The Effects of Al₃(Sc, Zr) Precipitation Phase on Selective Laser Melting Al-Mg-Sc-Zr alloy: Microstructure, Mechanical properties and Fatigue resistance

Liang-Yan Lee
Kai-Chieh Chang
Jun-Ren Zhao
Fei-Yi Hung ( fyhung@mail.ncku.edu.tw )
National Cheng Kung University

Research Article

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Abstract

In this study, an Al-Mg-Sc-Zr alloy fabricated through selective laser melting (SLM) was subjected to a single-stage heat-treatment process and a two-stage heat-treatment process to determine the effect of heat treatment on the tensile properties and fatigue properties of the alloy at room temperature and high temperatures. The results indicated that heat treatment caused the precipitation of Al$_3$(Sc, Zr), thus increasing the tensile strength. The dynamic strain aging of the SLM Al-Mg-Sc-Zr alloy disappeared as the tensile temperature increased. The alloy exhibited the highest tensile strength after it was subjected to the single-stage heat treatment at both room temperature and high temperatures owing to the precipitated phase distribution at the melting pool boundaries. However, fatigue resistance and high-temperature necking of the as-printed SLM Al-Mg-Sc-Zr alloy were problems that could not be resolved with the single-stage heat treatment. In the two-stage heat treatment, the precipitated phases exhibited a uniform distribution in the matrix, thereby reducing the high-temperature necking phenomenon. The two-stage heat treatment helped reduce the melting pool interface effect and strengthen the matrix, restricting the propagation of fatigue cracks and increasing the fatigue life of materials.

1. Introduction

Selective laser melting (SLM) has been used to fabricate various aluminum alloys, such as Al-Si, Al-Mg, and Al-Zn [1–5]. However, these alloys are unsuitable for high-temperature applications [6, 7]. Compared with nickel and titanium alloys, Al-Mg alloys have superior corrosion resistance properties [8–10]. Al-Mg alloys have been widely used in the automobile, shipbuilding, and aerospace industries [11]. According to the literature [11–14], the addition of Sc and Zr to an Al-Mg alloy forms an Al-Mg-Sc-Zr alloy in the Al matrix, which precipitates Al$_3$(Sc, Zr) after aging treatment. This improves the mechanical properties of the material. Another study found that that the Al$_3$(Sc, Zr) phase is similar to Al matrix, which increases the alloy strength and reduces grain coarsening [15]. This indicates that the Al-Mg-Sc-Zr alloy has potential for use in applications that require high-strength materials.

Many studies have been conducted on the heat treatment and mechanical properties of Al-Mg-Sc-Zr alloys [9, 10, 16–18]. Most of the heat treatments were performed in a single stage with the aim of increasing the strength of the Al-Mg-Sc-Zr alloy [17]. However, the single-stage heat-treatment process cannot homogenize SLM Al-Mg-Sc-Zr alloys. The melting pool interface in the matrix cannot effectively resist crack propagation. Therefore, we combined high-temperature solid solution heat treatment and medium-temperature aging treatment into a two-stage heat treatment process to obtain a homogeneous and highly fatigue-resistant material.

Studies on the heat treatment and mechanical properties of SLM Al-Mg-Sc-Zr alloys are still lacking. Research has shown that Al-Mg-Sc alloys produced through extrusion exhibit superplasticity at temperatures between 250 and 500°C [19]. However, no studies have investigated the use of SLM Al-Mg-Sc-Zr alloys in high-temperature applications. The interaction between the SLM Al-Mg-Sc-Zr alloy
precipitation of $\text{Al}_3(\text{Sc, Zr})$ and the melting pool affects the fatigue behavior of the material \cite{14,20,21}. Based on the above reasons, systematically investigating the high-temperature properties of SLM Al-Mg-Sc-Zr alloys and evaluating their fatigue characteristics are essential. In this study, we compared the microstructural characteristics of SLM Al-Mg-Sc-Zr alloys subjected to single-stage heat treatment and two-stage heat treatment and evaluated their tensile properties at room temperature and high temperatures. The fatigue resistance and failure mechanism are also discussed. The results obtained can help promote the use of SLM Al-Mg-Sc-Zr alloys in the automobile, shipbuilding, and aerospace industries.

