Effect of rare earth elements and heat treatment process on microstructures and properties of 6016 aluminum alloy

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Abstract: The effects of different contents of rare earth elements (erbium (Er) and yttrium(Y) ) on the microstructures and properties of 6016 aluminum alloy have been investigated. The results indicate that Er and Y can refine the grain size of 6016 aluminum alloy, and the precipitation of the second phase increases obviously and most of them are distributed in the grain boundary; the hardness values of the as-cast, rolled, T4 and T4P samples with Er and Y are obviously increased , which can be improved by 10~30HV, and the effect of Er on hardness improvement is better than that of Y. When both the addition mass fraction of Er and Y are 0.5%, the tensile property, work hardening coefficient and plastic strain ratio of aluminum alloy in T4P state are better, and they are respectively: 172.663MPa, 345.218MPa, 34.2%, 0.37, 0.79.

1. Introduction

Aluminum alloy because of its good formability, high recovery rate, lightweight and other characteristics, can meet the requirements of various industries for lightweight and recyclable materials, and has been widely concerned. Among a variety of fields, the application rate of aluminum alloy in automobiles industry is gradually expanding. In China, 6016 aluminum alloy has become the preferred lightweight material for automobile lightweight due to its moderate strength and good corrosion resistance[1-3]. However, with the development of the automobile industry, higher requirements have been put forward for the comprehensive performance of body plates and other parts of vehicles, and the lack of dent resistance of 6016 alloy still exists. Therefore, it is of great significance to further improve the performance of 6016 aluminum alloy[4].

Heat treatment strengthening and microalloying are important means to improve the properties of aluminum alloys. Some studies have found that rare earth elements(RE) can play a better strengthening effect on aluminum alloy[5–8]. Ding et al.[9–11] found that the Y element would exist in the form of AlTiY particles and Al3Y while adding Y in the aluminum alloy, meanwhile, it could
reduce the size of Mg$_2$Si phase and promote the transformation of brittle phase. Liu Yonggang and Xing Zebing et al.\cite{12,13} found that the nanoscale Al$_3$Er particles formed when Er was added to Al-Mg alloy could significantly refine the as-cast microstructure, and the subgrain boundaries and dislocations could be nailed during processing, thus improving the strength and recrystallization temperature. The experiments show that the improvement effect is more obvious and the mechanism is more complex after compositely adding some RE into the aluminum alloy\cite{14~16}. Peng et al.\cite{17} found that through the compound addition of Sc and Zr elements to Al-5.98Mg-0.47Mn alloy, the tensile strength of the alloy was increased to 440 MPa, which was 22% higher than that of the one added with zirconium alone, and the corrosion resistance reached PA level. Al$_3$Er and Al$_3$(Zr$_x$Er$_{1-x}$) particles precipitated in the heat treatment process of aluminum alloy after compound addition of Zr and Er elements. The particles have good dispersion strengthening effect, high melting point, good stability, and can form a semi-coherent structure with matrix\cite{17~21}.

The addition of Er or Y to 6016 aluminum alloy can significantly improve its comprehensive properties. However, there are few researches on the compound addition of the two elements and its strengthening mechanism. Therefore, this paper studies the effect of Er and Y compound addition on the microstructure and mechanical properties of the 6016 aluminum alloy in the state of rolled, T4 and T4P to provide experimental basis for the manufacture of high-performance aluminum alloy.

2 Experiment Procedure

The raw materials used in the experiment are industrial pure aluminum with purity of 99.9%, and the added master alloys are Al-20Si, Al-10Mg, Al-50Cu, Al-10Mn, Al-20Cr, Al-20Zn, Al-5Ti-B, Al-20Er and Al-10Y. The design composition of the alloys are showed in Table 1. The as-cast alloy samples were prepared by metallography. After corrosion with Keller reagent, the microstructures were analyzed by metallographic microscope and electron microscope, and the microhardness values were tested.

| Samples | Si  | Cu  | Mn  | Mg  | Cr  | Zn  | Ti  | Er  | Y   | Al  |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 0#      | 1.10| 0.40| 0.13| 0.74| 0.03| 0.20| 0.02| -   | -   | Balance |
| 1#      | 1.10| 0.40| 0.13| 0.74| 0.03| 0.20| 0.02| 0.1 | 0.3 | Balance |
| 2#      | 1.10| 0.40| 0.13| 0.74| 0.03| 0.20| 0.02| 0.2 | 0.2 | Balance |
| 3#      | 1.10| 0.40| 0.13| 0.74| 0.03| 0.20| 0.02| 0.3 | 0.1 | Balance |
| 4#      | 1.10| 0.40| 0.13| 0.74| 0.03| 0.20| 0.02| 0.35| 0.35| Balance |
| 5#      | 1.10| 0.40| 0.13| 0.74| 0.03| 0.20| 0.02| 0.5 | 0.5 | Balance |

