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Thermal expansion behavior of CNT reinforced AlSi10Mg composite fabricated via laser powder bed fusion

I. Y. Jiang, T. T. Liu, C. D. Zhang, K. Zhang, T. Yang, C. C. Zhang and W. H. Liao

National Joint Engineering Research Center of NC forming technology and equipment, School of Mechanical Engineering, Nanjing University of Science and Technology, Nanjing 210094, People’s Republic of China

E-mail: liutingting@mail.njust.edu.cn and cnwho@njust.edu.cn

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Abstract

Carbon nanotube (CNT) reinforced Al matrix composite exhibit good dimensional stability and can be a promising material with low coefficient of thermal expansion (CTE). The thermal expansion behavior of CNT/AlSi10Mg composite with the CNTs content from 0 wt% to 2 wt% fabricated via laser powder bed fusion (LPBF) was investigated in this research. The CTE of all samples increased generally as the temperature increasing. At the same temperature, the CTE of the samples was smaller when CNTs content is lower. The best dimensional stability was reached as CNTs content was 2 wt%. The CTE deceased to 29.08 $\times$ 10$^{-6}$ C$^{-1}$, as much as 16% reduction compared with unreinforced AlSi10Mg at 300 $^\circ$C. The solid solution and precipitation behavior of Si in matrix and the interruptions of Si network resulted in the fluctuation during dimension increase. With the CNTs distributed along the cell boundary, the Si network was consolidated. Hence, the microstructure of the composite became more stable and the CTE got lower with the addition of CNTs increasing. Moreover, the experimental results were similar to the predicted value of the thermoelastic model.

1. Introduction

Aluminum alloys are widely used in aerospace, aviation and automobile industries because of their high specific strength. However, the high coefficient of thermal expansion of aluminum alloy limits its application in the fields requiring high dimensional stability, such as inertial navigation system and electronic packaging system. Fortunately, aluminum matrix composites show good performance in dimensional stability [1, 2] and they are gradually applied in the fields of high dimensional accuracy requirements for their high designability [3, 4].

Zhang et al [4] fabricated a 70 vol% SiC/pure-Al composite by the pressure infiltration technique. The addition of high volume fraction of SiC particles led to a 60% decrease of CTE ($20^\circ$–$100^\circ$C), Chen et al [2] utilized 45 vol% Si$_3$N$_4$ particles to reinforce 2024Al alloy by pressure infiltration. The CTE of Si$_3$N$_4$/2024Al composite was 50% lower than the matrix’s. Tayebi et al [5] employed hot pressing method to prepare the Al/B$_4$C composite with the B$_4$C volume fraction from 5% to 35% and the results showed that 25% B$_4$C/Al had the lowest CTE. From the previous studies, it can be found that the excellent thermal strain resistance can be normally got with high fraction of reinforcement in the field of the ceramic reinforced composites.

CNTs has excellent properties [6] and is known as one of the new generation reinforcements of composites [7, 8]. The properties of CNT reinforced aluminum matrix composites are expected to exceed those of traditional aluminum matrix composites reinforced by ceramic particles and carbon fibers. The CTE of CNTs is close to zero [9], which can effectively inhibit the thermal expansion behavior of matrix materials with relatively low content. Deng et al [10] prepared 1.0 wt% CNT/2024Al composite by cold pressing and hot extrusion. They found that the CTE of aluminum alloy matrix was significantly reduced by adding CNTs and it decreased 11% compared with that of 2024 aluminum alloy (50 $^\circ$C). Zhao et al [11] studied the thermal expansion properties of CNT/Al composites prepared by sintering and hot extrusion. It showed that the CTE of samples with 4.5 wt% CNTs could be 17% lower than that of Al matrix (100 $^\circ$C). Liu et al [12, 13] studied the thermal expansion
properties of CNT/2009Al composite specimens treated by friction stir processing. The results showed that a small amount of CNTs could significantly reduce the CTE of the specimens. When the CNTs content was 4.5 vol%, the CTE of the composite decreased about 25% compared with the matrix (300 °C). Those researches showed great potential of CNTs in improving the dimensional stability of the matrix materials. However, these manufacturing method can merely fabricate the Al matrix composites with regular structures.