2. Experimental Procedure

The SLM Al-Mg-Sc-Zr alloy used in this study was printed by ANJI Technology Co., Ltd. The printing parameters and chemical composition are listed in Table 1 and Table 2, respectively \cite{4,20}. The average size of the powder particles was 30 µm (Fig. 1). According to the results of X-ray diffraction (XRD), which was used to analyze the phase composition, the powder had no clear precipitation phase \cite{22}. The printing direction and the dimensions of the tensile and fatigue specimens are shown in Fig. 2(a), (b), and (c). Figure 2(d) shows the printed specimens.

| Laser Power | Scanning Speed | Beam Size | Hatch Space | Layer Thickness |
|-------------|----------------|-----------|-------------|----------------|
| 300 W       | 700 mm/s       | 35 µm     | 100 µm      | 30 µm          |

Table 2
SLM Al-Mg-Sc-Zr alloy powder composition.

| Element | Al   | Mg   | Sc   | Zr   | Mn   |
|---------|------|------|------|------|------|
| Wt.%    | Bal. | 4.50 ~ 5.10 | 0.68 ~ 0.88 | 0.21 ~ 0.52 | 0.30 ~ 0.81 |
| Element | Si   | Fe   | Ti   | O    | H    |
| Wt.%    | ≤ 0.40 | ≤ 0.40 | ≤ 0.15 | ≤ 0.05 | ≤ 0.01 |

In this study, as-printed refers to SLM Al-Mg-Sc-Zr alloys before heat treatment. The as-printed specimens were subjected to two heat-treatment processes: a single-stage aging treatment at 350°C for 6 h \cite{6,10,11} and a two-stage heat treatment (solution heat treatment at 500°C for 1 h followed by an aging treatment at 350°C for 6 h) \cite{23–25}. In the first stage of the two-stage heat treatment, the melting pool structure was partially decomposed, and the elements were uniformly dissolved into Al matrix \cite{24,25}. The heat-treatment parameters are listed in Table 3.
Table 3
Heat-treatment parameters of the Al-Mg-Sc-Zr alloy.

|   | As-printed                        | Selective Laser Melting as-printed |
|---|-----------------------------------|------------------------------------|
| A |                                   |                                    |
| B | 350°C-6 h                         | Air cooling                        |
| C | 500°C-6 h + 350°C-6 h             | Water quenching and Air cooling    |

The specimens were sequentially ground with #80 to #5000 SiC sandpaper and subsequently polished with 1- and 0.3-µm Al₂O₃ and 0.04-µm SiO₂, in sequence. Finally, etching was performed using a solution of 5 mL of HNO₃ + 3 mL of HCl + 2 mL of HF + 190 mL of H₂O. An optical microscope (OLYMPUS BX41M-LED, Tokyo, Japan) was used to evaluate the microstructure. XRD spectroscopy (Bruker AXS GmbH, Karlsruhe, Germany) was employed to analyze the phase composition and thus confirm the fracture mechanism.

HRF hardness measurement was conducted using a hardness machine (Mitutoyo AR-10). A universal testing machine (Hung HT-8336, Taichung, Taiwan) was used for testing the tensile strength of the alloys; the strain rate was 1 mm/min, meaning the initial strain rate was $8.33 \times 10^{-4}$ s⁻¹. The as-printed and heat-treated specimens were respectively stretched at room temperature and high temperatures in the range of 100–350°C [19].

