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A Novel Two-Stage Heat Treatment with Medium-Temperature Aging Influence on Microstructure, Al₃(Sc, Zr) Nanoprecipitation, and Application Properties, Enhancing Selective Laser Melting of Al–Mg–Sc–Zr Alloy

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Abstract: Al–Mg–Sc–Zr alloy fabricated through selective laser melting (SLM) is an additive manufacturing alloy with promising industrial potential. In this study, as-printed specimens were subjected to either single-stage or two-stage heat treatment processes to investigate the effect of temperature from room temperature to high temperature on the specimens' tensile and fatigue properties to establish a reliable reference for aerospace applications. The tensile test results indicated that the heat treatment contributed to determine the properties of the nanoprecipitate Al₃(Sc, Zr) with a strengthening phase, improving tensile strength. Moreover, the dynamics strain aging (DSA) effect vanished as temperature increased. It is noteworthy that the nanoprecipitation was distributed at the boundary of the melting pool after single-stage heat treatment with the highest tensile properties in all tests. In addition, the microstructure observed after the two-stage heat treatment indicated a melting pool interface decomposition, and the nanoprecipitation was homogeneously scattered over the Al matrix, increasing strength and further delaying fatigue crack transmission. Those features build a high-fatigue-resistance foundation. TEM analysis also confirmed the promotion of Sc thermal diffusion and an Al₃(Sc, Zr) precipitation transformation mechanism under two-stage heat treatment, corresponding to aforementioned inferences. The SLM Al–Mg–Sc–Zr alloy with two-stage heat treatment brings about balance between tensile properties and fatigue resistance, providing new insight into additive manufacturing with Al alloys.

Keywords: Al–Mg–Sc–Zr; selective laser melting (SLM); heat treatment; medium-temperature aging; high-temperature tensile; rotation fatigue

1. Introduction

Considering high corrosion-resistance and ductility, Al–Mg alloys have been widely utilized in the automobile, shipbuilding, and aerospace industries [1–5]. According to the literature [6–9], combining Sc and Zr elements with Al–Mg alloys to form Al–Mg–Sc–Zr alloys can precipitate Al₃(Sc, Zr) after heat treatment, thereby improving mechanical properties. Previous study has revealed that the Al₃(Sc, Zr) precipitation's crystal structure is similar to α-Al, which can increase matrix strength and reduce grain coarseness [10]. These characteristics set Al–Mg–Sc–Zr alloys as high-strength application potential material.

To date, many researchers have mentioned the heat treatment and the mechanical properties of SLM Al–Mg–Sc–Zr alloys [2,3,8,11,12], and the single-stage heat treatment process SLM Al–Mg–Sc–Zr alloy is subjected to causes it to gain strength but lose ductility [12]. It is a remarkable fact that the single-stage heat treatment process neither homogenizes SLM Al–Mg–Sc–Zr alloys, nor does the residual melting pool interface resist crack propagation effectively. In view of these disadvantages in the single-stage heat treatment process, this study introduces heat treatment comprised of solution treatment and medium-temperature...
aging, which form a two-stage heat treatment process, achieving homogeneous and high-fatigue-resistance materials.

SLM technology has widely been introduced to fabricate various aluminum alloys, such as Al–Si, Al–Mg, and Al–Zn alloys [13–17]; however, these alloys are not characterized by high-temperature applications [18,19]. In terms of high-temperature applications, the literature has pointed out the traditional extrusion Al–Mg–Sc alloys with excellent superplasticity in the range of 250-500 °C [20]. Even now, investigation through heat treatment and high-temperature mechanical properties of SLM Al–Mg–Sc–Zr alloys is still lacking; in addition, the interaction of Al3(Sc, Zr) precipitates and the melting pool structure, which affects fatigue resistance, demands further research [9,21,22]. Taking the above reasons into account, systematically investigating high-temperature properties and evaluating their fatigue characteristics are essential. This study compares the microstructure and precipitation of SLM Al–Mg–Sc–Zr alloy as-printed specimens, single-stage-heat-treated specimens, and two-stage-heat-treated specimens to evaluate the mechanical properties at room and high temperatures (RT-350 °C) as well as clarify fatigue life and failure mechanism. Notably, TEM analysis is applied to confirm the phase transformation mechanism resulting from two-stage heat treatment of the SLM Al–Mg–Sc–Zr alloy. The relevant results include significant discoveries, providing reference for the application of SLM Al–Mg–Sc–Zr alloy in the automobile, shipbuilding, and aerospace industries.