Laser powder bed fusion creates parts by scanning powdered materials with a laser beam to melt and fuse the material into a solid, the parts being manufactured layer by layer direct from the CAD file data. This process, one of the widely used additive manufacturing technologies, shows great advantages in the fabrication of complex structures parts [14, 15] and homogeneous reinforced composite parts [16–18]. Considering the excellent performance of CNTs, more and more researchers have given the focus on the research of CNT/Al composites fabricated by LPBF. Zhao et al [19] conducted the research including powder preparation, forming process and performance of 1 wt% CNT reinforced AlSi10Mg composite printed by LPBF. The results showed that the hardness and conductivity of the composite samples were improved with the addition of CNTs. In the study of Du et al [20], 1 wt% CNT reinforced AlSi10Mg composites formed by LPBF and FSP were compared. The mechanical properties of LPBF specimens (tensile strength of 287 ± 11 MPa) were better than those of FSP specimens (tensile strength of 187 ± 28 MPa). CNTs were detected at the fracture surface of LPBF specimens, and the strengthening effect of CNTs was speculated. Wang et al [21] studied the properties and microstructures of 1 wt% CNT/AlSi10Mg specimens under different bulk energy densities. The microstructures of this composite were refined and the hardness of it was improved a lot (143.7 HV0.1) with a proper parameter.

Those researches above exhibited the possibility of fabricating CNT/AlSi10Mg composites by LPBF and remarkable improvements of mechanical properties to the LPBF parts with the introduction of CNTs. However, there has been no report on the thermal expansion behavior of CNT reinforced AlSi10Mg composites fabricated via LPBF yet. In this paper, the CNT/AlSi10Mg composite with the content of CNTs from 0 wt% to 2 wt% was fabricated and the role of CNTs on dimensional constraints of composite prepared by LPBF was estimate. The effect mechanism of CNTs on the thermal expansion behavior of LPBF CNT/AlSi10Mg composites were analyzed according to the microstructures of samples with different CNTs contents before and after thermal expansion experiments.

2. Experimental procedure

2.1. Materials
The AlSi10Mg powders, D50 of 53.8 μm (figure 1(a)) (Concept Laser GmbH Co. Ltd, Germany) and CNTs (Chengdu Organic Chemistry Co., Ltd, China), length of 10–30 μm and diameter of 20–30 nm (figure 1(d)), were utilized in our research. Two materials were weighed according to the specific ratio. Then mixed powders with 1 wt% CNTs (figures 1(b) and (e)) and 2 wt% CNTs (figures 1(c) and (f)) were obtained by the colloidal mixing method [22]. The CNTs on the surface of AlSi10Mg powders were marked with the yellow arrows (figures 1(e) and (f)).
2.2. LPBF process
The Concept Laser M2 Cusing (400 W) equipment was utilized to fabricate the samples with CNTs content of 0 wt%, 1 wt% and 2 wt%. The argon with purity of 99.999% was used as protective gas. Based on the previous research [23], the LPBF parameters were set as: the laser power of 370 W, the scan speed of 1300 mm s⁻¹, the scan hatch of 105 μm, and the layer thickness of 30 μm. The scan strategy of zigzag scan in same layer and 90° rotation between adjacent layers was utilized (figure 2(a)).

The cubes with dimension of 10 × 10 × 10 mm³ were printed for microstructure morphology study of as-built samples. The cylinder parts (figure 2(b)) with the dimension of φ6 × 20 mm³ were cut from cuboids of 8 × 8 × 25 mm³ by wire electrical discharge machining (WEDM) for the investigation of thermal expansion behavior of composites. Cubes of 5 × 5 × 3 mm³ cut along the axis of cylinder parts (figure 2(c)) were used to investigate the microstructure of samples after CTE test.

