Fabrication of Al/SiC composite foams with TiH₂ foaming agent by laser melting deposition

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Abstract

In this work, SiC reinforced aluminum composite foams were successfully fabricated by laser melting deposition (LMD). The microstructure, energy absorption capacity, and compressive properties of Al/SiC composite foams were investigated systematically, and the results indicated that with increasing the content of SiC nanoparticles, the porosity and average pore size of aluminum foams decreased. Compared with the pure aluminum foams, the compressive strength and energy absorption of the Al/SiC composite foams increased by 43.8% and 16.0%, from 28.5 MPa and 55.5 × 10⁶ J m⁻³ to 41.0 MPa and 64.4 × 10⁶ J m⁻³, respectively. The research indicates that SiC nanoparticles are effective additives to homogenize the pore size and enhance the mechanical performance of aluminum foams by the LMD method.

1. Introduction

Due to the exceptional performance on energy absorption, sound insulation, and electromagnetic shielding, aluminum foams (Al foams) are widely used as structural and functional materials in aerospace, automobiles, and transportation fields [1–4]. Various methods, including melt foaming [5–7] and powder metallurgical [4, 8–10], have been developed to manufacture Al foams with different porosity. However, Al foams fabricated by these traditional methods have low flexibility for adjusting pores’ shape, size, and distribution [11–13]. Compared with these conventional methods, additive manufacturing (AM) technology with rapid solidification shows the capability to prepare porous material with uniform pore structure and enhanced mechanical strength. Laser melting deposition (LMD), as one of the AM methods [14, 15], was used to prepare porous Al foams due to its convenient operation and low cost. Al foams with different sound-insulated through LMD were prepared using nickel (Ni) modified TiH₂ as the foaming agent by tuning porosity and pore size [16]. See et al [17] used zirconium hydride (ZrH₂) as a foaming agent to prepare Al foams with a maximum porosity of 25% by LMD, confirming that the excessive ZrH₂ would cause brittle fracture of the Al foams. However, the metal foams prepared by the LMD method have the problem of uneven distribution of pores, mainly caused by the low local viscosity of the molten Al metal due to the concentration of laser energy [16, 18, 19]. On the other hand, the mechanical properties of Al foams also decreased. Earlier research suggested that adding ceramic particles such as SiC, MnO₂, and Al₂O₃ to melt was an excellent manner for increasing the viscosity in the powder metallurgy route [20–22]. As well known, SiC has the characteristics of high-temperature stability, high strength, and low thermal expansion coefficient [23]. Therefore, SiC addition could increase the viscosity of the melt to prevent bubbles from escaping and coalescing and enhance the high-temperature resistance. On the other hand, SiC could also act as the grain growth inhibitor to refine grains and adsorb on the gas-liquid interface to inhibit the expansion of pores and then optimize the pore structure. To the best of our knowledge, the influence of SiC on the microstructure and properties of Al foams manufactured by LMD has not been reported.

In this study, Al/SiC composite foams were successfully manufactured by LMD with TiH₂ foaming agent. The influence of TiH₂ and SiC contents on the microstructure, pore size, and pore distribution were investigated
systematically. Moreover, the mechanism of SiC reinforcement on the compressive properties of Al/SiC composite foams was also discussed in detail.

2. Materials and methods

The raw materials of Al powders (50–75 μm, 99.5 wt% in purity) and TiH₂ powders (75 μm, 99.5 wt% in purity) were used to prepare Al foams by LMD in this study. Besides, to improve the strength of Al foams, SiC nanoparticles (500 nm, 99.9 wt% in purity) were employed to enhance the Al matrix by forming Al/SiC composite foams. The amount of SiC addition was set as 0.1 wt%, 0.2 wt%, 0.3 wt%, 0.5 wt%, 0.75 wt% and 1.0 wt%, respectively. For comparison, the Al foams and Al/SiC composite foams were fabricated under the same conditions and the manufacturing parameters as listed in Table 1. The Al powders, SiC nanoparticles (Al/SiC composite foams only), and TiH₂ powders were firstly mixed by ball-milling at 200 rpm for 2 h in a planetary ball mill. Then, the mixed powder was sprayed into an Al substrate and melted with the substrate under a high-energy laser beam under high pure argon protection. The schematic of preparing Al foams and laser path strategy is illustrated in Figure 1. The following formula calculated the porosity of Al foams:

\[
\varepsilon = \left(1 - \frac{m}{\rho V}\right)
\]

Where \(\varepsilon\) and \(m\) are the porosity and the mass of Al foams, \(\rho\) is the density of the alloy, and \(V\) is the volume of the Al foams measured by the weighing method.

