The Effect of Annealing on Microstructure and Mechanical Properties of Selective Laser Melting AlSi10Mg

Changchun Zhang¹, Haihong Zhu²*, Yang Qi³, Xiaoyan Zeng⁴
Wuhan National Laboratory for Optoelectronics, Huazhong University of Science and Technology, Wuhan, Hubei, P.R. of China

*zhuhh@hust.edu.cn

Abstract. The effect of annealing treatment on SLM AlSi10Mg on microstructure and mechanical properties has been investigated. It is observed that, with the increase of the temperature, the melt pools gradually become blurred and completely disappear when the temperature rises to 320°C. The microhardness and strengths of as-built samples are the highest due to the solid solution strengthening together with grain refinement strengthening. After heat treatment, the microhardness and strengths all decrease due to the weakening of solid solution strengthening but the elongation increases.

1. Introduction
Selective laser melting (SLM) is an additive manufacturing (AM) technique which can directly fabricate 3D parts from metal powders [1]. Compared with traditional manufacturing methods, it has the advantages of the high utilization rate of raw materials, short manufacturing cycle, near net forming, free from the limitation of the complex shape of parts. AlSi10Mg is a widely used aluminum alloy in aerospace and automobile industry for its high specific strength and excellent corrosion resistance. Furthermore, SLM Al-Si alloy has much better corrosion resistance than the cast counterpart, and different planes exhibit various corrosion properties[2,3]. The microstructure of the as-built aluminum parts is reported to be a very fine cellular-dendrite structure, the tensile strength is high but the elongation is poor. In order to balance the strength-elongation and obtain better comprehensive mechanical properties. It is necessary to control the microstructure and properties through heat treatment.

Li et al. [4] investigated the influence of solution and artificial aging heat treatments on the microstructure and mechanical properties of SLM AlSi10Mg parts. After heat treatment, the tensile strength decreased significantly. Takata et al. [5] examined changes in microstructure and mechanical properties of AlSi10Mg alloy at 300°C or 500°C. Heat treatment made fine Si phase precipitates within the columnar α-Al matrix and coarsening of the eutectic Si particles. Heat treatment eliminated the direction dependence of tensile ductility. Rosenthal et al. [6] provided a correlation between the mechanical properties of vertically built AlSi10Mg specimens subjected to T5 stress relief treatment, modified T5 treatment and Hot Isostatic Pressing treatment. It was found that the modified T5 treatment resulted in an increase in strength values beyond those of as-built condition. It can be seen that the current research on the heat treatment of AlSi10Mg mainly focus on the solid solution and aging heat treatment. Very little research has been done on low temperature annealing of AlSi10Mg. Fiocchi et al. [7] investigated changes in microstructure of AlSi10Mg after annealing. However, the
study of mechanical properties has only hardness but not tensile strength. It is necessary to study the relationship between the microstructure and mechanical properties especially tensile properties. This work investigated the effect of low temperature annealing on SLM AlSi10Mg. The phase, microstructure, microhardness and tensile properties were discussed.

2. Materials and Experiment Procedures

2.1. Materials

The gas atomized spherical AlSi10Mg powders with an average particle size of 33.7μm were used in the experiments. The chemical composition (wt. %) was 9.15 Si, 0.18 Fe, 0.015 Cu, 0.225 Mn, 0.3 Mg, 0.01 Zn, 0.011 Ti and Al (balance).

2.2. SLM Process

The SLM experiments were conducted on a self-developed machine (LSNF-2), whose details have been given elsewhere [8]. All samples were deposited in an argon environment with the concentrations of O₂ controlled below 200 ppm. The processing parameters of cubic and tensile samples were chosen as follows: laser power (P) 490 W, scanning speed (V) 2000mm/s, layer thickness (δ) 40 μm, hatch spacing (S) 0.10 mm and a zigzag scan pattern with 90° rotation between adjacent layers. The samples were heat treated at 260°C, 280°C, 300°C and 320°C for 2 hours, furnace cooling, respectively.