2.3. Characterization
The CTE test was conducted on a DIL 402C thermomechanical analyzer (NETZSCH Group, Germany) at a heating rate of 5 °C min⁻¹ from 29 to 300 °C. XRD measurements were conducted on a D8 X (Bruker, Germany), operating with a cobalt anticathode in the angular range (2θ) from 10° to 80° and scan step of 0.02° min⁻¹. After grinding and polishing, the parts for microstructure investigation were etched with Keller reagent (2.5 ml HNO₃, 1.5 ml HCl, 1 ml HF and 95 ml H₂O) for 30 s. A Quanta 250F (FEI, Czech) scanning electron microscope (SEM) was used to analyze the microstructure morphology of samples at the test voltage of 30 kV. An FEI Tecnai G2 transmission electron microscope (TEM) was used to characterize the CNTs in samples prepared by ion milling at 200 kV test voltage.

3. Results
3.1. The CTE of samples
The dimension increase ratio (DIR) curves of the samples are shown in figure 3. When the CNTs content was a constant, the DIR along the test direction increased with the increase of temperature generally. When the temperature was lower than 150 °C, there was no obvious difference among the DIR curves of samples with different CNTs contents. When the temperature rose from 150 °C to 250 °C, the difference among the DIR curves of samples increased with the increase of temperature. The higher the CNTs content was, the smaller DIR...
the samples was. When the temperature was higher than 250 °C, the difference among the DIR curves of the samples tended to be stable. The DIR curves of all samples had inflection points near 120 and 230 °C, showing a change with first steep and then smooth.

The CTE of samples can be calculated (figure 4) from the following equation:

\[ \alpha = \frac{\Delta l}{L_0 \cdot \Delta t} \]  

where \( \alpha \) is the coefficient of linear expansion, \( \Delta l \) is the absolute value of the change of sample length, \( L_0 \) is the original length of the sample, \( \Delta t \) is the absolute value of temperature difference during test. With the increase of CNTs content, the CTE of the samples decreased and the dimensional stability of the sample increased when the temperature was a constant. When the content of CNTs was 2 wt%, CTE of the sample was 29.08 \( \times \) 10\(^{-6} \) °C\(^{-1} \) at 300 °C, which was about 16% lower than that of the sample without CNTs.

3.2. Phase characterization

XRD patterns (figure 5) showed that all the diffraction peaks from Al or Si, indicating that the phase of samples did not transform during the CTE test. However, the (111) and (200) characteristic peaks of Al phase showed some differences from the local enlargement diagram of XRD curve (figure 5(b)). After CTE test, the peak position of the sample shifted to the left. Because of its extremely fast cooling rate [24], the solid solubility of Si in Al of AlSi10Mg part fabricated via LPBF was as high as 5.4 wt%, much higher than that of 0.5 wt% after solution treatment [25]. Therefore, the supersaturated solid soluble Si atoms in the sample will gradually precipitate from \( \alpha \)-Al during the process of temperature rising. Since the radius of Si atom is smaller than that of Al atom, the lattice constant of Al increased after the precipitation of Si atom, which made the characteristic peak of Al shift to the left. In addition, the characteristic peaks of CNTs, Al\(_4\)C\(_3\) and Mg\(_2\)Si phases were not obvious in XRD patterns. This was a result of the relative low content of these phases in the samples.

3.3. Microstructure

The microstructures of the LPBF samples with different CNTs contents were similar (figures 6(a)–(c)). They had the typical structure of Al-Si alloy LPBF samples [26–28]: The \( \alpha \)-Al solidified in a cellular morphology, surrounded by discontinuous network of the residual Si concentrating at the cellular, and the width of the cells was about submicron size. Image J software was used to calculate the mean cell size of the sample with 0 wt% to 2 wt% CNTs content (table 1). The results showed that the cell size of the sample decreased with the increase of CNTs content.

