Research Article

Investigation on the Mechanical and Thermal Insulation Properties of Hollow Microspheres/Phenolic Syntactic Foams

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Hollow microspheres are widely used in syntactic foam as a lightweight filling material. Hollow glass microspheres (HGM) and hollow phenolic microspheres (HPM) were added to the phenolic resin to prepare phenolic syntactic foams. Then the mechanical properties, thermal insulation properties, and thermal property stability of them were studied. The mechanical test result shows that the flexural strength of phenolic syntactic foam gradually decreases with the increasing volume fraction of microspheres at room temperature. When the volume fraction of HGM is 20%, the specific strength of phenolic syntactic foam can rise to 0.0334 Nm/kg. HGM reduces the density of the phenolic syntactic foam and remains at high strength. In addition, the thermal conductivity of phenolic syntactic foam decreases with the increasing volume fraction of the hollow microspheres, indicating that the microspheres can effectively improve the thermal insulation performance of the phenolic syntactic foam. Meanwhile, the thermal conductivity of phenolic syntactic foam also increases as the heat treatment temperature rises. In addition, the thermal insulation performance of phenolic syntactic foam containing HGM is better than that containing HPM. Thermal analysis experiments show that the thermal weight loss rate becomes slower as the content of HGM increases. Therefore, HGM improves the thermal stability of the containing phenolic syntactic foam. However, the HPM reduces the thermal decomposition temperature and the thermal stability of the phenolic syntactic foam. This work provides the technical basis for applying phenolic syntactic foam as a heat insulation material.

1. Introduction

With the further development of aerospace technology, the requirements of spacecraft for thermal insulation technology have become more stringent. For example, there is a thermal protection system on the spacecraft's surface. It usually consists of a thermal protection layer and a thermal insulation layer. The thermal protection layer dissipates heat through the mass ejection effect, and the heat insulation layer further blocks the remaining heat.

Common aerospace thermal insulation materials include aerogels [1–3], inorganic fiber thermal insulation materials [4–6], and syntactic foam [7–13]. Syntactic foam is a kind of polymer with an internal pore structure that can be well bonded with the ablation-resistant polymer to play a role in comprehensive thermal protection. Ordinary heat-preserving and heat-insulating syntactic foams generally use fly ash as a filling material, but their low heat resistance limits their use at high temperatures [14, 15]. The hollow microspheres realize the lightweight of syntactic foam and ensure that it has specific mechanical properties [16–18]. However, there are still few studies on the mechanical and thermal insulation properties of hollow microspheres and phenolic resin composite foams.

In this work, we chose phenolic resin as the matrix material by mixing it with hollow glass microspheres (HGM) and hollow phenolic microspheres (HPM). Then we investigated the effects of the hollow microspheres on the syntactic foam's mechanical and thermal insulation properties.

2. Experimental

2.1. Raw Materials. The main raw materials and reagents used in this experiment are listed in Table 1.

2.2. Preparation of Hollow Microsphere/phenolic Syntactic Foam. This experiment used a casting process to prepare hollow microsphere/phenolic syntactic foam. The preparation process is as follows: first, 100 mL of the phenolic resin solution is stirred at a constant temperature in a water bath at 40°C until the alcohol is fully volatilized. After stirring for two hours, a certain amount of HGM or HPM was weighed and added to the stirred phenolic resin in batches to continue stirring. After thoroughly stirring, a certain amount of curing agent (toluene-4-sulfonic acid) was added to the phenolic resin. After stirring evenly, the resin slurry was poured into the mold that had been coated with the release agent. Then the mold was placed in an electric heating oven for curing. They were cured at 40°C for 10 hours, 60°C for 8 hours, 80°C for 6 hours, 100°C for 4 hours, and 120°C for 2 hours. Finally, the mold is taken out and demolded when the temperature drops to room temperature.

2.3. Heat Treatment of Hollow Microsphere/phenolic Syntactic Foam. The prepared phenolic syntactic foam was placed into a muffle furnace at a temperature of 300°C for about 15 minutes. Then it was taken out of the furnace and cooled for later use. Then the temperature of the muffle furnace was raised to 600°C. The sample was placed in the furnace for about 15 minutes and cooled for later use. Finally, the temperature of the muffle furnace was raised to 900°C. The sample was kept warm for about 5–10 minutes and then cooled for later use.

2.4. Characterization Tests

2.4.1. Density. According to the standard of fiber-reinforced plastics test method GB/T 1463–2005, the density of phenolic resin, phenolic syntactic foam containing 10vol%–50vol% HGM and phenolic syntactic foam containing 10vol%–30vol% HPM were measured.

2.4.2. Micromorphology. A JSM-7500F field emission scanning electron microscope (SEM) from Japan JEOL was used to observe the micromorphology of the HGM and HPM.

The micromorphology of the curved fracture surface of the HGM/phenolic syntactic foam with a volume fraction of 10%–50% and the HPM/phenolic syntactic foam with a volume fraction of 10%–30% was also observed and analyzed by SEM.

2.4.3. Mechanical Performance Experiment. A universal testing machine (RGM4100) was used to detect the bending strength of pure phenolic resin, phenolic syntactic foam with a volume fraction of 10vol%–50vol% HGM, and phenolic syntactic foam with a volume fraction of 10vol%–30vol% HPM. According to the test methods for properties of resin casting bodies (GB/T 2567–2008), an 80 mm × 15 mm × 4 mm test sample was cast in the mold. During the experiment, the loading rate was 2 mm/min and the span was 64 mm.

The specific steps are as follows: the test sample was placed in the middle of the test bench. The universal testing machine continuously applied a bending moment to the test sample during the experiment until the sample broke. The maximum bending load that the sample bears when it is broken is the bending strength of the sample.