2. Experimental Procedure

The SLM Al–Mg–Sc–Zr alloys in this study are fabricated by ANJI Technology Co., Ltd (Tainan, Taiwan). The printing parameters and chemical composition are listed in Tables 1 and 2, respectively [16,21]. The average size of the prealloyed powder is 30 µm (Figure 1). According to the results of the X-ray diffraction (XRD) used to analyze the phase composition, the powder has no clear precipitation phase [23]. The appearance, printing direction, and the dimensions of the tensile and fatigue specimens are shown in Figure 2a–c.

In this study, as-printed referred to SLM Al–Mg–Sc–Zr alloy before heat treatment. The as-printed specimens were subjected to single-stage heat treatment at 350 °C for 6 h [3,6,18] and the two-stage heat treatment: solid solution heat treatment at 500 °C for 1 h and aging treatment at 350 °C for 6 h [24–26]. The heat treatment parameters are listed in Table 3.

![Figure 1. SEM and XRD analysis of the Al–Mg–Sc–Zr powders.](image-url)
Table 1. Parameters of the SLM process.

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

Table 2. Al–Mg–Sc–Zr 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 |

Figure 2. Schematic of the specimen of the SLM Al–Mg–Sc–Zr alloy: (a) macroscopic morphology, (b) manufacturing direction, and (c) size of tensile specimen and fatigue specimen.

Table 3. Heat treatment parameter of SLM Al–Mg–Sc–Zr alloys.

| Specimen ID | Specimen Type                  | Heat Treatment                                      |
|-------------|--------------------------------|------------------------------------------------------|
| A           | as-printed                     | None                                                 |
| B           | single-stage heat treatment    | 350 °C for 6 h/air cooling                           |
| C           | two-stage heat treatment       | 500 °C for 1 h/water quenching + 350 °C for 6 h/air cooling |

These specimens were ground through #80 to #4000 SiC sandpaper in sequence and polished using 1 µm, 0.3 µm Al₂O₃, and 0.04 µm SiO₂ to polish. Finally, etching was performed with a solution of 5 mL of HNO₃ + 3 mL of HCl + 2 mL of HF + 190 mL of H₂O. Optical microscope (OM, OLYMPUS BX41M-LED, Tokyo, Japan) and XRD spectroscopy (Bruker AXS GmbH, Karlsruhe, Germany) were employed to analyze the phase composition.

HRF hardness measurement was conducted by using a hardness machine (Mitutoyo AR-10, Kanagawa, Japan). Universal testing machine (HUNGTA, HT-8336, Taichung, Taiwan) was used for tensile testing the strength with a strain rate of 1 mm/min and an initial strain rate of 1.83 × 10⁻³ s⁻¹ at both room temperature and high temperatures in the range of 100–350 °C [20].

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) established...
fatigue properties [21]. In addition, fatigue resistance was compared using a Stress–Number of cycles to failure curve (S-N curve). A scanning electron microscope (HITACHI SU-5000, HITACHI, Tokyo, Japan) inspected the fatigue fracture surface and explored the fracture mechanism. An electron probe microanalyzer (EPMA, JEOL JXA-8900R, Taipei, Taiwan) was used to compare the distribution of the alloying elements between as-printed and two-stage-heat-treated specimens. Finally, a transmission electron microscope (JEM-2010-200 KV, JEOL Ltd., Tokyo, Japan) with an energy dispersive spectrometer (EDS) was further applied to clarify the thermal diffusion mechanism of elements in the two-stage heat treatment process.

3. Results and Discussion

3.1. Microstructure and Phase Analysis

Figure 3a,b displays the microstructure of as-printed SLM Al–Mg–Sc–Zr alloy, which exhibited typical melting pool structure similar to that of other SLM Al alloys [13,16,21]. The width and depth of the melting pool were approximately 200 and 100 µm, respectively. Figure 3c,d presents the microstructure of the single-stage-heat-treated specimens with obvious melting pool structure the same as what the as-printed specimens showed, which indicated that the process could not completely decompose the melting pool structure. Figure 3e,f shows the microstructure of the specimens subjected to two-stage heat treatment. Observed by 3D metallographic diagram, the melting pool structure partially decomposed after high-temperature solid solution treatment was confirmed.