The samples of pure Al foams and Al/SiC composite foams were cut by Electro-Discharge Machining. The coarse surface of pieces was removed using sandpaper and polished by Electrolytic Polishing Instrument (EP-06). The pore structure was observed by Stereo Microscope (OLYMPUS SZX10). The microstructure of samples was analyzed by an optical microscope (OM, IEM500) and Scanning Electron Microscopy (SEM, JOEL JSM-7900F). The electronic universal testing machine (Instron 5985) tested the compression performance of the samples with a size of \(15 \times 10 \times 10\) mm³ at room temperature with a compression rate of 1 mm min⁻¹.
3. Results and discussion

3.1. Pore structure tuning by the addition of TiH₂

The effects of foaming agent content on the microstructure and porosity of Al foams were investigated firstly, and the results are shown in figure 2. It can be seen from figures 2(a)–(e) that the porosity increases gradually with increasing the content of TiH₂, while the size and shape of the pores in Al foams are not uniform when the TiH₂ content is 2 wt%, there are many tiny pores with ∼100 μm generated in the foams, which are close to a circular shape. Further increasing the TiH₂ content to 4 wt%, the size of pores significantly increased to ∼250 μm. Moreover, when the TiH₂ content is up to 10 wt%, the diameter of pores reaches 2 mm. Figure 2 shows the variation of pore size distribution and the average diameter, respectively, with adding different TiH₂ content. It is found that as the foaming agent content increases, the proportion of tiny pores (<250 μm) reduces while the ratio of mesoporous (250–500 μm) and large pores (>500 μm) increases. The results suggested that with the increase of TiH₂ foaming agent, there was agglomeration and merging of tiny pores during the foaming process, and consequently, the average diameter of pores increased.

Figure 2(f) shows the relationship between the porosity of Al foams and the addition of TiH₂. With the increase of TiH₂ content, the porosity gradually increased and reached the maximum ∼47.2 ± 2% when the...
TiH$_2$ content was 8 wt%. However, when the TiH$_2$ content continued to increase to 10 wt%, the porosity decreased slightly to $\sim 44.7 \pm 2\%$. This is mainly related to the addition of excess foaming agents. When the foaming agent is excessive, it affects the fluidity of the mixed powder. It generates a large amount of hydrogen, which leads to the continuous expansion of the pores until they are broken, thereby reducing the porosity [16].

Figures 3(a)–(e) shows the OM microstructure of Al foams with different TiH$_2$ content. It was found that there are needle-like Al$_3$Ti precipitates in the aluminum foams structure after adding TiH$_2$, and with the increase of TiH$_2$ content, the quantity of Al$_3$Ti precipitates increases significantly. Unlike the traditional Al foams preparation process, which consists of three stages: stirring, keeping foaming, and solidification cooling [19], the LMD shows Marangoni convection instead of a stirring step. At this stage, the Marangoni flow will produce a specific blending effect to make the pore uniform [24]. When the amount of Al$_3$Ti is relatively tiny, the fluidity of the melt is better, so the Marangoni heat flow has a more substantial effect of making large pores become tiny pores, which is also confirmed by the results in figure 2. The highest porosity was achieved by adding 8 wt% TiH$_2$ based on the above results. Therefore, to further investigate the effect of SiC addition on the microstructure and properties of SiC reinforced aluminum composite foams, the content of TiH$_2$ was set as 8 wt%.

Figure 3(f) shows the XRD patterns of samples with different TiH$_2$ content. It can be seen that the matrix is the elemental aluminum phase, and with the addition of TiH$_2$, some new Al$_3$Ti phase is generated during the process of LMD. Those Al$_3$Ti phases with high hardness dispersed in the Al matrix, reinforcing against the aluminum foams.