The samples had been homogenizing under 550℃ for 10h in chamber electric furnace, then were quenched by water to room temperature. The hardness values were tested. These homogenized alloys were rolled by multi-pass rolling. The specific process was as follows: first preheating to 560 °C, then hot rolling ( 20mm-4mm ), and finally cold rolling ( 4mm-2mm-1mm ). Then, the samples rolled were done for solution treatment: 560 °C (chamber electric furnace) ×10 min, quenched by water to room temperature; and some of the samples had been placing at room temperature for one week to obtain T4 samples; and the other samples had been pre-aging at 170 °C for 10 min and then placing at room temperature for one week to obtain the T4P samples. At last, the microstructures, tensile property and hardness of the rolled, T4 and T4P samples had been studied.

HVS-1000 Vickers hardness tester was used, the load was 0.9807N, the pressure holding time was 15s, 10 points were measured for each sample, and the average value was taken as the experimental value. The Shimazu 100kN electronic universal testing machine was used to measure the tensile properties of the sample (shown as Fig. 1) under 25℃. Three sets of data were measured for each sample, and the average value was taken. The DM2500 Leica metallographic microscope was used to observe the microstructures after grinding, polishing and etching. The Rigaku 2500V X-ray diffractometer (XRD) was used to perform phase analysis at a scanning rate of 6°/min in the range of diffraction angle 2θ of 20°~80°. The SU-8020 field emission scanning electron microscope and
X-Max 80 spectrometer were used to analyze the microstructures and the composition of the samples.

![Fig.1 Size diagram of tensile specimen](image1)

![Fig.2 XRD analysis results of samples](image2)

3. Results and Discussion

3.1 XRD analysis
The results of XRD of the as-cast samples are shown as Fig. 2. It can be seen that the main phases in the samples are Al and Mg2Si phases. After the composite micro-alloying treatment, ErSi1.16 and Y phases are generated. With the increase of the addition amount of RE, the peak value of the diffraction peak gradually increases, and the diffraction peak broadens. Combined with the Bragg equation nλ = 2dsinθ, it can be seen that when the values of n and λ are constant, θ increases, and the interplanar crystal plane spacing (d) decreases, and the grain size also decreases, indicating that the addition of RE will play a role in refining the grains.

3.2 Microstructures analysis
The metallographic microstructures of the as-cast and T4P samples are shown as Fig. 3 and Fig. 4, and the results of grain size measurement are shown as Fig. 5.

From these microstructures and grain size, it can be seen that the grain size of aluminum alloy decreased obviously after the addition of Er and Y, which indicates that the addition of RE plays the effect of refining grain. When the total amount of Er and Y added is the same, the refining effect is the most obvious when the total mass of Er and Y added is the same, and under the same corrosion conditions, the degree of grain boundary corrosion is more significant (as 1#, 2# and 3# samples in Fig. 3); when the mass ratios of Er and Y elements are the same, the grain refining effect disappears with the further increase of RE content (as 2#, 4# and 5# samples in Fig. 3), which may be due to the cylindrical grain coarsening caused by the excessive addition of RE[22]. Meanwhile, with the increase of the addition of RE, the number of pores in the sample increases, indicating that excess RE will affect the as-cast properties and fluidity of aluminum alloy. In Fig. 4, the grains of samples become more uniform and refined. With the increase of the addition amount of RE, the refining effect becomes increasingly apparent, and the fine grain effect of 5# sample is the most significant. This is because the atomic radius of Er and Y are larger than that of Al, and their solubility values in Al are small. When the addition amount is small, they are solidly dissolved in Al matrix and converge at grain boundaries, phase boundaries and dendritic boundaries. Just like the previous analysis of ErSi1.16 phase and Y phase by XRD, the phases including RE precipitate in the matrix increase the resistance of grain boundary migration, promote heterogeneous nucleation, improve grain morphology and hinder grain growth that refines grains.