2.4.4. Thermal Conductivity. In this experiment, the LFA457 laser thermal conductivity meter (NETZSCH instrument company, Germany) was used to measure the thermal conductivity of the HGM/phenolic syntactic foam. The test temperature range is from room temperature to 1000°C.

2.4.5. Thermal Stability. The thermal stability of HGM/phenolic syntactic foam and HPM/phenolic syntactic foam was tested using the STA449 C TG-DSC integrated thermal analysis mass spectrometer from NETZSCH in Germany. TG-DSC in N_2 atmosphere at a heating rate of 5°C/min from room temperature to 900 °C to explore phenolic syntactic foam's thermal weight loss behavior in different temperature ranges.

3. Results and Discussion

3.1. Microscopic Morphology Analysis of Hollow Microspheres. Figure 1 shows the microstructure of HGM at different magnifications. The HGM is spherical in a regular shape in the scanning electron micrograph. The size of the HGM is uniform, and the HGM surface has more impurities. Figure 2 shows the microstructure of HGM at different magnifications. In the scanning electron micrograph, the HPM is irregularly spherical in size. The size of the microspheres is uneven, and the HPM surface is relatively smooth with fewer impurities.

3.2. Density Analysis of Hollow Microsphere/phenolic Syntactic Foam. The density of glass microspheres/phenolic syntactic foam is shown in Figure 3. It can be observed that the density of the syntactic foam gradually decreases as the volume of HGM increases. The density of pure phenolic resin is 1.199 g/cm^3, and the density of the phenolic syntactic foam is reduced to 0.901 g/cm^3 when the volume of the HGM increases to 50%. This is because the density of HGM is lower than that of phenolic resin. As the content of HGM increases, the quality of the HGM/syntactic foam decreases. As a result, its density also decreases.

The density of HPM/phenolic syntactic foam is shown in Figure 4. It can be observed that the density of the phenolic syntactic foam gradually decreases as the volume of HPM increases. When the volume of HPM increase to 30%, the density of the phenolic syntactic foam decreases to 0.712 g/cm^3. Since the density of HPM is less than that of phenolic resin, the increase in the content of phenolic microspheres reduces the

TABLE 1: The main raw materials and reagents of the experiment.

| Raw material name	Manufacturer	Product information
Phenolic resin	Bengbu Tianyu high-temperature resistant resin co., ltd.	Viscosity: 2000–3000mpa· s
HGM(S60HS)	American 3M company	Density: 0.6 g/cm^3
HPM(BJO-0930)	Uniwell chemical co., ltd.	Average bulk density: 0.104 g/cc
GZ-I of high vacuum insulating silicone grease	Tongxin chemical plant, Caidian district, Wuhan city	Release agent
Toluene-4-sulfonic acid, monohydrate	Sinopharm chemical reagent co., ltd.	Analytically pure
Absolute ethanol	Sinopharm chemical reagent co., ltd.	Analytically pure
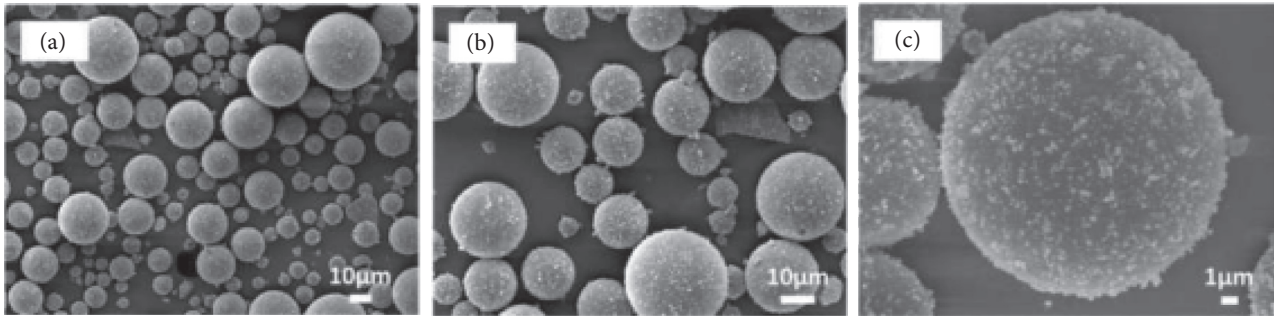
FIGURE 1: (a) SEM image of hollow glass microspheres under 500 times magnification; (b) SEM image of hollow glass microspheres under 1000 times magnification; (c) SEM image of hollow glass microspheres under 4000 times magnification.
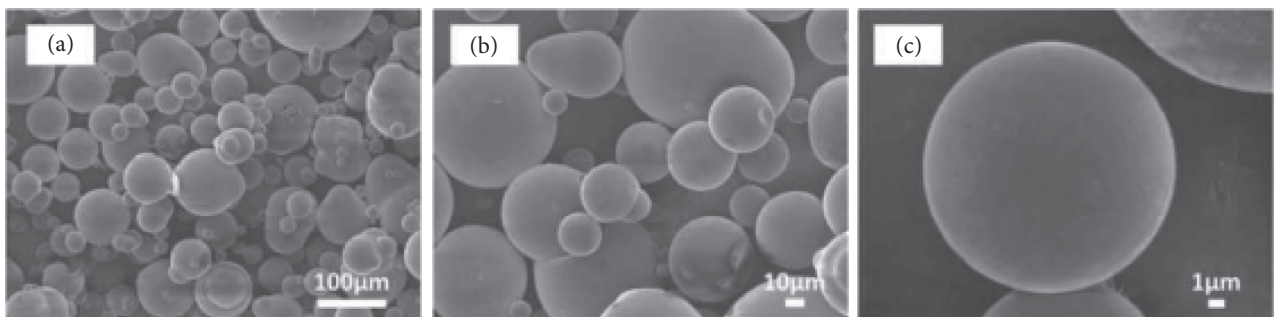