3.2. The effect of SiC addition on the structure and properties of Al foams
The pore structure of Al/SiC foams with different SiC additions are shown in figures 4(a)–(f). The proper process and rapid solidification characteristics enable SiC and pore uniformly distributed in the Al/SiC composite foams. With the introduction of SiC nanoparticles, the nanoparticles SiC existing on the gas-liquid interfaces would increase the melt viscosity and reduce the fluidity [8, 25]. Meanwhile, the SiC and TiH$_2$ can be
acted as the core of pore heterogeneous nucleation [26]. Those are helpful to improve the pore structure of Al foams.

On the other hand, with the increase of SiC content, the viscosity of the melt increases, which would inhibit the expansion of pores and help decrease the pore diameter and obtain a uniform distribution of pore size, as shown in figures 4(g)–(h). However, the addition of SiC has an essential effect on the porosity, as shown in figure 4(h). When the amount of SiC reached 1.0 wt%, the porosity was reduced from 42.7 ± 2% to 28.3 ± 2%. This is because the SiC absorbed on the pore interface can also hinder the expansion of pores and reduce the porosity during the foaming process.

The effects of SiC addition on the microstructure evolution of composite foams are shown in figure 5. It can be seen that with the increase of SiC addition, the grain size of Al3Ti precipitates decreases gradually. When the addition amount of SiC does not exceed 0.2 wt%, SiC addition has little influence on the melt fluidity and growth inhibition of Al3Ti precipitates. SEM was used to observe further the microstructure evolution caused by SiC addition and to analyze the microstructure of pure Al foams and 1.0 wt% Al/SiC composite foams, as shown in figure 6. From figures 6(a)–(b), it was found that the grain size of Al3Ti precipitates in 1.0 wt% SiC sample is more refined than that of pure Al foams. The Al3Ti formed in 1.0 wt% Al/SiC composite foams is 160.4% smaller than pure Al foams. Figures 6(a)–(b) shows that the presence of SiC nanoparticles as a reinforcing phase will cause the matrix material to recrystallize, leading to grain refinement [23].

The mechanical properties of 0.5 wt% Al/SiC composite foams and pure Al foams were tested, and the results are shown in figures 7(a)–(d). In this case, both two kinds of foams have approximately the same porosity, about 39.0%. It can be seen clearly from figures 7(a)–(b) that the pores in 0.5 wt% Al/SiC composite foams are more dispersed than pure Al foams.

Furthermore, the average pore diameter of pure Al foams reaches up to 321 μm, which is 33.8% larger than that of 0.5 wt% Al/SiC composite foams. Figures 7(a)–(b) also shows the statistical data distribution of pore size, indicating that the pore distribution of Al/SiC composite foams is more uniform. Early research suggested that small pore size and evenly distributed pores were beneficial for improving the compression performance of foams [27, 28]. Pores with variable sizes are more likely to cause stress concentration under external load, leading
to cracking of the pore structure. Figure 7(c) shows the compressive stress-strain curves of pure Al foams and Al/SiC composite foams. It could be found that the shape of the curve is similar to that of Al foams prepared by the SLM method, which has three-stage characteristics like traditional Al foams: the elastic stage, the plastic plateau stage, and the densification stage [17, 29]. The area where the strain is less than 0.5% is the elastic deformation stage, and the area from the compressive yield stress $\sigma_c$ to the densification strain $\varepsilon_D$ is the plastic plateau stage. This study obtained the compressive yield stress $\sigma_c$ of the foams material by the 0.2% offset method. The densification strain, which indicates the compressive energy absorption capacity, is the strain where the plastic plateau’s tangent line intercepts the densification region’s slope. The energy absorption curve of Al foams and Al/SiC foams are shown in figure 7(d), and the energy absorption capacity is defined as follows [30]:

$$E = \int_0^\infty \sigma(\varepsilon) d\varepsilon$$  \hspace{1cm} (2)

Where $E$ represents the quantity of energy absorption, $\varepsilon$ is the strain of materials, and $\sigma(\varepsilon)$ is stress at $\varepsilon$ strain.