After CTE test, the microstructures of the samples had obvious difference (figures 6(d)–(f)). Compared with the microstructures before test, the Si atoms segregated from initial network by diffusion, leading to the interruptions of Si network, and grew up at the nucleation point forming coarser particles. Fortunately, with the increase of CNTs content, the Si network in the sample remained more complete. The average cell size of samples with 2 wt% CNTs was the smallest, which was about 533 nm.
Figure 5. (a) is the XRD results of samples before and after CTE test. (b) is the local enlargement diagram of as-built AlSi10Mg sample and the part after CTE test.

Figure 6. Microstructures of samples before and after CTE test. (a)–(c) were the micrographs before the test of samples with 0 wt%, 1 wt% and 2 wt% CNTs, respectively. (d)–(f) were the micrographs after the test of samples with 0 wt%, 1 wt% and 2 wt% CNTs, respectively.
4. Discussion

Solid solution and phase transition in the material are considered as the common causes of CTE change \[29, 30\]. The change of microstructure with CNTs addition will also affect the thermal expansion behavior of the material \[31\]. Si has a diamond lattice \((a = 5.430 \text{ Å})\) \[32\], 8 atoms per unit cell, and Al has a fcc lattice \((a = 4.041 \text{ Å})\) containing 4 atoms per unit cell. Therefore, the volume of AlSi10Mg matrix will increase when Si atoms precipitate from Al lattice. In this study, with the increase of temperature, the supersaturated Si in \(\alpha\)-Al gradually precipitated (figure 5(b)), which made the curve of sample DIR steep. And the DIR curves of the samples became smooth after the precipitation finished. TEM results showed that CNTs were evenly dispersed at the cell boundary in LPBF samples (red arrows figure 7) and partially reacted to Al\(_4\)C\(_3\) (yellow arrows in figure 7). Because of the location limitation, CNTs had little influence on the precipitation process of Si atoms from \(\alpha\)-Al. It can be inferred that the sudden change near 120 °C (figure 3) was dominated by the precipitation of supersaturated Si.

As the temperature increase, the DIR curves of the samples became steep again after 150 °C. It had the similar phenomenon in the thermal expansion behavior study of Al-50Si LPBF part by Jia et al \[33\]. Thus, the interruptions of Si network at this stage was thought to be the reason for this sudden change of samples’ dimension increase. In the samples with CNTs, CNTs were distributed at the cell boundary (figure 7) and reinforced the Si network in the matrix. This effectively hindered the aggregation of Si during continuous heating (figure 6), thus limiting the softening of matrix. The morphologies of CNTs in samples with 1 wt% and 2 wt% content were quite different: the length of CNTs in samples with 1 wt% content was about 200 nm (figure 7(a)), which was less than the cell boundary length; the length of CNTs in samples with 2 wt% content was about 900 nm (figure 7(b)). The length and distribution density of CNTs in samples with 2 wt% content were higher, so the contact area between CNTs and the cell boundaries was larger. As a result, the constraints on the matrix were relatively large \[34, 35\], which was conducive to limiting the increase of cell size in the process of temperature rise. Therefore, the DIR curves and CTE curves of samples with different CNTs contents showed increasing difference in the test range from 150 to 250 °C, and the CTE reached the lowest point when CNTs content was 2 wt%.

The solubility of Si in Al increased when the temperature was above 250 °C. Some Si re-dissolved into the lattice of Al, which reduced the volume of the material and partially neutralized the volume expansion of matrix with the increase of temperature \[36\]. Thus the DIR curves of the samples were flattened and the CTE decreased.

Generally, the CTE results showed strong dependence on the content of CNTs. Several models have been proposed based on thermoelastic theories for the prediction of CTE of MMCs, which can be used to explain the dependence of CTE on the CNTs fraction of CNT/AlSi10Mg composite. The three most commonly used models are Schapery, Kerner and Turner.