2.3. Characterization

For microstructure analysis, samples were etched by Keller reagent which consists of 2.5 mL HNO₃, 1.5 mL HCl, 1.0 mL HF and 95 mL deionized water. The microstructure of samples was observed by an EPIPHOT 300 optical microscopy (OM) and scanning electron microscopy (FEI NOVA NanoSEM450). Phase analysis was conducted by X-ray diffraction (PANalytical X’pert PRO) with Cu Kα radiation. Si particle size and density in the microstructure were estimated by business software [9]. Microhardness was tested using an HVS-1000 microhardness tester at a load of 200g and holding time of 15s. Tensile test samples were designed according to GB/T228.1-2010 standard (nearly equivalent with ISO 6892-1:2009), which was shown in Fig.1. Tensile tests were carried out using Zwick/Roll tester at the speed of 2mm/min.

![Figure 1. Configuration of tensile samples in this work.](image)

3. Results and Discussions

3.1. Microstructure Characterization

Fig.2 shows the optical micrographs of AlSi10Mg samples of as-built condition as well as different annealing temperatures. The layer-wise feature intrinsic to SLM technology is obvious. The layer to layer and melt track to melt track are closely stacked to form a good metallurgical bonding. With the increase of annealing temperature, the boundaries of melt pool gradually become blurred. When the temperature rises to 320°C, the boundaries of melt pool disappear completely, as shown in Fig.2 (e). Furthermore, SEM images with higher magnification were shown to investigate the details of the microstructure. The Si of as-built sample presents dendritic morphology and extends to the center of the melt pools, as shown in Fig.3 (a). After annealing treatment, Si changes to particles. Additionally, as the temperature increases, Si particles tend to grow up. It can be seen from Fig.3 (b)-(e).
Figure 2. Optical microscopy images of etched AlSi10Mg samples with different annealing temperatures: (a) as-built, (b) 260°C, (c) 280°C, (d) 300°C, (e) 320°C.

Figure 3. SEM images of etched AlSi10Mg samples with different annealing temperatures: (a) as-built, (b) 260°C, (c) 280°C, (d) 300°C, (e) 320°C.

Figure 4. Size and density of Si particles of samples with different annealing temperatures.

Figure 5. X-ray diffraction patterns of samples with different annealing temperatures.
The Si particle size and density at different annealing temperatures were calculated. The statistical results are shown in Fig.4. The results show that the Si particle size increases and the density decrease gradually with the increase of annealing temperature. This is because the heat treatment promotes atomic diffusion. Dendritic Si breaks into a discontinuous state, agglomerates and grows up to particles. Additionally, solid solution Si precipitates from the supersaturated α-Al matrix. This can be confirmed from the XRD test results shown in Fig.5.

3.2. Phase Analysis

Fig. 5 shows the phase analysis results on the samples after different annealing temperatures. Only Al peak and Si peak were detected in all the samples. With the increase of the annealing temperature, the diffraction peaks of Al phase slightly shift to lower angle, which implies the precipitation of Si phase from the supersaturated α-Al matrix. The higher the temperature, the more Si precipitation.

3.3. Microhardness

As for microhardness, 15 points were tested for each sample, and the average value was taken as the microhardness value of the sample. The test results were shown in Fig. 6. The results show that the microhardness of as-built samples is the highest. Annealing treatment reduces microhardness. The higher the temperature, the lower the microhardness. By calculating, the annealing temperature increases by 20°C, the microhardness decreases by 10-20 HV.

![Figure 6. Microhardness of samples with different annealing temperatures.](image)

![Figure 7. Tensile properties of samples with different annealing temperatures.](image)

3.4. Tensile Properties

Fig.7 shows the tensile results of AlSi10Mg samples with different annealing temperatures. With the increase of annealing temperature, The ultimate tensile strength (UTS) and yield strength (YS) decrease and the elongation (EL) increase, the annealing temperature increases by 20°C, the UTS decreases by 30-40MPa and the EL increases by 2-3%. Due to the high solidification rate, a supersaturated solid solution of aluminum is formed during the SLM process. Furthermore, the grains of the as-built sample are very fine [10]. Therefore, the strength of the as-built samples is the highest due to the solid solution strengthening together with grain refinement strengthening. After annealing treatment, Si precipitates from supersaturated α-Al matrix and grow up to particles. The resistance of lattice distortion to dislocation motion reduced. So the effect of solid solution strengthening is weakened. Similar phenomenon has been reported by Rabadia et al. [11]. This leads to a decrease in strength and increase in ductility.
4. Conclusions
The effect of annealing temperature on microstructure, phase and mechanical properties of SLM AlSi10Mg were investigated in this work. The main conclusions are as follows:

(1) With the increase of the annealing temperature, the melt pools gradually become blurred. When the temperature rises to 320°C, the melt pools disappear completely. The morphology of Si changes from dendritic into particles, agglomerates and grow up.