Fig. 6 shows the area of SEM energy spectrum analysis for each group of samples, Table 2 shows...
the results of energy spectrum analysis. In Fig. 6, the matrix of each samples is distributed with white bright colored granules, blocks or long strips and slightly gray spherical structures, which have a tendency to disperse along grain boundaries. From Table 2, the gray granules (such as D, G, I) and white rods (such as K, L, M, N) are enriched in Er and Y. Combined with XRD, ErSi$_{1.16}$, Y and the second phases containing Er and Y are located in these regions. These phases become elongated with the increase of the addition content of RE. The strengthening phase, that is β-phase (Mg$_2$Si) in 6016 aluminum alloy, is distributed in the granular, massive and long, spheroidal structures. These structures can effectively hinder the dislocation movement and improve the strength of the alloy. Combined with the results of microstructures analysis, it can be seen that after adding RE, the grain of the alloy is refined. This is because the formation of new alloy phase and strengthening phase provides a new nucleation site, and the formation of new phase can further hinder the grain growth.

Fig.3 Microstructures of as-cast samples

Fig.4 Microstructures of T4P samples
Fig. 5 Grain size measurement results of each group of samples

Fig. 6 Scanning electron microscope analysis of as-cast samples

Table 2 Results of energy spectrum analysis of characteristic points of each sample (mass fraction, %)

| Area | Al      | Mg      | Si      | Cu      | Fe | Mn | Er | Y     |
|------|---------|---------|---------|---------|----|----|----|-------|
| A    | 75.53   | 2.85    | 12.13   | 9.02    | -  | 0.46| -  | -     |
| B    | 81.27   | 15.86   | 2.87    | -       | -  | -  | -  | --    |
| C    | 94.46   | 0.89    | 4.66    | -       | -  | -  | -  | -     |
| D    | 37.31   | 1.54    | 13.13   | -       | 13.18| 3.03| 17.67| 13.73 |
| E    | 48.30   | 7.79    | 36.05   | 7.85    | -  | -  | -  | -     |
| F    | 75.63   | -       | 1.67    | 22.70   | -  | -  | -  | -     |
| G    | 59.55   | 5.52    | 15.64   | 4.90    | 4.80| 0.98| 6.82| 1.79  |
| H    | 30.48   | 2.49    | 56.25   | 10.79   | -  | -  | -  | -     |
| I    | 49.06   | 7.24    | 27.79   | 5.65    | 2.96| -  | 5.30| 2.00  |
| J    | 52.08   | 3.09    | 38.16   | 6.66    | -  | -  | -  | -     |
| K    | 51.38   | 4.74    | 13.99   | 14.45   | 1.26| -  | 9.35| 4.72  |
| L    | 78.38   | -       | 5.48    | -       | -  | -  | 10.40| 5.75  |
| M    | 38.43   | 11.22   | 30.90   | 11.13   | 2.28| -  | 3.35| 2.68  |
| N    | 52.09   | -       | 17.29   | 9.46    | -  | -  | 11.71| 9.46  |
3.3 Mechanical properties analysis

Fig. 7 is the hardness test results of the samples. It can be seen that the addition of RE can improve the hardness of the alloys. In as-cast samples, the hardness of 2 # is the highest (81.02 HV), which is related to the highest degree of grain refinement. The hardness of other samples can also be kept at 75–80 HV. After homogenization treatment, the hardness values of each group samples decrease, because homogenization treatment could make the composition of as-cast samples more uniform, which was more conducive to the later roll operation, and the hardness (79 HV) of 4 # is the highest. According to 1 # and 3 #, under the same conditions, the hardening degree by Er is better than that of Y. Moreover, when Er and Y add the same mass fraction, the best hardening effect can be obtained, and the total amount of Er and Y has little effect on the hardness of samples. After T4P treatment, the hardness values of each group of samples increase. Compared with those of as-cast samples, the hardness of T4P samples from 0 # to 5 # increase by 38.36, 27.89, 30.71, 39.04, 27.63 and 28.65 HV respectively. The hardness (118.308 HV) of 3 # is the highest. The results also show that the hardness of the alloy with more Er element after T4P treatment is higher than that of the alloy with more Y element. When the addition amount of RE increases gradually, the hardness of the samples do not increase significantly. It indicates that when the total amount of RE is 0.4 %, the ability of improving the hardness of aluminum alloy of RE reaches the limit.