Schapery’s model was derived through extremum principles of thermoelasticity. The expression of it is given as \[5\]:

| CNTs Fraction | 0 wt% | 1 wt% | 2 wt% |
|---------------|-------|-------|-------|
| Before CTE test (nm) | 380.991 | 321.136 | 292.34 |
| After CTE test (nm) | 658.828 | 609.441 | 533.697 |

Figure 7. CNTs in the as-built samples with different CNTs contents: (a) 1 wt% and (b) 2 wt%.
where, \( c, r \) and \( m \) indexes represent the composite, the reinforcement and the matrix, respectively. \( \alpha_c^u \) is the upper bound of the CTE of the composite \( K_c \) is the lower bound of the bulk modulus of the composite and the expression is given as:

\[
\alpha_c^u = \alpha_r + \frac{K_m (K_r - K_m^l) (\alpha_m - \alpha_r)}{K_c^l (K_r - K_m)}
\]

\[
K_c^l = K_m + \frac{V_r}{K_r - K_m} + \frac{V_c}{K_c^l V_r (K_r - K_m) + 4G_m/3}
\]

where, \( G_m \) is the shear modulus of the matrix. The upper bound of the bulk modulus can be calculated by exchanging \( r \) and \( m \) in the equation (3).

Kerner’s model takes the shear modulus of the constituents into account. And the upper band of the Schapery is coincided with the Kerner [37].

Turner’s model takes the elastic modulus and the Poisson’s ratio of each component materials into account. The expression of it is given as [38, 39]:

\[
\alpha_c = \frac{\alpha_r V_r K_r + \alpha_m V_m K_m}{V_r K_r + V_m K_m}
\]

The results predicted by Kerner model and Turner model were showed in figure 8. The experimental data of matrix was utilized to brought into the calculation in the temperature range of 50 °C–300 °C. The comparison of experimental and theoretical data showed that the experimental data was closer to Turner’s model generally. Especially, the test results of both samples were almost coincide with Turner model at temperatures below 100 °C. There was a slight higher away trend of experimental data to Turner’s model in the results of sample with 1 wt% CNTs (figure 8(a)). And the trend in the results of sample with 2 wt% CNTs was more obvious, which exceeded Kerner model (figure 8(b)). This trend was consistent with the analysis of the effect by Si network interruptions. At temperatures above 250 °C, the experimental CTE was even smaller than Turner model. Overall, figure 8 showed that the experimental data lay closer to Turner model and lower than the prediction of Turner model at high temperature (above 250 °C).

From the comparison of the theoretical and experimental results, it showed that the experimental conditions can be basically described by these models. Especially the Turner model showed high precision and validation of the prediction on CTE. The main reason for major difference of experimental and theoretical results around 150 °C was that thermoelastic models do not consider the effect of precipitation and solid solution of the material during heating process. Because of the high aspect ratio of CNTs, the experimental CTE was smaller than prediction at high temperature.

5. Conclusion

The thermal expansion behavior of laser powder bed fusion specimens of 0 wt%, 1 wt% and 2 wt% carbon nanotube reinforced AlSi10Mg composites was studied. The following conclusions were drawn:

(1) During thermal expansion test, Si undergoes the process of precipitation then solid solution in \( \alpha \)-Al, and the Si network was gradually interrupted. This two reasons made the dimension increase ratio curve of the
samples fluctuate in the process of temperature rising. The precipitation of Si atoms made the diffraction peaks of Al(111) and Al(200) shift left in the XRD results of the sample after thermal expansion test. There was no obvious phase transition in the samples before and after test.

(2) The uniform distribution of CNTs at the cell boundary plays a role in stabilizing the Si network in the matrix. With the increase of CNTs content, the more complete CNTs were survived in the sample, which reduced the CTE of LPBF samples. The CTE of the sample of 2 wt% CNTs content was the lowest, decreasing to $29.08 \times 10^{-6} \text{ C}^{-1}$, which was about 16% lower than that of unreinforced AlSi10Mg at 300 °C.

(3) The experimental CTE lay closer to Turner model and lower than the prediction of Turner model at high temperature (above 250 °C). The main reason for major difference of experimental and theoretical results around 150 °C was that these thermoelastic models do not consider the effect of precipitation and solid solution of the material during heating process, as well as the high aspect ratio of CNTs.

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ORCID iDs

T T Liu https://orcid.org/0000-0002-0699-8196

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