(2) Annealing treatment makes the Si phase to precipitate from the supersaturated α-Al matrix.

(3) The microhardness of as-built samples is the highest. Annealing treatment reduces microhardness.

(4) The strength of the as-built samples is the highest due to the solid solution strengthening together with grain refinement strengthening. After annealing, Si precipitates from the supersaturated α-Al matrix. The effect of solid solution strengthening is weakened. This leads to a decrease in strength and increase in ductility.

Acknowledgements
This work was supported by the National Natural Science Foundation of China (61575074). The Authors also thank the Analytical and Testing Center of HUST for the XRD and SEM analysis.

References
[1] B.L. Zhang, H. Attar, Selective laser melting of titanium alloys and titanium matrix composites for biomedical applications: a review, Adv. Eng. Mater 18 (2016) 463–475.
[2] Y. Yang, Y. Chen, J. Zhang, X. Gu, P. Qin, N. Dai, X. Li, J.-P. Kruth, L.-C. Zhang, Improved corrosion behavior of ultrafine-grained eutectic Al-12Si alloy produced by selective laser melting, Mater. Des. 146 (2018) 239-248.
[3] Y. Chen, J. Zhang, X. Gu, N. Dai, P. Qin, L.C. Zhang, Distinction of corrosion resistance of selective laser melted Al-12Si alloy on different planes, J. Alloys Compd. 747 (2018) 648–658.
[4] W. Li, S. Li, J. Liu, A. Zhang, Y. Zhou, Q. Wei, C. Yan, Y. Shi, Effect of heat treatment on AlSi10Mg alloy fabricated by selective laser melting: Microstructure evolution, mechanical properties and fracture mechanism, Mater. Sci. Eng. A. 663 (2016) 116–125.
[5] N. Takata, H. Kodaira, K. Sekizawa, A. Suzuki, M. Kobashi, Change in microstructure of selectively laser melted AlSi10Mg alloy with heat treatments, Mater. Sci. Eng. A. 704 (2017) 218–228.
[6] I. Rosenthal, R. Shneck, A. Stern, Heat treatment effect on the mechanical properties and fracture mechanism in AlSi10Mg fabricated by additive manufacturing selective laser melting process, Mater. Sci. Eng. A. 729 (2018) 310–322.
[7] J. Fiocchi, A. Tuissi, P. Bassani, C.A. Biffi, Low temperature annealing dedicated to AlSi10Mg selective laser melting products, J. Alloys Compd. 695 (2017) 3402–3409.
[8] J. Yang, J. Han, H. Yu, J. Yin, M. Gao, Z. Wang, X. Zeng, Role of molten pool mode on formability, microstructure and mechanical properties of selective laser melted Ti-6Al-4V alloy, Mater. Des. 110 (2016) 558–570.
[9] L.C. Zhang, J. Das, H.B. Lu, C. Duhamel, M. Calin, J. Eckert, High strength Ti – Fe – Sn ultrafine composites with large plasticity, Scripta Mater.57 (2007) 101–104.
[10] C. Zhang, H. Zhu, H. Liao, Y. Cheng, Z. Hu, X. Zeng, Effect of heat treatments on fatigue property of selective laser melting AlSi10Mg, Int. J. Fatigue. 116 (2018) 513–522.
[11] C.D. Rabadia, Y.J. Liu, S.F. Jawed, L. Wang, Y.H. Li, X.H. Zhang, T.B. Sercombe, H. Sun, L.C. Zhang, Improved deformation behavior in Ti-Zr-Fe-Mn alloys comprising the C14 type laves and β phases, Mater. Des. 160 (2018) 1059-1070.