves of the relative Young's modulus change with the microsphere volume fraction. In this figure, the relative Young's modulus of the composites presented a different change rule with a different relative wall thicknesses of the microsphere. When the relative wall thickness was 0.02, the relative Young's modulus decreased with increasing microsphere volume fraction, but it increased with increasing microsphere volume fraction when the relative wall thickness exceeded 0.04. This result shows that the addition of a microsphere with a small relative wall thickness reduced the Young's modulus of the syntactic foams, but the addition of a microsphere with a large relative wall thickness enhanced their Young's moduli. The reason was that the mechanical properties of the microsphere with a small relative wall thickness were not as good as those of the resin matrix with the same shape and size. The critical relative wall thickness was between 0.04 and 0.08. Therefore, the modulus of syntactic foams greatly decreased with the addition of a higher content of microspheres

whose relative wall thickness was less than the critical relative wall thickness. Otherwise, the modulus of the syntactic foams were enhanced. Figure 6(b) gives the relative Young's modulus change curve with the relative wall thickness of the microsphere. As shown in Figure 6(b), the Young's modulus values of the composites increased with increasing relative wall thickness of the microsphere under all kinds of microsphere volume fractions. Obviously, the Young's modulus values of the composites were enhanced with increasing relative wall thickness. The figure also shows that the higher the microsphere volume fraction was, the larger the curve slope was. This rule indicates that the relative wall thickness had a more obvious effect on the modulus values of the composites when the microsphere volume fraction was larger. In addition, when the relative wall thickness of the microspheres was 0.04, the Young's modulus of the composite was smaller with higher microsphere volume fractions. However, the Young's modulus of the composite was larger with a higher microsphere volume fraction when the relative wall thickness of the microspheres exceeded 0.06. This figure shows that the critical relative wall thickness value was between 0.04 and 0.06.

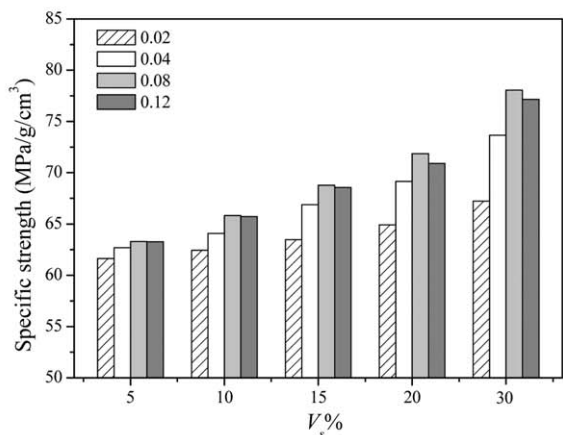
Figure 7 shows that the yield limit and specific strength of the RVE model of fiber-reinforced syntactic foams changed with changing microsphere volume fraction. As shown in Figure 7(a), when the microsphere volume fraction increased, the yield limit of the composites decreased when the wall thickness of the microsphere was smaller, but it increased when the wall thickness of the microsphere was larger. The reason was that the mechanical properties of the microspheres with a small relative wall thickness were not as good as those of the resin matrix with the same shape and size. Therefore, the strength of the syntactic foams was greatly reduced by the addition of a higher content of microspheres whose relative wall thickness was less than the critical relative wall thickness. Otherwise, it was enhanced.

Figure 7(b) gives the relationship change between the specific strength and the microsphere volume fraction. The specific strength was the ratio of the strength to the density. As shown in the figure, whatever the relative wall thickness of microsphere was, the microsphere volume fraction was larger, and the specific strength of the composite was higher. The difference in the specific strength of the syntactic foams with different microsphere relative wall thicknesses also increased with increasing microsphere volume fraction. It is shown in Figure 7(b) that the specific strength value of the composites with a microsphere relative wall thickness of 0.08 was always at the maximum. This indicated that the microsphere relative wall thickness of 0.08 was a good ratio, and the microsphere wall thickness had a great influence on the specific strength of the syntactic foams. Therefore, it was a significant factor in the composite design.