![Fig.7 Hardness values of each group of samples](image1)

![Fig.8 Stress-strain curve of rolled samples](image2)

### Table 3: Mechanical property of the samples

| Sample | Yield strength (MPa) | Tensile strength (MPa) | Elongation (%) | Work hardening coefficient (n) | Plastic strain ratio (r) |
|--------|----------------------|------------------------|----------------|-------------------------------|------------------------|
| rolled |
| 0#     | 282.193              | 298.351                | 7.28           | 0.43                          | 0.75                   |
| 1#     | 358.485              | 371.412                | 6.72           | 0.45                          | 0.78                   |
| 2#     | 373.065              | 378.945                | 4.08           | 0.54                          | 0.80                   |
| 3#     | 295.281              | 311.909                | 4.92           | 0.52                          | 0.70                   |
| 4#     | 290.582              | 304.475                | 5.24           | 0.49                          | 0.77                   |
| 5#     | 413.785              | 425.571                | 10.4           | 0.44                          | 0.84                   |
| T4     |
| 0#     | 193.691              | 325.315                | 21.48          | 0.32                          | 0.74                   |
| 1#     | 164.226              | 301.832                | 31.72          | 0.34                          | 0.77                   |
It can be seen from Fig. 8 and Table 3 that the plasticity of rolled samples is poor. When both the addition of Er and Y elements are 0.5 % (5#), the sample has large yield strength, tensile strength and elongation. The maximum increment of yield strength can reach 131.592 MPa, and the maximum increment of tensile strength can reach 127.22 MPa. This results from fine grain strengthening and second phase precipitation dispersion strengthening because of the addition of Er and Y. At the same time, the work hardening coefficients of alloys are improved after adding Er and Y, which shows that Er and Y can effectively improve the formability of 6016 aluminum alloy, which is beneficial to the later processing.

Due to the poor plasticity of the rolled samples, it is not conducive to further use. Therefore, it is necessary to conduct solid solution treatment for the aluminum alloy samples to fully dissolve the second phase to improve its mechanical properties. The stress-strain curves and tensile-related data of T4 samples are shown as Fig. 9 and Table 3. It can be seen that after T4 treatment, the plasticity of each group of specimens increased significantly. The mechanical properties of 5# is still the best, the tensile strength of it is 351.347 MPa, and elongation can reach 38.88%. Its work hardening coefficient (n) and plastic strain ratio (r) are also the highest, indicating that it has good forming ability and is conducive to secondary processing application.

Compared with that of T4 samples, the tensile strength and yield strength of T4P samples decreased slightly, and the plasticity doesn't change much. The mechanical properties of 5# sample is the best, tensile strength is 345.218 MPa, elongation can reach 34.2%. Combined with the previous microstructure analysis, it can be seen that the grain of 5# sample is the smallest and the fine grain strengthening effect is the strongest. At the same time, the number of precipitates is more and the distribution is more dispersed, which plays an effective role in precipitation strengthening and dispersion strengthening in the matrix, pinning grain boundaries, hindering dislocation movement and improving strength.

Among all the samples, it can be seen that when the contents of Er and Y (5# sample) are same, the mechanical properties is the best, and that of 2# sample is the second. This may be because when the contents of Er and Y are same, they can jointly promote nucleation and precipitation of the second phase, and do not inhibit each other. When the addition of RE is 1.0, the effect of performance enhancement is more significant.
4. Conclusion

The effects of different contents of rare earth elements (erbium (Er) and yttrium(Y)) on the microstructures and mechanical properties of 6016 aluminum alloy had been investigated, the results are following:

(1) The addition of Er and Y has the effect of fine grain strengthening on the aluminum alloy, which is conducive to the nucleation and precipitation of the second phase. When both the addition amount of Er and Y element are 0.5%(mass%), the refining effect is the most obvious.

(2) The addition of Er and Y can promote the generation of new phases in 6016 aluminum alloy, in which the main precipitates are Mg2Si, ErSi1.16. With the increase of additive amount, the number of strengthening phase increases.

(3) The hardness of 6016 aluminum alloy can be greatly improved by adding Er and Y, and the effect of Er on hardness improvement is better than that of Y.

(4) The 6016 aluminum alloy treated by Er and Y composite microalloying has good mechanical properties. When both the addition mass fraction of Er and Y are 0.5%, the tensile properties, work hardening coefficient and plastic strain ratio of aluminum alloy in T4P state are better, and they are respectively: 172.663MPa, 345.218MPa, 34.2%, 0.37, 0.79.

Acknowledgement

This study was funded by project of the Project Sponsored by National University Student Innovation and Entrepreneurship Program (Grant No.202010593210), MOE Key Laboratory of New Processing Technology for Non-ferrous Metals and Materials, Guangxi Key Laboratory of Processing for Non-ferrous Metals and Featured Materials (2021GXMPSF02), and The First Batch of Guangxi Innovation-driven Development Projects in 2017 (Major Project of Science and Technology) (grant number: Guike AA17202008).

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