Finally, a fatigue test was conducted using a rotating fatigue testing machine (HUNGTA HT-810, Taichung, Taiwan) with loadings of 7, 12, 17, and 22 kg (i.e., stress of 33.01, 56.59, 80.17, and 103.75 kg/mm²) [20]. The fatigue resistance of SLM Al-Mg-Sc-Zr alloys with different heat treatments were compared using a Stress-Number of cycles to failure curve (S-N curve); a stereomicroscope (OLYMPUS SZ61, Tokyo, Japan) and scanning electron microscope (HITACHI SU-5000, HITACHI, Tokyo, Japan) were used to inspect the fatigue fracture surface and determine the fracture mechanism.

3. Results And Discussion
3.1 Microstructure and Phase Analysis

Figures 3(a) and (b) display the microstructure of the as-printed SLM Al-Mg-Sc-Zr alloy, which exhibits a typical melting pool structure similar to that of other SLM Al alloy systems [1, 4, 20]. The width and depth of the SLM Al-Mg-Sc-Zr melting pool in this study was approximately 200 and 100 µm, respectively. Figures 3(c) and (d) present the microstructure of the specimens subjected to single-stage aging heat treatment at 350°C for 6 h. Clear melting pool structure was observed in the matrix, indicating that the single-stage heat treatment could not completely decompose the melting pool structure. Figures 3(e) and (f) show the microstructure of the specimens subjected to two-stage heat treatment at 500°C for 1 h and then at 350°C for 6 h. The melting pool structure was partially decomposed after the high-temperature solution treatment.
Figure 4 displays the XRD results of the SLM Al-Mg-Sc-Zr alloy before and after heat treatments. Double diffraction peaks corresponding to the (311) and (222) planes were observed in the as-printed specimen. According to other studies \cite{14,26}, double peaks are caused by the precipitation of Al$_3$(Sc, Zr), which results in residual heat during the SLM process. After heat treatments, the precipitation peak of Al$_3$(Sc, Zr) could be clearly observed, indicating that Al$_3$(Sc, Zr) precipitated either in a single stage or in two stages. Al$_3$(Sc, Zr) could be used as a strengthening phase to improve the mechanical properties of the alloy \cite{14}.

The XRD results revealed that the single-stage heat treatment significantly reduced the peak strength of the (111) and (200) planes, whereas this reduction in peak strength was less noticeable after the two-stage heat treatment process. The main reason for this finding is that the single-stage heat treatment belongs to medium-temperature aging, which not only reduces the residual stress, but also the incoherent precipitation behavior will result in the reduction of (111) and (200) planes. The two-stage heat-treatment process exhibited a high-temperature solid solution effect, and the precipitates were more uniform and finer. Hence, the diffraction surface effect could be reduced \cite{27}. Notably, the two-stage heat treatment decomposed the structure of the SLM Al-Mg-Sc-Zr alloy melting pool and produced Al$_3$Zr and Al$_3$(Sc, Zr) precipitates \cite{14,27−30}.

### 3.2 Mechanical Properties of the Alloy at Room Temperature

Figure 5 displays the HRF results. XRD showed the Al$_3$(Sc, Zr) peaks after the single-stage heat treatment. According to the literatures, Al$_3$(Sc, Zr) would precipitate at the melting pool boundary \cite{31,32}, thus increasing the hardness of the as-printed sample from HRF95 to HRF107. After the two-stage heat treatment, the melting pool decomposed and the Sc and Zr elements dissolved into the matrix. Simultaneously, the structure became equiaxed, and the strengthening behavior of Al$_3$(Sc, Zr) occurred, so the hardness did not change significantly.