FIGURE 2: (a) SEM image of hollow phenolic microspheres under 200 times magnification; (b) SEM image of hollow phenolic microspheres under 1000 times magnification; (c) SEM image of hollow phenolic microspheres under 4000 times magnification.
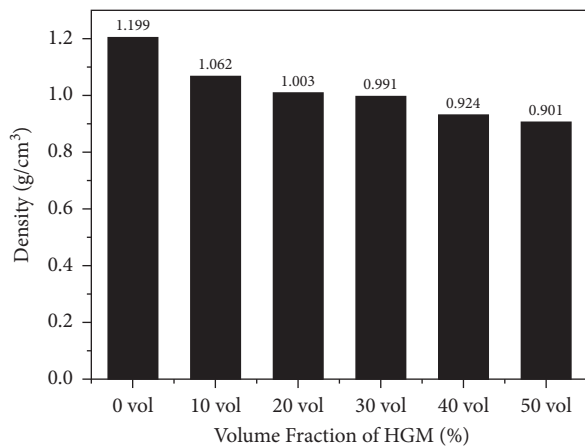

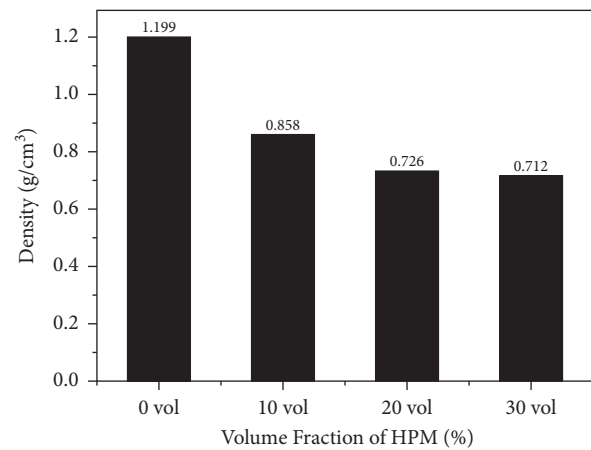FIGURE 3: The density of HGM/phenolic syntactic foam.

FIGURE 4: The density of HPM/phenolic syntactic foam.

density of the phenolic syntactic foam. Comparing Figures 3 and 4, it can be seen that the HPM/phenolic syntactic foam has a lower density than the HGM/phenolic syntactic foam. This is because HPM has a lower density than HGM.

3.3. Mechanical Properties of Syntactic Foam at Different Volume Fractions. The flexural strength and specific strength of the HGM/phenolic syntactic foam are shown in Figure 5. It can be seen that the bending strength of the

phenolic syntactic foam gradually decreases as the volume of HGM increases. The bending strength of pure phenolic resin is 37.59 MPa. When the volume fraction of the HGM increases to 50%, the flexural strength of the phenolic syntactic foam is reduced to 12.31 MPa. This is because the void proportion of phenolic resin increases with the increasing volume of the HGM. The void in the phenolic resin reduces the bending strength of the phenolic syntactic foam.

In addition, the specific strength, as an index that comprehensively describes the strength and density. It can be seen that the specific strength of the HGM/phenolic syntactic foam first increases and then decreases as the content of HGM increases. When the volume of HGM reaches 20%, the peak specific strength of HGM/phenolic syntactic foam is 0.0334 Nm/kg. This displays that HGM increases the specific strength of the phenolic syntactic foam.

The flexural strength and specific strength of the HPM/phenolic syntactic foam are shown in Figure 6. It can be seen that the flexural strength of the syntactic foam gradually decreases as the volume of HPM increases. The bending strength of pure phenolic resin is 37.59 MPa. When the volume fraction of HPM is 30%, the flexural strength of the phenolic syntactic foam reduces to 14.23 MPa. This is because the proportion of phenolic resin decreases as the content of HPM increases.

In addition, the specific strength of the phenolic syntactic foam gradually decreases as the volume of HPM increases. The specific strength of the pure phenolic resin is 0.0314 Nm/kg. When the volume fraction of HPM reaches 10%, the specific strength of the phenolic syntactic foam is 0.0313 Nm/kg. This is very close to the specific strength of pure phenolic resin. Thus, the phenolic syntactic foam with 10 vol% HPM guarantees its bending strength and meets the lightness requirement.

3.4. Mechanical Properties of Syntactic Foam after Heat Treatment at Different Temperatures.

Figure 7 shows the bending strength of HGM/phenolic syntactic foam at room temperature, 300°C, 600°C, and 900°C, respectively. It can be seen that the flexural strength of the phenolic syntactic foam at room temperature is greater than that of the phenolic syntactic foam at different heat treatment temperatures. As the volume fraction of HGM increases, the flexural strength of the syntactic foam gradually decreases. However, the flexural strength of the composite foam is significantly reduced under elevated temperature conditions. In addition, the flexural strength of the syntactic foam increases slightly as the volume fraction of HGM increases. This is because the pyrolysis degree of the phenolic resin becomes more significant as the heat treatment temperature increases. After the released small molecules escaped, many pores were left in the phenolic resin. This phenomenon is more evident when the volume fractions of microspheres are small. On the other hand, the HGM, as the inorganic reinforcing phase, has better thermal stability. It strengthens the phenolic resin after high-temperature pyrolysis and reduces the pores that cause the sample to break. Therefore, the HGM can improve the bending strength of the phenolic syntactic foam after high-temperature heat treatment.
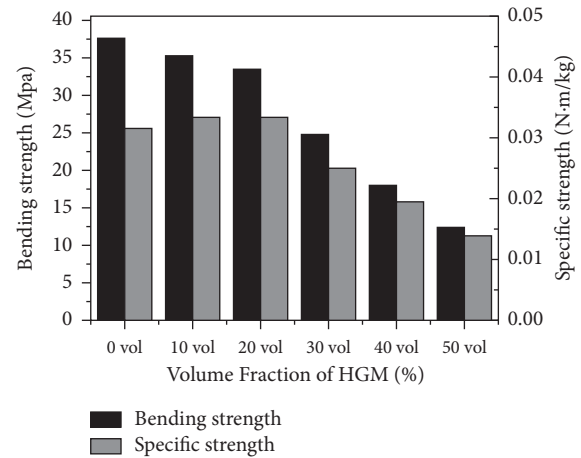