XRD results are shown in Figure 4a for all specimens. Compared with the powder XRD results (Figure 1), double diffraction peaks corresponding to the (311) and (222) crystal planes were observed (Figure 4b). According to the literature [9,27], the double peaks represent precipitation of Al3(Sc, Zr) resulting from residual heat throughout the SLM process, and the peaks of Al3(Sc, Zr) were clearly divided after heat treatments. This indicates that Al3(Sc, Zr), serving as a strengthening phase, precipitated either in single-stage or in two-stage heat treatment to improve the mechanical properties of alloys [9].

3.2. Mechanical Properties at Room Temperature

Figure 5a compares the hardness (HRF) results of all specimens. After the single-stage heat treatment, Al3(Sc, Zr) precipitated at the melting pool boundary, increasing the hardness of SLM Al–Mg–Sc–Zr alloys from HRF95 to HRF107. On the other hand, the melting pool was decomposed and the Sc and Zr elements were dissolved into the α-Al matrix after the two-stage heat treatment. Simultaneously, a precipitation process involving Al3(Sc, Zr)-strengthening precipitation occurred, producing strength and bringing about the observed slight change in hardness.

Figure 5b presents the tensile curves of the SLM Al–Mg–Sc–Zr alloys. All specimens exhibited jagged characteristics of dynamic strain aging (DSA) at room temperature [6,11]. The principle of the DSA phenomenon was the dissolved Mg in Al–Mg–Sc–Zr alloys forming a dislocation atmosphere, causing the stress and strain to be released in stages [28,29]. After solid solution treatment, more Mg element dissolved into the α-Al matrix, promoting the more significant jitter phenomenon and ductility improvement (Figure 5c,d, data lists in Table 4) [29–31]. The single-stage-heat-treated specimens revealed the highest strength but the lowest ductility due to the presence of grain boundaries, of melting pool boundaries, and of Al3(Sc, Zr) precipitation under medium-temperature aging treatment. Therefore, the strength increased, but the ductility could not meet standards of industrial application, and the tensile strength was limited in the printing direction [9,21]. Notably, the two-stage heat treatment process with solid solution effect contributes to the decomposition of melting pool boundaries, alloying element homogeneous dissolution, and reduction in the dependence of the tensile failure direction [25,26]. Thus, the two-stage-heat-treated specimens exhibited a better combination of tensile strength and ductility.
Figure 3. Three-dimensional microstructure of the SLM Al–Mg–Sc–Zr alloys: (a,b) as-printed, (c,d) single-stage heat-treated, (e,f) two-stage heat-treated specimens.

Figure 4. (a) X-ray diffraction pattern of the SLM Al–Mg–Sc–Zr alloys, obtained over a wide range of 2θ values. (b) X-ray diffraction pattern in the vicinity of the peak of α-Al (2θ = 82°).

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Figure 5. (a) Hardness, (b) tensile stress–strain curve, (c) tensile strength, and (d) elongation of the SLM Al–Mg–Sc–Zr alloys at room temperature (YS: yield strength, UTS: ultimate tensile strength, UE: uniform elongation, and TE: total elongation).

Table 4. Average tensile properties and hardness of SLM Al–Mg–Sc–Zr alloys.

|   | 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

The high-temperature tensile stress–strain curves at temperatures ranging from room temperature to 350 °C are shown in Figure 6 (data lists in Table 5). All specimens exhibited the highest strength while at 100 °C, decreasing beyond 100 °C. According to the literature [32,33], DSA often induces the characteristics of the Lüders band of uneven plastic deformation. This feature caused internal crack connection, resulting in decreased strength. After DSA was eliminated at 100 °C, SLM Al–Mg–Sc–Zr alloy had the best strength performance. In addition, the serrated jitter in stress–strain curves smoothened owing to sufficient kinetic energy for the dislocation movement and diffusion of solute atoms served by high temperature, which has a tendency toward the plastic deformation mechanism [20,34].

Figure 7 shows the high-temperature tensile mechanical properties of three specimens. The single-stage-heat-treated specimens exhibited the highest strength in the range of 100–350 °C [34,35]; however, a dramatic decrease in strength at 250 °C was observed. In addition, the as-printed specimens also revealed a brittle effect (low ductility) at 250 °C. Despite the two-stage-heat-treated specimens’ increasing elongation after 250 °C, their strength is insufficient for industrial applications; therefore, it could be confirmed that the upper limit of the high-temperature applicability of SLM Al–Mg–Sc–Zr alloys is approximately 200 °C [36,37].