Figures 6 (a) and (b) present the tensile curves of the SLM Al-Mg-Sc-Zr alloy at room temperature and the macroscopic fracture morphology of the samples, respectively. At room temperature, all three specimens exhibited jagged characteristics of dynamic strain aging (DSA) \cite{11,16}. The main reason for this is that Mg in the Al-Mg alloy forms a dislocation atmosphere, causing the stress and strain to be released in stages \cite{33,34}. After solution heat treatment, the solute atoms are dissolved into the matrix to improve the ductility \cite{35,36,37}. Figures 7 (a) and (b) show the tensile properties at room temperature (the values are listed in Table 4). The single-stage heat treatment is a medium-temperature aging process, which can make the Al$_3$ (Sc, Zr) strengthening phase precipitate at the boundary of the melting pool, exhibiting the highest strength but the lowest ductility \cite{14,20,27,31,32,38}. Furthermore, the tensile direction was closely related to the printing direction. The two-stage heat-treatment process had a high-temperature solid solution effect, which reduced the dependence of the tensile failure direction. Therefore, a better trade-off was achieved between tensile strength and ductility.
### Table 4
Average tensile properties and hardness of the Al-Mg-Sc-Zr alloy.

|    | YS (MPa) | UTS (MPa) | UE (%) | TE (%) | HRF |
|----|----------|-----------|--------|--------|-----|
| A  | 244      | 315       | 21.1   | 22.7   | 95  |
| B  | 356      | 384       | 9.6    | 11.7   | 107 |
| C  | 255      | 334       | 15.6   | 17.1   | 93  |

### 3.3 High-Temperature Mechanical Properties

Figure 8 displays the high-temperature tensile stress-strain curves of the three specimens at temperatures ranging from room temperature to 350°C. Figure 9 presents a photo of each specimen after tensile fracture. Table 5 lists the values of the tensile properties. All three specimens exhibited the highest strength when the temperature was 100°C; the strength decreased as the temperature increased beyond 100°C. According to the phase diagram \[39, 40\], the SLM Al-Mg-Sc-Zr alloy started to precipitate at 100°C, increasing the strength. When the temperature exceeded 150°C, the significant thermal diffusion of solute atoms and activated sliding surfaces reduced the strength. The ductility of the three specimens increased with tensile temperature. Additionally, the serrated jitter of the stress-strain curves smoothened as the tensile temperature increased. This is because the high temperature provided kinetic energy for the movements of dislocation and diffusion on solute atoms, which made the material more plastic \[19\].
Table 5
Average high-temperature tensile properties and hardness of the SLM Al-Mg-Sc-Zr alloy.

| Temperature   | YS (MPa) | UTS (MPa) | UE (%) | TE (%) |
|---------------|----------|-----------|--------|--------|
| A             | Room temperature | 244 | 315 | 21.1 | 22.7 |
|               | 100°C     | 269 | 332 | 16.1 | 18.1 |
|               | 150°C     | 244 | 285 | 16.9 | 21.1 |
|               | 200°C     | 230 | 237 | 1.1  | 20.7 |
|               | 250°C     | 208 | 219 | 2.2  | 13.0 |
|               | 300°C     | 142 | 169 | 2.7  | 17.0 |
|               | 350°C     | 70  | 75  | 0.9  | 15.1 |
| B             | Room temperature | 356 | 384 | 9.6  | 11.8 |
|               | 100°C     | 385 | 414 | 11.8 | 13.7 |
|               | 150°C     | 331 | 351 | 1.1  | 15.5 |
|               | 200°C     | 318 | 324 | 0.6  | 13.8 |
|               | 250°C     | 156 | 167 | 1.3  | 11.7 |
|               | 300°C     | 151 | 164 | 1.7  | 15.8 |
|               | 350°C     | 65  | 72  | 1.5  | 30.8 |
| C             | Room temperature | 255 | 334 | 15.6 | 17.1 |
|               | 100°C     | 268 | 352 | 16.1 | 18.1 |
|               | 150°C     | 185 | 266 | 17.1 | 19.5 |
|               | 200°C     | 160 | 183 | 9.6  | 21.5 |
|               | 250°C     | 130 | 137 | 4.1  | 22.0 |
|               | 300°C     | 73  | 83  | 2.1  | 23.4 |
|               | 350°C     | 16  | 19  | 1.2  | 15.2 |