Figure 5: Flexural strength and specific strength of HGM/phenolic syntactic foam.
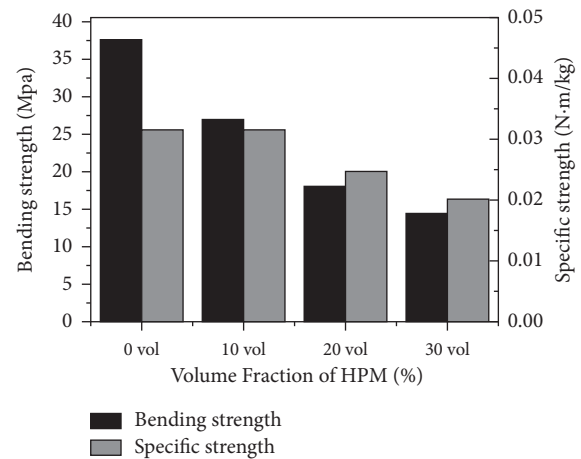

Figure 6: Flexural strength and specific strength of HPM/phenolic syntactic foam.

The flexural strength of phenolic syntactic foam at room temperature, 300°C, 600°C, and 900°C is shown in Figure 8. It can be seen that the flexural strength of the phenolic syntactic foam at room temperature is greater than that at other temperatures. As the volume fraction of HPM increases, the strength of the phenolic syntactic foam gradually decreases at room temperature. This is because more stress is concentrated in the microspheres and resin matrix with the increase of HPM content. In addition, it triggers the premature rupture of large-sized HPM and microcrack formation in the phenolic matrix. When the temperature increases, the strength of the phenolic syntactic foam reduces. After high-temperature treatment, the strength of HPM/phenolic syntactic foam is reduced more significantly than that of HGM/phenolic syntactic foam. This is because high-temperature pyrolysis occurs both in the phenolic matrix and in the phenolic microspheres. Small molecules of the pyrolysis product create many pores in the phenolic resin matrix. The microcracks connect adjacent pores to form large cracks during the propagation process, making the sample easy to fracture and fail.
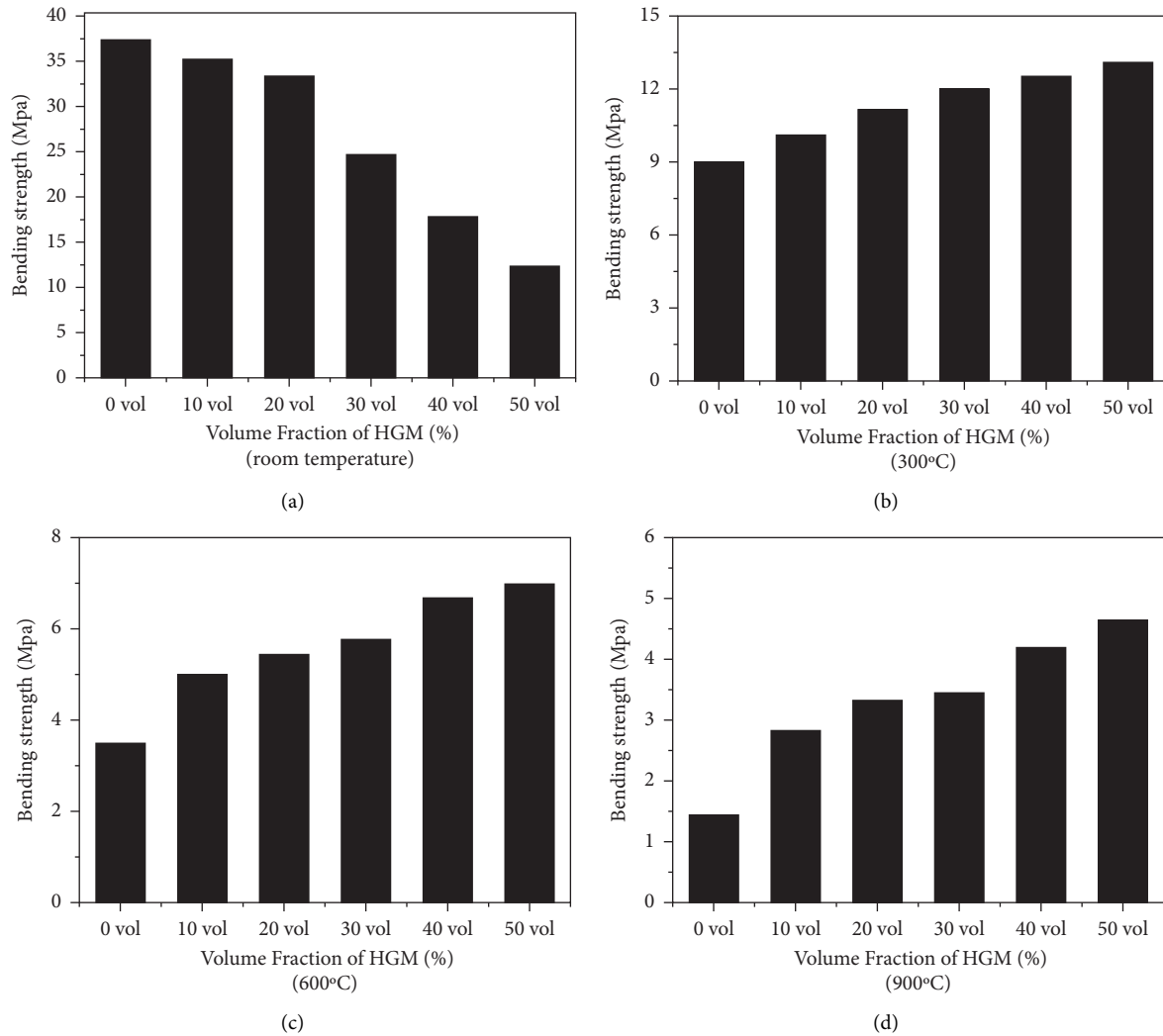
(a)