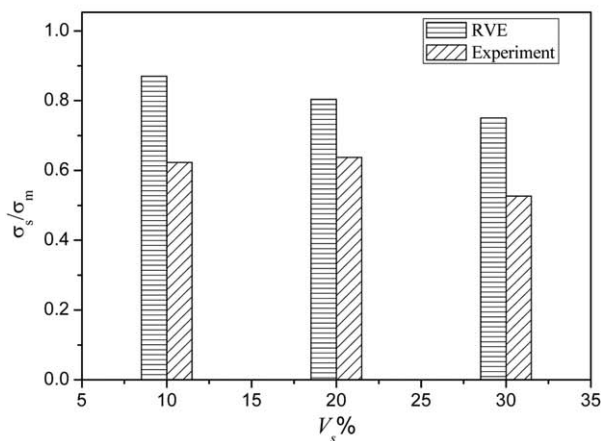
Figure 7(c) shows the comparison of the relative strengths in the experimental results and RVE results without fibers (relative wall thickness = 0.02); the experimental results were taken from the literature.¹⁵ The relative strength was the ratio of the yield limits between the syntactic foams and the epoxy resin matrix. As shown in Figure 7(c), the experimental results were lower



(a)



(b)

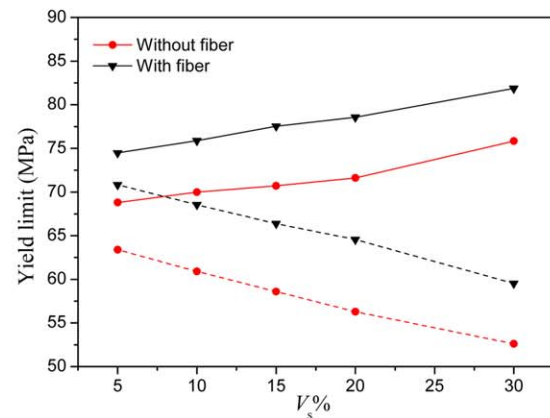


(c)

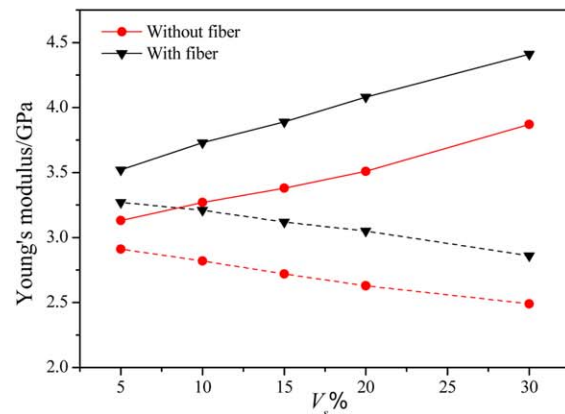
Figure 7. Yield limit and specific strength of the fiber-reinforced syntactic foams: (a) yield limit, (b) specific strength, and (c) comparison of the relative strength (σ_s/σ_m) from the experimental results and the RVE (relative wall thickness = 0.02).

than those of RVE. One of the reasons for this difference was that there were always a lot of defects in the experimental materials, including microspheres clustering, breaking, and forming

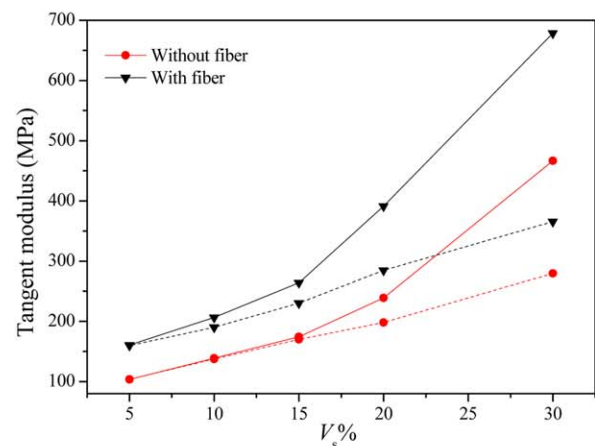
air bubbles, but there were no such defects in the simulation model. These defects had a large effect on the strength of composites, and the effect on the strength was greater than that on



(a)



(b)



(c)

Figure 8. Comparison of the (a) yield limit, (b) Young's modulus, and (c) tangent modulus: relative wall thickness (η) = (---) 0.02 and (—) 0.12. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