Figure 10, Fig. 11, and Fig. 12 show the high-temperature tensile mechanical properties of the as-printed, single-stage heat-treated, and two-stage heat-treated specimens, respectively. The results indicate that the strength of the single-stage heat-treated specimen was higher than that of the as-printed specimen and the two-stage heat-treated specimen in the tensile temperature range of 100–350°C [41,42]. A comparison revealed that at 250°C, the as-printed specimen was brittle (low ductility), and the strength of the single-stage heat-treated specimen was significantly reduced. Notably, the elongation of the two-stage heat-
treated specimen increased significantly at 200°C, thus confirming that the maximum temperature that can be applied to the SLM Al-Mg-Sc-Zr alloy is approximately 200°C\textsuperscript{[43,44]}. Because the SLM Al-Mg-Sc-Zr alloy exists a significant texture effect (the existence of a melting pool structure), using only tensile results to evaluate the material properties was inadequate. Therefore, a rotation fatigue test was performed to further clarify the relationship between material texture and the failure mechanism.

### 3.4 Rotation Fatigue Characteristics

Figure 13 shows the fatigue S-N curves of the three specimens, and Table 6 lists the fatigue resistance values of all specimens. The fatigue resistance of the two-stage heat-treatment specimen was higher than that of the other two specimens. Rotation fatigue breakage occurred toward the right side of all specimens (Fig. 14) because the motor torque output was on that side. The macro morphology of the fatigue fracture of the specimens is displayed in Fig. 15. The fatigue fracture zone can be observed, including the initial fracture zone, fatigue zone, and final fracture zone\textsuperscript{[45,46]}. The three zones of the as-printed specimen were similar in area, and there were many brittle fracture characteristics at the melting pool boundary. The area of the final fracture zone in the single-stage heat-treated specimen was the smallest. The two-stage heat-treated specimen exhibited multiple brittle fracture characteristics in the initial fracture zone and the large final fracture zone. Figure 16 shows the microscopic characteristics of the fatigue fracture surface. The three samples all exhibited uneven and dimple-like fractures. This fatigue fracture was inferred to be due to a ductile fracture mechanism\textsuperscript{[47,48]}.
Table 6
Fatigue resistance of the SLM Al-Mg-Sc-Zr alloy.

| Load (kg) | Stress (kg/mm²) | N (Average Number of Cycles to Failure) |
|-----------|-----------------|--------------------------------------|
| A (As-printed)                                      |
| 7         | 33.0            | 35,129                               |
| 12        | 56.6            | 22,861                               |
| 17        | 80.2            | 10,373                               |
| 22        | 103.7           | 8,005                                |
| B (350°C-6 h)                                      |
| 7         | 33.0            | 31,272                               |
| 12        | 56.6            | 20,241                               |
| 17        | 80.2            | 15,696                               |
| 22        | 103.7           | 11,288                               |
| C (500°C-1 h + 350°C-6 h)                           |
| 7         | 33.0            | 42,455                               |
| 12        | 56.6            | 30,353                               |
| 17        | 80.2            | 26,785                               |
| 22        | 103.7           | 21,303                               |

After the single-stage heat treatment, the precipitation strengthening phase combined with the melting pool structure improved the tensile mechanical properties and heat strength of the as-printed specimen. By contrast, two-stage heat treatment had three characteristics to delay the propagation of fatigue cracks and increase fatigue resistance: (1) The melting pool interface decomposition effect (dissolved back to the Al matrix) avoid the intergranular fracture behavior, (2) Al matrix uniform precipitation strengthening mechanism, and (3) Reduction of SLM materials texture effect. Figure 17 presents a schematic of the metallurgical structure of the three specimens in the study. Through the tensile test and fatigue test in this research, the mechanical properties and strain failure direction of the SLM Al-Mg-Sc-Zr alloy were objectively evaluated. Finally, it was concluded that the two-stage heat treatment process has better industrial applications.