(b)

(c)

(d)

FIGURE 7: Flexural strength of HGM/microsphere syntactic foam at different temperatures.

3.5. Mechanical Properties of Phenolic Syntactic Foams. The bending strengths of HGM/phenolic syntactic foam and HPM/phenolic syntactic foam are shown in Figure 9. It can be seen that the bending strength of the HGM/phenolic syntactic foam is higher than that of the HPM/phenolic syntactic foam containing the same volume fraction of the microsphere. On the one hand, because the compressive strength of HGM is greater than that of HPM, the HGM/phenolic syntactic foam exhibits better mechanical properties at the same volume fraction of microspheres. On the other hand, the inner cavity of the phenolic microspheres encloses more air due to their larger particle size. This result indicates that the mechanical properties of HPM/phenolic syntactic foam are inferior to those of HGM/phenolic syntactic foam.

The specific strengths of HGM/phenolic syntactic foam and HPM/phenolic syntactic foam are shown in Figure 10. It can be seen that the specific strength of the HGM/phenolic syntactic foam is greater than that of the HPM/phenolic syntactic foam at the same volume fraction of the microsphere. This also indicates that the HGM/phenolic syntactic foam has better mechanical properties than the HPM/phenolic syntactic foam.

The micromorphologies of an HGM and HPM in phenolic syntactic foam are shown in Figure 11. It can be observed that there is a clear gap at the interface between an HGM and the phenolic resin in Figure 11(a). These gaps are also the reason why the mechanical properties of the HGM/phenolic syntactic foam decrease. Figure 11(b) shows that there is no gap at the interface between an HPM and the phenolic resin, and the interface is tightly bonded. It can also be seen from Figure 11 that the particle size of HGM is larger than that of HPM. When the phenolic syntactic foam is subjected to an external load, HPM is more prone to rupture than HGM. This is the main reason why the mechanical properties of HPM/phenolic syntactic foam are lower than those of HGM/phenolic syntactic foam at the same volume fraction of microspheres.

3.6. Thermal Conductivity of the Phenolic Syntactic Foam. The thermal conductivity of HGM/phenolic syntactic foam and HPM/phenolic syntactic foam at different heat treatment temperatures is shown in Figure 12. It can be observed that the thermal conductivity of the phenolic syntactic foam
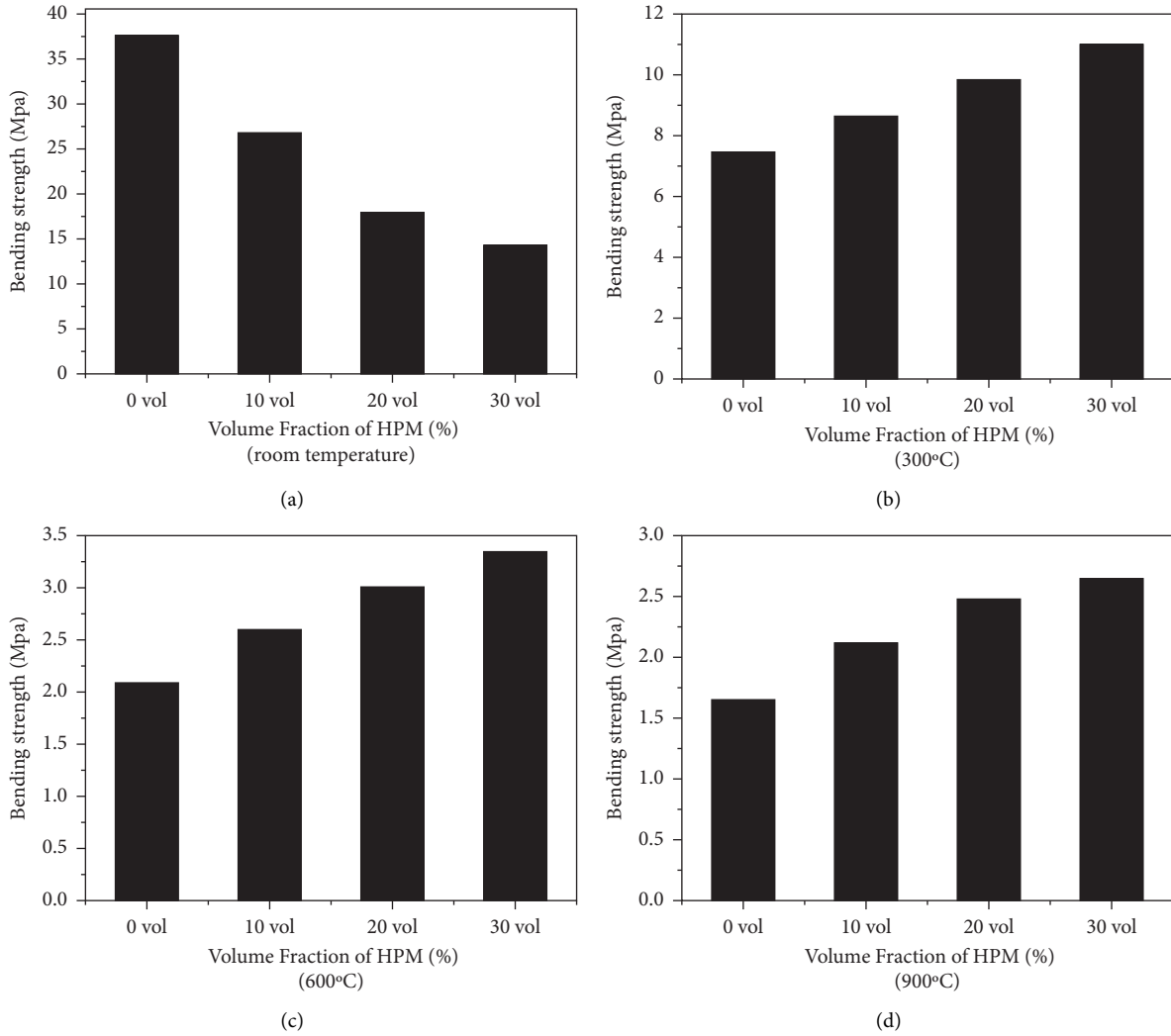
(a)