The SLM Al alloys were proven to possess significant melting pool texture effects in the previous study, so it is inadequate to evaluate their material properties merely by the
tensile results [21]. Hence, the rotation fatigue test was performed in order to further clarify the relationship between texture effect and failure mechanism.

Figure 6. High-temperature tensile stress–strain curve of the SLM Al–Mg–Sc–Zr alloys: (a) as-printed and (b) single-stage- and (c) two-stage-heat-treated specimens.
Table 5. Average high-temperature tensile properties and hardness of SLM Al–Mg–Sc–Zr alloys.

| 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   |

3.4. Rotation Fatigue Characteristics

Figure 8 presents the rotation fatigue S–N curves (data lists in Table 6). The two-stage heat treatment specimen shows more promising fatigue resistance than the other two specimens. On the basis of macro fatigue fracture morphology (Figure 9), the fatigue-failure section is apparently divided into three zones: initial fracture zone, crack transmission zone, and final fracture zone [38,39]. The three zones of the as-printed specimens are similar in area, while a number of cracks are found on the surface morphology, suggesting brittle fracture characteristics. Compared to the other specimens, the single-stage-heat-treated specimen exhibited the smallest final fracture zone in area; on the other hand, the two-stage-heat-treated specimen showed unapparent brittle fracture characteristics and the largest final fracture zone in area. Figure 10 displays the microscopic morphology of the fatigue fracture surface, with all exhibiting uneven and dimple-like fractures. According to these characteristics, the fatigue mechanism can be inferred as a ductile fracture mechanism [40,41].

3.5. Thermal Diffusion and Strengthening Effect during Two-Stage Heat Treatment

Figure 11a,b demonstrate the EPMA results of as-printed and two-stage-heat-treated specimens, respectively. As observed from the element mapping, the two-stage-heat-treated specimen has an influence on the melting pool structure decomposition and Al–Mg particle formation. In the as-printed melting pool structure, Mg element presents an ununiformly arc-shaped distribution, and the distribution transforms into Mg-rich fine particles homogeneously scattered in Al matrix after two-stage heat treatment. This phenomenon contributes to the improvement of fatigue resistance of the two-stage-heat-treated specimens. Figure 11c offers a schematic presenting the two-stage-heat-treated specimens’ microstructure, with α-Al matrix, Al–Mg particles and Al3Sc precipitation visibly shown in the bright-field TEM image [14].
Figure 7. Hightemperature tensile properties of as-printed specimens’ (a) strength and (b) ductility, single-stage-heat-treated specimens’ (c) strength and (d) ductility, and two-stage-heat-treated specimens’ (e) strength and (f) ductility.

Figure 8. S–N curve of the SLM Al–Mg–Sc–Zr alloys. 
... SLM Al–Mg–Sc–Zr alloys under 7 kg load: (a) as-printed and (b) single-stage- and (c) two-stage-heat-treated specimens.

Figure 8. S–N curve of the SLM Al–Mg–Sc–Zr alloys.
Table 6. Fatigue resistance of SLM Al–Mg–Sc–Zr alloys.

| Load (kg) | Stress (kg/mm²) | N (Average Number of Cycles to Failure) |
|-----------|-----------------|----------------------------------------|
|           |                 | A (as-printed)                         |
| 7         | 33.0            | 24,714                                  |
| 12        | 56.6            | 17,571                                  |
| 17        | 80.2            | 10,373                                  |
| 22        | 103.7           | 8005                                    |
|           |                 | B (single-stage)                       |
| 7         | 33.0            | 31,272                                  |
| 12        | 56.6            | 20,241                                  |
| 17        | 80.2            | 15,696                                  |
| 22        | 103.7           | 11,288                                  |
|           |                 | C (two-stage)                          |
| 7         | 33.0            | 42,455                                  |
| 12        | 56.6            | 30,353                                  |
| 17        | 80.2            | 26,785                                  |
| 22        | 103.7           | 21,303                                  |

Figure 8. S–N curve of the SLM Al–Mg–Sc–Zr alloys.

Figure 9. Macrostructure of fatigue fracture of the SLM Al–Mg–Sc–Zr alloys under 7 kg load: (a) as-printed and (b) single-stage- and (c) two-stage-heat-treated specimens.