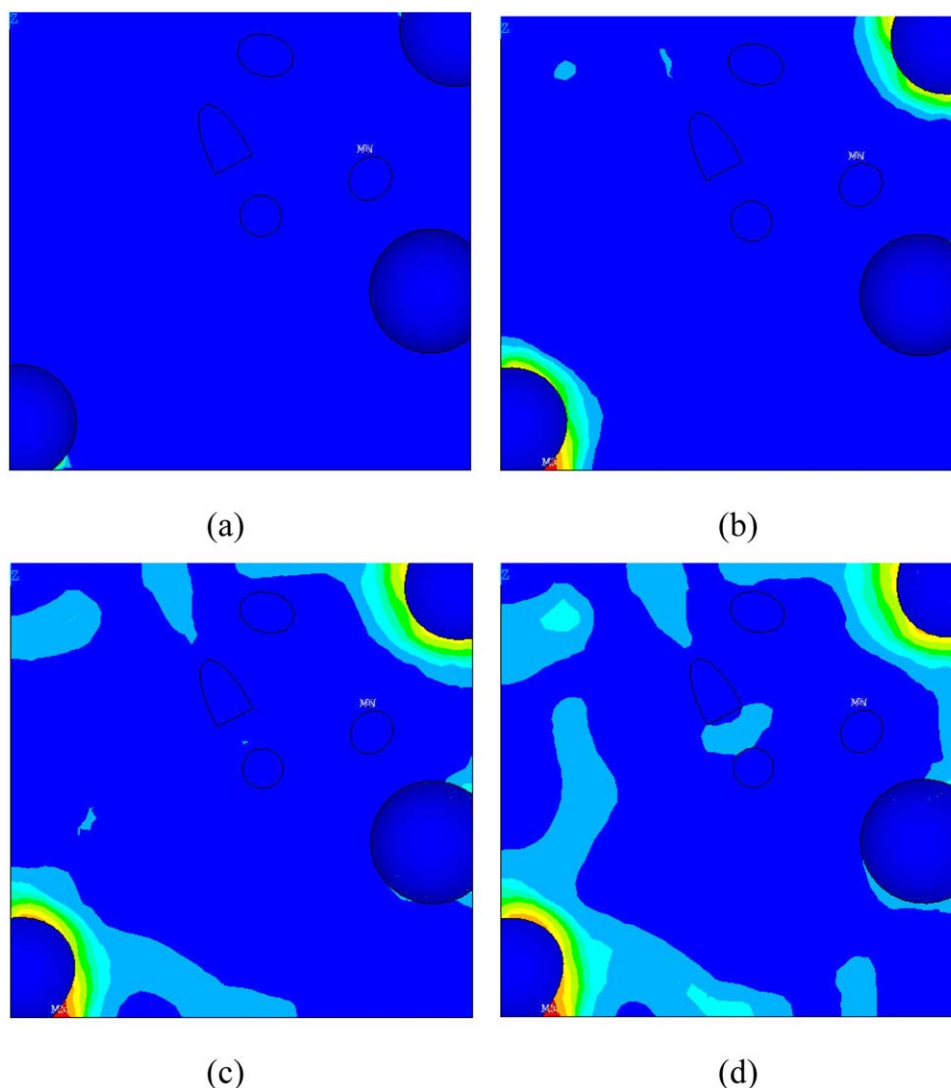


Figure 9. Evolution of the plastic strain during compression (relative wall thickness = 0.02): (a) 1, (b) 2, (c) 4, and (d) 6% strain. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the elastic modulus because these defects took place at crack generation. Therefore, in this model, the influence of the defect will be an important research topic. The other reason may have been the difference in the microsphere diameter compared with that of the experimental microspheres. The diameter of the microsphere in RVE was constant at 50 μm , but the average diameter of the microspheres in the experiment was 70 μm . The microsphere diameter of the syntactic foams also had an effect on the mechanical properties of the syntactic foams.

Figure 8(a) shows a comparison of the yield limit of the composites with fibers and without fibers. The solid line shows a relative wall thickness of the microsphere of 0.12, and the dashed line shows a microsphere relative wall thickness of 0.02. As shown in Figure 8(a), whatever the relative wall thickness and microsphere volume fraction were, the yield limit of the fiber-reinforced syntactic foams was higher than that of the unreinforced syntactic foams. When the microsphere volume fraction was 30%, the yield limit of the fiber-reinforced foams increased to 8.0 and 13.1%, respectively, compared with those

of the unreinforced foams for these two microsphere relative wall thicknesses of 0.12 and 0.02. Figure 8(b) gives the comparison of the Young's modulus. It shows that the Young's modulus values of the fiber-reinforced foams were obviously higher than those of the foams without fiber contents. The Young's modulus values of the fiber-reinforced foams increased 13.9 and 14.8%, respectively, compared with those of the unreinforced foams for the two microsphere relative wall thickness with 30 vol % microspheres. This indicated that the addition of fibers was beneficial for improving the mechanical properties of the composites.