(b)

(c)

(d)

FIGURE 8: Flexural strength of phenolic syntactic foam at different temperatures.
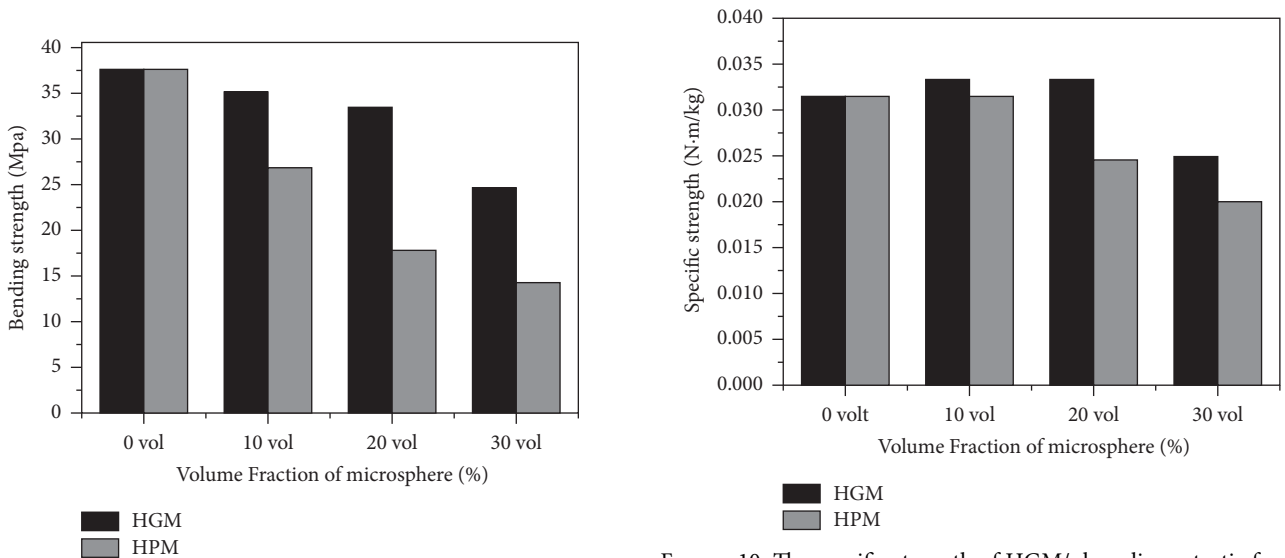

FIGURE 9: Bending strength of phenolic syntactic foams.

FIGURE 10: The specific strength of HGM/phenolic syntactic foam and HPM/phenolic syntactic foam.

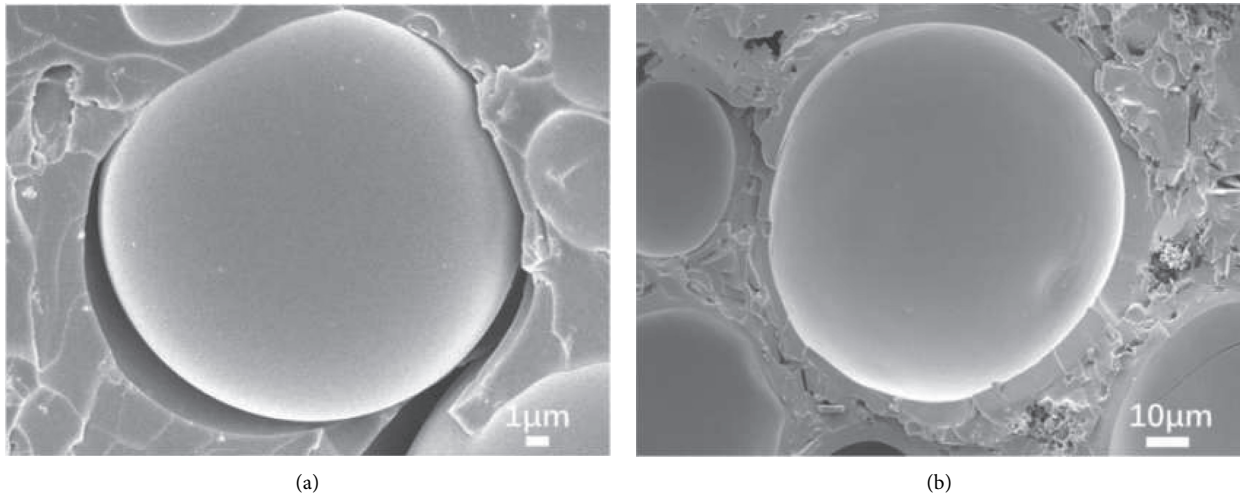(a)   (b)

FIGURE 11: (a) SEM photograph of a single glass microsphere in HGM/phenolic syntactic foam and (b) SEM photo of a single phenolic microsphere in HPM/phenolic syntactic foam.