Figure 10. Microstructure of fatigue fracture of the SLM Al–Mg–Sc–Zr alloys under 7 kg load propagation region: (a) as-printed and (c) single-stage- and (e) two-stage-heat-treated specimens; final fracture region: (b) as-printed and (d) single-stage- and (f) two-stage-heat-treated specimens.
3.5. Thermal Diffusion and Strengthening Effect during Two-Stage Heat Treatment of the two-stage-heat-treated specimens.

![Figure 10](image-url)

**Figure 10.** Microstructure of fatigue fracture of the SLM Al–Mg–Sc–Zr alloys under 7 kg load propagation region: (a) as-printed and (e) single-stage- and (g) two-stage-heat-treated specimens; final fracture region: (b) as-printed and (d) single-stage- and (f) two-stage-heat-treated specimens.

![Figure 11](image-url)

**Figure 11.** (a) EPMA image (Al and Mg elements) of the as-printed specimen; (b) EPMA image (Al and Mg elements) of the two-stage-heat-treated specimen; (c) schematic diagram and TEM image of the two-stage-heat-treated specimens.
Figure 12 provides the results of TEM equipped with EDS mapping microanalysis on the interface between $\alpha$-Al matrix and Al–Mg particles, confirming that Mg had homogeneous distribution, but Sc was distributed inside the grains and Zr was distributed in the grain boundary. According to the results of the EDS point analysis and SAED pattern in Figure 13, Al$_3$Sc precipitation exhibits the highest Sc content, followed by $\alpha$-Al matrix, and Al–Mg particles are the least. The most notable point is the observation of Sc solid dissolved in the Al-matrix diffraction pattern (yellow circle in Figure 13 SAED pattern). It is certain that Sc atoms diffused from Al$_3$Sc precipitation under two-stage heat treatment contribute to crystal lattice deformation of the $\alpha$-Al matrix, resulting in a solid solution strengthening effect (schematic as shown in Figure 14) [42,43]. After thermal diffusion, the concentration of Sc has the tendency to achieve equilibrium, and Al$_3$Sc precipitation shrink [43,44].

Figure 12. TEM image and EDS mapping of two-stage-heat-treated specimen.
Figure 13. TEM data of two-stage-heat-treated matrix (point (1): Al–Mg particle; point (2): α-Al matrix; point (3): Al3Sc precipitation phase).

Figure 14. Schematic diagram of atomic diffusion in two-stage-heat-treated SLM Al–Mg–Sc–Zr alloys.
4. Conclusions

1. After single-stage heat treatment, $\text{Al}_3(\text{Sc}, \text{Zr})$ precipitated at the boundaries with a residual melting pool texture effect increasing its strength but decreasing its ductility. The combination of single-stage heat treatment and solid solution treatment (two-stage heat treatment) decomposes the melting pool structure and induces a homogeneous precipitation, thereby apparently increasing the fatigue resistance.

2. As the tensile temperature increased, the DSA effect of each specimen decreased. At $100^\circ\text{C}$, where the DSA effect decreased, SLM Al–Mg–Sc–Zr alloy exhibited the highest high-temperature strength, and the upper limit for high-temperature applications was approximately $200^\circ\text{C}$.

3. After the two-stage heat treatment process, the melting pool boundaries of SLM Al–Mg–Sc–Zr alloys decomposed and precipitated homogeneously. The Sc strengthening mechanism was composed of $\text{Al}_3\text{Sc}$ precipitation and Sc solid solution after thermal diffusion under heat treatment, increasing matrix strength and inhibiting fatigue crack propagation to provide high fatigue resistance.

4. Overall, the two-stage-heat-treated SLM Al–Mg–Sc–Zr alloy shows better mechanical tensile properties and fatigue resistance, providing wide applicability as an additive manufacturing Al alloy.

Author Contributions: Methodology, L.-Y.L.; investigation, L.-Y.L.; data curation, L.-Y.L.; writing—original draft preparation, L.-Y.L.; writing—review and editing, K.-C.C., J.-R.Z. and F.-Y.H.; supervision, J.-R.Z. and F.-Y.H. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: All data generated or analyzed during this study are included in this published article.

Data Availability Statement: The data presented in this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethics.

Acknowledgments: The authors are grateful to the Instrument Center of National Cheng Kung University and the Ministry of Science and Technology of Taiwan (Grant No. MOST 108-2221-E-006-140-MY3) for their financial support. They thank the Taiwan Circle Metal Powder Co., Ltd. and Taiwan ANJI Technology Co., Ltd. for providing the alloy powder and SLM printer, respectively.

Conflicts of Interest: The authors declare that they have no competing interests.

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