Figure 8(c) presents the contrast curves of the tangent modulus, which was the rate of the compressive stress–strain curve of the plastic stage. The value of the tangent modulus is one of the mechanical properties of composites in the plastic stage. As shown in Figure 8(c), the tangent modulus with the fiber-reinforced syntactic foams was higher than that of the unreinforced syntactic foams. The higher the microsphere volume fraction was, the more obvious the reinforcing effect was. The

tangent modulus values increased by 30.7 and 45.4%, respectively, for the two different microsphere relative wall thickness when the microsphere volume fraction was 30%. We observed that the addition of fibers effectively enhanced the tangent modulus of the microspheres. In addition, whether the composites were with or without fiber contents, the tangent modulus increased with increasing microsphere volume fraction. In particular, for the microsphere relative wall thickness of 0.12, the tangent modulus showed a significant increase when the microsphere volume fraction was 30%. The reason was considered to be the fact that an increase in the microsphere volume fraction put the resin matrix into a more plastic state with a syntactic foams yield.

We also observed that the effect of the microsphere relative wall thickness on the tangent modulus was not obvious when the microsphere volume fraction was smaller [see Figure 8(c)]. The reason was the low content of microspheres. When the microsphere volume fraction increased, the reinforcing effect of the wall thickness on the tangent modulus of the composite significantly increased. In particular, for the fiber-reinforced syntactic foams, the effect was more obvious. For the syntactic foams without fiber contents, the tangent modulus with a relative wall thickness of 0.12 increased by 66.8% at 30 vol % microsphere content compared with that with a relative wall thickness of 0.02. Under the same conditions of wall thickness and microsphere volume fraction, the tangent modulus with the fiber-reinforced syntactic foams increased by 85.7%. We concluded that the addition of fiber had a greater effect on the mechanical properties of the composites at the plastic stage than on those of the composites without fiber contents. The reason was that the addition of fibers changed the stress distribution inside the composites and put more resin matrix into the plastic state when the syntactic foams were yielded.

Figure 9 shows the evolution of equivalent plastic strain on a two-dimensional cross section of the model with a 20% microsphere volume fraction and a relative wall thickness of 0.02 (x - z plane). In the figure, the blue section indicates no plastic strain, and the other colors represent the plastic strain. As shown in Figure 9(a,b), the plastic strain started to develop from the matrix region in contact with the microspheres [see the red section in Figure 9(b)]. The plastic strain in the area where the distance between the microsphere and fiber was closer developed faster [see Figure 9(c,d)]. When the microsphere volume fraction was certain, the addition of fiber contributed to the increase in that area. The matrix material showed a more rapid development to plasticity after the composites reached the plastic stage; this made the mechanical properties of the composites in the plastic stage better. That is, at the same strain condition, the addition of fibers made more matrix material enter the plastic state; this was beneficial to the improvement of the mechanical properties of the syntactic foams. The addition of fibers enhance the mechanical properties of the composites in the plastic stage. These results were consistent with the conclusions obtained from Figures 5 and 8(c).

CONCLUSIONS

RVEs of syntactic foams with short glass fiber and hollow glass microsphere random filling in epoxy resin were established. The

effects of the wall thickness and microsphere volume fraction on the elastic-plastic mechanical properties were investigated. The numerical results show that there was a critical microsphere relative wall thickness with a value between 0.04 and 0.06. The Young's modulus and yield limit of the syntactic foams decreased with increasing microsphere volume fraction when the microsphere relative wall thickness was less than the critical value, but these properties were enhanced with increases in the microsphere volume fraction when the microsphere relative wall thickness was beyond the critical value. However, the specific strength always increased with increasing microsphere volume fraction, and it showed a maximum value when the relative wall thickness was 0.08. The elastic-plastic mechanical properties of the syntactic foams were enhanced by the addition of fibers. The yield stress, Young's modulus values, and tangent modulus values of the fiber-reinforced syntactic foams increased by 13.1, 14.8, and 30.8%, respectively, compared with those of the unreinforced foams, when the microsphere volume fraction was 30% and the relative wall thickness was 0.02. In addition, the addition of fibers had a greater reinforcement effect on the tangent modulus of the composites than on that of the unreinforced composites when the microsphere relative wall thickness changed. The internal plastic strain in the composites started to develop from the resin in contact with microsphere, and the matrix material saw more rapid development to plasticity in the area where the microsphere was close to the fiber.

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