(a)   (b)
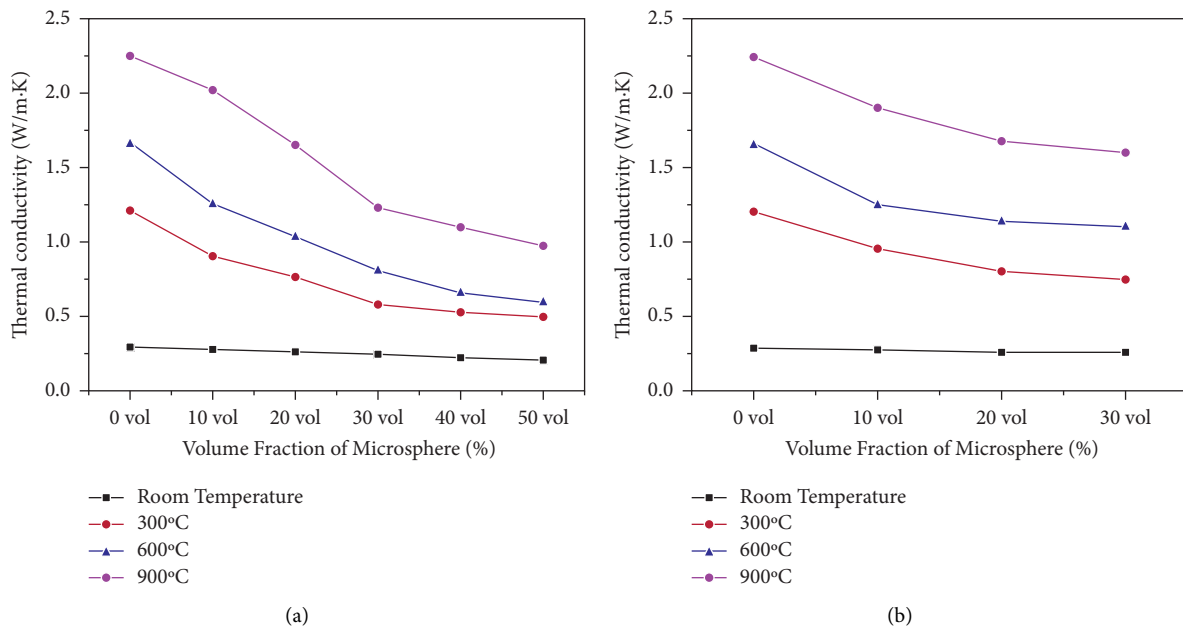
FIGURE 12: (a) The thermal conductivity of HGM/phenolic syntactic foam and (b) The thermal conductivity of HPM/phenolic syntactic foam.

decreases as the volume fraction of the microsphere increases.

It indicates that the microspheres can improve the thermal insulation of the phenolic syntactic foam. On the other hand, the thermal conductivity of phenolic syntactic foam gradually increases as the heat treatment temperature increases. It shows that the thermal insulation performance of the phenolic syntactic foam reduces under high-temperature conditions. In addition, it can be observed that the thermal conductivity of HGM/phenolic syntactic foam is lower than that of HPM/phenolic syntactic foam with the same volume fraction of microspheres.

3.7. Thermal Stability of Phenolic Syntactic Foam.

The TG curves of HGM/phenolic syntactic foams are shown in Figure 13. The thermal weight loss rates of HGM/phenolic syntactic foams at different temperatures are shown in Table 2. From Figure 13 and Table 2, three different stages of thermal weight loss can be observed. In the first stage, when the temperature is between room temperature and 300°C, the weight loss of the syntactic foam is 10.71–18.18%. In the second stage, when the temperature is between 300 and 600°C, the weight loss of the HGM/phenolic syntactic foam is from 23.96% to 38.21%. This stage is the main stage of the thermal decomposition of the syntactic foam. The remaining
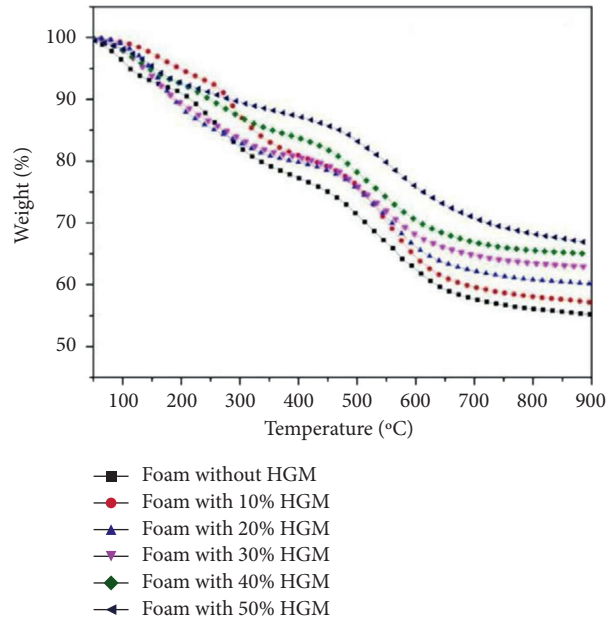
FIGURE 13: TG curves of HGM/phenolic syntactic foams.