4. Conclusions

1. SLM Al-Mg-Sc-Zr alloy undergoes single-stage heat treatment, the precipitates at the boundary of the melting pool help to improve the alloy’s tensile mechanical properties. A two-stage heat-treatment
process decomposes the boundary of the melting pool and promotes uniform precipitation, thereby increasing the ductility of the material.

2. As the tensile temperature increases, the DSA of each specimen disappears. The maximum temperature that can be applied to the SLM Al-Mg-Sc-Zr alloy is approximately 200°C.

3. After two-stage heat treatment, SLM Al-Mg-Sc-Zr alloy decomposes the melting pool interface and exhibits a uniform precipitation mechanism, thus inhibiting the propagation of fatigue cracks and increasing fatigue resistance.

Declarations

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-Consent to Participate: All data generated or analyzed during this study are included in this published article.

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Figures

Figure 1

SEM and XRD analysis results of the SLM Al-Mg-Sc-Zr alloy powders.
Figure 2

Schematic of the specimen of the SLM Al-Mg-Sc-Zr alloy: (a) manufacturing direction, (b) tensile specimen, (c) fatigue specimen, and (d) macroscopic morphology photographs.
Figure 3

3D microstructure of the SLM Al-Mg-Sc-Zr alloy: (a) and (b) as-printed, (c) and (d) heat treated at 350 °C for 6 h, and (e) and (f) heat treated at 500 °C for 1 h and at 350 °C for 6 h.
Figure 4

(a) X-ray diffraction pattern of the SLM Al-Mg-Sc-Zr alloy, obtained over a wide range of 2θ values. (b) X-ray diffraction pattern in the vicinity of the peak of α-Al (2θ = 38.3°).
Figure 5

Hardness of the SLM Al-Mg-Sc-Zr alloy.
Figure 6

Room temperature tensile test results of the SLM Al-Mg-Sc-Zr alloy: (a) stress-strain curve and (b) macro view of the fracture specimen.
Figure 7

Tensile properties of the SLM Al-Mg-Sc-Zr alloy: (a) strength and (b) ductility.
Figure 8

High-temperature tensile stress-strain curves of the SLM Al-Mg-Sc-Zr alloy: (a) as-printed, (b) heat treated at 350 °C for 6 h, and (c) heat treated at 500 °C for 1 h and at 350 °C for 6 h.
Figure 9

High-temperature tensile test macro morphology of the fracture specimen of the SLM Al-Mg-Sc-Zr alloy: (a) as-printed, (b) heat treated at 350 °C for 6 h, and (c) heat treated at 500 °C for 1 h and at 350 °C for 6 h.
Figure 10

High-temperature tensile properties of as-printed specimen: (a) strength and (b) ductility.
Figure 11

High-temperature tensile properties of the specimen subjected to heat treatment at 350 °C for 6 h: (a) strength and (b) ductility.
Figure 12

High-temperature tensile properties of the specimen subjected to heat treatment at 500 °C for 1 h and at 350 °C for 6 h: (a) strength and (b) ductility.
Figure 13

S-N curve of the SLM Al-Mg-Sc-Zr alloy.
Figure 14

Macro morphology of fatigue fracture of the SLM Al-Mg-Sc-Zr: (a) as-printed, (b) heat treated at 350 °C for 6 h, (c) and heat treated at 500 °C for 1 h and at 350 °C for 6 h.
Macro morphology of the fatigue fracture of the SLM Al-Mg-Sc-Zr alloy under a 7-kg load: (a) as-printed, (b) heat treated at 350 °C for 6 h, and (c) heat treated at 500 °C for 1 h and at 350 °C for 6 h.
Figure 16

Microstructure of the fatigue fracture of the SLM Al-Mg-Sc-Zr under a 7-kg load: propagation region: (a) as-printed, (b) heat treated at 350 °C for 6 h, and (c) heat treated at 500 °C for 1 h and at 350 °C for 6 h.
Figure 17

Schematic of the SLM Al-Mg-Sc-Zr alloy precipitation distribution.