# Customized Heat Treatment Process Enabled Excellent Mechanical Properties in Wire Arc Additively Manufactured Mg-RE-Zn-Zr Alloys

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Abstract: Customized heat treatment is essential for enhancing the mechanical properties of additively manufactured metallic materials, especially for the alloys with complex phase constituents and heterogenous microstructure. However, the interrelated evolutions of different microstructure features make it difficult to establish optimal heat treatment processes. Herein, we proposed a method for customized heat treatment process exploration and establishment to overcome this challenge for such kind of alloys, and a wire arc additively manufactured (WAAM) Mg-Gd-Y-Zn-Zr alloy with layered heterostructure was used for feasibility verification. Through this method, an optimal microstructure (fine grain, controllable amount of LPSO structure and nano-scale  $\beta'$ precipitates) and the corresponding customized heat treatment process (520 °C/ 30 min + 200 °C/ 48 h) were obtained to achieve a combination of a high strength of 364 MPa and a considerable elongation of 6.2 %, which surpassed those of other state-of-the-art WAAM-processed Mg alloys. Furthermore, we evidenced that the favorable effect of the undeformed LPSO structure on the mechanical properties was emphasized only when the nano-scale  $\beta'$ precipitates were present. It is believed that the findings promote the application of magnesium alloy workpieces and help to establish customized heat treatment process for additively manufactured materials.

**Keywords:** wire arc additive manufacturing; heat treatment; Mg-RE-Zn-Zr alloys; LPSO structure; mechanical properties

## **1** Introduction

The multiple thermal cycles and rapid cooling behavior of additive manufacturing process make the microstructure different from the traditional components, so it is difficult to optimize through the traditional heat treatments to fully tap its mechanical properties potential. The exploration and establishment of customized heat treatment is one of the current research hotspots in the field of additive manufacturing. In addition, the evolution of complex phase characteristics of Mg-RE-Zn alloy during heat treatment is interrelated. For example, the dissolution of grain boundary eutectic phase (detrimental phase) is usually accompanied by grain growth, and the formation of LPSO structure also affects the subsequent  $\beta'$  enhanced phase precipitation. In view of the above problems, this work presents a method to explore and establish a custom heat treatment of additive manufacturing metal materials. Through the design and comparison of the microstructure of different solution treatments, the evolution was clarified, the strengthening and toughening mechanism was explored, and the optimal heat treatment process for additive manufacturing of magnesium alloys was finally determined.

#### 2 Experimental procedure

We employed two criteria to preliminary screen heat treatment processes to prevent the time waste caused by blind heat treatment process optimization. Firstly, the grain-boundary eutectic phase should be eliminated, since it not only occupies the strengthening element and affects the following precipitation of strengthening phase, but also becomes the crack origin site during bear loading process and causes premature failure. Secondly, the grain size should remain almost unchanged after heat treatment. Based on the above two principles and our preliminary experiments, the solution heat treatment processes utilized in this work were set as follow: 460 °C/ 1 h, 490 °C/ 1 h, 520 °C/ 30 min, 520 °C/ 1 h, 520 °C/ 8 h and 580 °C/ 15 min. For the solid solution heat treatment and aging treatment, the samples were put in the quartz tubes and oil bath and then quenched in water.

## 3 Result and discussion

The detailed phase evolution with temperature and time of heat treatment is shown in Fig. 1. The (Mg, Zn)<sub>3</sub>(Gd, Y) phase (yellow arrows) was gradually dissolved and transformed into the block-like LPSO structure (blue arrows) on the grain boundary with increasing the duration at 520 °C. With further prolonging the duration, Fig. 1d shows the transformation of the bulk LPSO structure into the lamellar LPSO structure (green arrow). Some reports thought the block-like LPSO structure is the 14H-type <sup>[1]</sup>. The TEM test results further evidenced the above statement by presenting both two type LPSO structures in sample after 520 °C for 1 h.



As the duration was extended from 1 h to 4 h, some lath LPSO structures (orange arrows) were formed long the grain boundary. Previous investigations demonstrated that the lath LPSO structure, also known as the X phase, was easily formed at temperatures over 500 °C. With further increasing the duration to 8 h, more X phase was obviously formed in the microstructure.

Under the same duration, the increase in heat treatment temperature will also have an important effect on the phase constituent evolution. High temperatures can effectively reduce the content of the (Mg, Zn)3(Gd, Y) phase, while the LPSO structures rarely precipitate from the microstructure.



Fig. 1 The OM characterization of the solution treated samples: (a) (520 °C/ 5 min); (b) (520 °C/ 15 min); (c) (520 °C/ 45 min); (d) (520 °C/ 1 h); (e) (520 °C/ 4 h); (f) (520 °C/ 8 h); (g) (540 °C/ 15 min); (h) (560 °C/ 15 min); (i) (580 °C/ 15 min); (j, k) STEM images of the LPSO structures and (I) SAED pattern of 14H-LPSO structure in (520 °C/ 1 h) sample.

Three kinds of heat-treated samples (460 °C/ 1 h, 490 °C/ 1 h and 520 °C/ 1 h) with different combinations of eutectic phase and LPSO structures were selected to determine the peak aging time at 200 °C. It is evident from Fig. 2a that the difference in phase constituent did not influence the peak aging time, which remained 48 h for all

the samples. In the following work, the peak aging time of 48 h was used for all the samples to induce the formation of the strengthening phase.

Fig. 2b-2d show the microstructure of the sample after 520 °C/ 30 min + 200 °C/ 48 h. The LPSO structures remained on the grain boundaries as evidence by TEM image. According to the EBSD test, the average grain size was  $16.3\pm2.4$  µm. The SAED result shows the  $\beta'$  precipitates precipitated after aging treatment. The lens-shaped  $\beta'$  precipitates can significantly increase the tensile strength of the Mg-RE alloy workpieces by hindering the basal dislocation slip.



Fig. 2 (a) The aging hardening curve of the solution treated (460 °C/ 1 h), (490 °C/ 1 h) and (520 °C/ 1 h) samples at 200 °C; (b-d) OM image, inverse pole figure and TEM images in (520 °C/ 30 min + 200 °C/ 48 h) sample.

#### 4 Conclusion

The phase constituents of the heterogeneous structure exhibit different evolution behaviors during the heat treatment process. Moreover, with continuously prolonging the duration at 520 °C, the metastable block-like structure is gradually changed into the lamellar LPSO structure, and then into the lath LPSO structures.

An optimal microstructure (fine grain + a certain amount of LPSO structures + nano-scale  $\beta'$  precipitates) and the corresponding customized heat treatment process (520 °C/ 30 min + 200 °C/ 48 h) are obtained to achieve a combination of a high strength of 364 MPa and a considerable elongation of 6.2%.

#### References

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