TABLE 2: The thermal weight loss rates of HGM/phenolic syntactic foams at different temperatures.

Thermal weight loss rate(%) (°C)	0vol% (%)	10vol% (%)	20vol% (%)	30vol% (%)	40vol% (%)	50vol% (%)
300	18.18	13.06	17.20	16.86	13.23	10.71
600	38.21	36.21	34.44	31.80	29.30	23.96
900	44.84	42.84	39.85	37.40	34.92	33.08

mass of the system is significantly reduced, and the weight loss rate increases. In the third stage, when the temperature is between 600 and 900°C, the weight loss of the HGM/ phenolic syntactic foam is from 33.08% to 44.84%. The thermal decomposition of the resin matrix is basically completed, and the weight loss rate is reduced. In addition, the thermal stability of the HGM/phenolic syntactic foam is improved as the content of HGM increases. This is because the unstable oxygen-containing molecular chain in the phenolic resin has wholly decomposed in the range of 450 to 700°C. The thermal decomposition of the main chain of the aromatic ring structure mainly occurs in the phenolic resin matrix at this temperature. The existence of HGM effectively hindered the thermal decomposition degree of the system. As a result, the thermal weight loss rate decreases as the volume fraction of HGM increases.

The TG curve of HPM/phenolic syntactic foam is shown in Figure 14. The thermal weight loss rates of HPM/phenolic syntactic foams at different temperatures are shown in Table 3. From Figure 14 and Table 3, three different stages of thermal weight loss can also be observed. In the first stage, when the temperature is between room temperature and 300°C, the weight loss of the syntactic foam is 20.16–21.61%. In the second stage, when the temperature is between 300 and 600 °C, the weight loss of the HPM/phenolic syntactic foam is 38.44–40.03%. This stage is the main stage of the thermal decomposition of syntactic foam. The remaining mass of the system is greatly reduced, and the rate of weight loss increases. In the third stage, when the temperature is
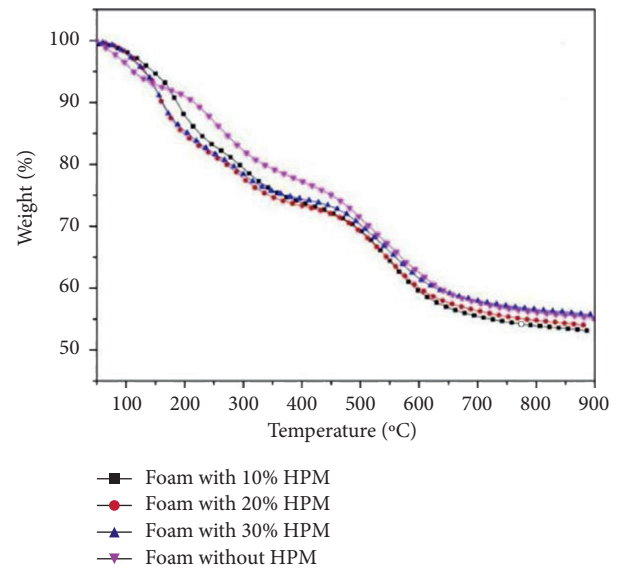

FIGURE 14: TG curves of HPM/phenolic syntactic foams.

between 600 and 900 °C, the weight loss of the HPM/phenolic syntactic foam is 44.36–46.53%. The thermal decomposition of the resin matrix is basically completed, and the weight loss rate is reduced.

In addition, it can be seen that 10 vol% of HPM does not effectively improve the thermal stability of the phenolic syntactic foam in Figure 14. There is no gap at the interface between an HPM and the phenolic resin to delay the heat

TABLE 3: Thermal weight loss rate of HPM/phenolic syntactic foam at different temperatures.

Thermal weight loss rate (%) (°C)	10vol% (%)	20vol% (%)	30vol% (%)
300	20.16	22.26	21.61
600	40.03	40.00	38.44
900	46.53	46.16	44.36

transfer in phenolic syntactic foam. Therefore, adding HPM to the phenolic resin decreases the thermal stability of the phenolic syntactic foam.

4. Conclusion

(1) The addition of HGM and HPM reduces the density of the phenolic syntactic foam. As the volume fraction of microspheres increases, the density of the phenolic syntactic foam gradually decreases. In addition, the HPM/phenolic syntactic foam has a lower density than the HGM/phenolic syntactic foam. Compared to HGM, HPM reduces the density of syntactic foam even more.

(2) Mechanical performance experiments show that the strength of phenolic syntactic foam decreases gradually with the increase in the volume fraction of microspheres at room temperature. At high temperatures, the bending strength of phenolic syntactic foam increases slightly with the rise of the volume fraction of microspheres. In addition, the bending strength of the HGM/phenolic syntactic foam is higher than that of the HPM/phenolic syntactic foam. The specific strength of the HGM/phenolic syntactic foam containing 20 vol% of HGM increases more obviously than other HGM/phenolic syntactic foams. HGM/phenolic composite foam has the advantages of being lightweight and having good mechanical properties.

(3) Thermal insulation experiments display that the thermal conductivity of phenolic syntactic foam decreases as the content of microspheres increases. As the heat treatment temperature rises, the thermal conductivity of the composite foam also increases. In addition, the thermal conductivity results confirm that HGM can improve the thermal insulation performance of phenolic syntactic foam more than HPM.

(4) Thermal analysis experiments prove that the thermal weight loss rate becomes smaller as the content of HGM increases. HGM improves the thermal stability of the phenolic syntactic foam. With the rise of the content of HGM, the thermal stability of the phenolic syntactic foam gradually increases. However, HPM does not enhance the thermal stability of the phenolic syntactic foam.

Data Availability

The data used to support the findings of this study are included in the article. Further data or information are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest with this study.

Acknowledgments

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