The densification strain, porosity, the average pore size, energy absorption, compressive yield stress, and the energy absorption $E_c$ derived from formula (2) are listed in Table 2. The compression yield strength and the amount of energy absorption (compressive strain was 60%) of the Al/SiC composite foams are 41.1 MPa and $64.4 \times 10^6$ J m$^{-3}$, respectively. In contrast, the pure Al foams are 28.5 MPa and $55.5 \times 10^6$ J m$^{-3}$. Compared with the pure Al foams, the compression yield strength of the Al/SiC composite increased by 43.8%, and the amount of energy absorption of it increased by 16.0%. Under the external load, the SiC particles and fined Al$_3$Ti can pin the dislocations and make the dislocation bend around them, which induces the Orowan strengthening [8].

![Figure 7: Stereo Microscope images and Pore size distribution of (a) pure Al foams and (b) Al/SiC foams, (c) Compressive stress-strain curves, (d) Energy absorption capacity curves of Al/SiC foams and pure Al foams.](image)

![Table 2. Compressibility and energy absorption capacity of porous materials.](table)

| Materials     | Porosity(%) | $\sigma_c$ (MPa) | $\varepsilon_D$(%) | $E_c$ (MJ/m$^3$) | The average pore size ($\mu$m) |
|---------------|-------------|------------------|--------------------|------------------|-------------------------------|
| Pure Al foams | 39.1        | 28.5             | 60.3               | 55.5             | 321                           |
| Al/SiC foams  | 38.8        | 41.0             | 59.5               | 64.4             | 240                           |

The number of energy absorption, $e$ is the strain of materials, and $\sigma(\varepsilon)$ is stress at $\varepsilon$ strain.
On the other hand, the difference of thermal expansion coefficients between SiC and Al matrix leads to thermal mismatch plastic strain, thereby releasing dislocation loops to relax the stress [23]. Then the high density-dislocation will strengthen the Al matrix. Furthermore, the grain refinement caused by SiC also plays a significant role in increasing the compressive yield strength. In addition, SiC particles and pores will also dissipate some energy through friction under the action of external load. These factors can both enhance the amount of energy absorption. Above all, one can be inferred that an appropriate addition of SiC nanoparticles is beneficial to achieve a homogenized distribution of pores, refined second phase and grains, thus enhancing the compression yield strength and energy absorption capacity significantly.

To further analyze the compressive properties of composite foams, we performed compression tests of Al/SiC foams with different porosity. Figure 8 shows that the higher the porosity of SiC composite foams, the lower the yield strength [16]. The yield strengths of the samples with porosity of 28.3%, 36.1%, and 40.0% were 55.1 Mpa, 39.9 Mpa, and 36.6 Mpa, respectively, and the energy absorption were $99.5 \times 10^6$ Jm$^{-3}$, $69.2 \times 10^6$ Jm$^{-3}$, and $53.9 \times 10^6$ Jm$^{-3}$, respectively. The low-porosity SiC composite foams have thicker cell walls to withstand larger loads. Therefore, the low-porosity SiC composite foams need a more significant load to make them deform. So the yield strength and energy absorption of the low-porosity SiC composite foams are higher.

4. Conclusions

The SiC particles were applied to enhance aluminum foams by the LMD, and the Al/SiC foams with high compressive strength, uniform pore dispersion and high energy absorption were obtained in this work. Firstly, the closed-cell Al foams with a maximum porosity of 47.2 ± 2% were prepared successfully when the TiH$_2$ content was 8.0 wt%. Meanwhile, 0.5wt% SiC nanoparticles addition is beneficial to realize a homogenized distribution of pores, a refined second phase, and grains. As a result, the Al/SiC composite foams have superior compression yield strength properties and energy absorption capacity with 41.1 MPa and $64.4 \times 10^6$ Jm$^{-3}$, respectively, which increased by 43.8% and 16% compared to the pure Al foams.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.
CRediT authorship contribution statement

Tao Zeng: Writing- original draft preparation, analysis, and interpretation of the data. Methodology- design of the experiment. Ying Liu: Writing- revising the manuscript critically for significant intellectual content. Lu Wang: Writing- Reviewing and editing. Yu Gao: Data curation, Writing- Original draft preparation. Renquan Wang: Writing- review and editing, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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