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Study on the Influence of Erbium and Preheating Process on Mechanical Properties of As-Cast 7055 Aluminum Alloy

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Abstract: Although 7055 aluminum alloy is a deformed aluminum alloy and will show excellent mechanical properties after recrystallization after large deformation, it is a method to enrich its application range if rare earth is added and the rare earth phase dispersion is promoted by heat treatment. This article used optical microscopy, scanning electron microscopy energy dispersive spectroscopy (SEM-EDS), X-ray diffraction (XRD), micro Vickers hardness and room temperature stretching methods to study the as-cast 7055-*x*Er (*x*=0wt.%, 0.2wt.%, 0.4wt.%, 0.6wt.%, 0.8wt.%) aluminum alloy after being subjected to 460 °C × 3h homogenization and 410 °C×1h solid solution+150 °C×12h aging treatment the changes in microstructure and properties. The results indicated that: when 0.2wt.%Er was added to 7055 aluminum alloy after solution at 410 °C×1h and aging at 150 °C×12h, the dendrite structure was significantly reduced, the grain thinning was obvious and the distribution was uniform, Al_8Cu_4Er phase appeared in the lamellar eutectic η-Mg(Zn,Al,Cu)$_2$ structure at grain boundaries, and the hardness reached 168.8HV. The yield strength, tensile strength and elongation were 542.12Mpa, 577.67MPa and 8.36%, respectively.

Keywords: 7055 aluminum alloy; erbium; mechanical properties; microstructure

1. Introduction

7055 aluminum alloy is not only high strength, but also has the advantages of low density and excellent processing formability, and is widely used in the aerospace field, often used in the manufacture of aircraft fuselage, wing beam and other structural products [1,2]. In recent years, with the continuous improvement of energy conservation and emission reduction requirements, low density and high strength have become the mainstream pursuit of aluminum alloy [3]. The conventional 7xxx alloy is a typical deformed aluminum alloy with excellent mechanical properties [4], but the deformation process is long and complicated, which increases the production cost and limits its application field. Cast aluminum alloys have the advantages of short process flow, near net forming, and low cost [5], and their largest consumers are the automotive industry, where they are used for various components, such as engine blocks and transmissions of internal combustion engine vehicles. In order to improve the performance of cast aluminum alloys to meet higher requirements of mechanical devices, heat treatment [6] and microalloying [7–9] can be used.

Because of its unique electron layer structure, rare earth elements can play a unique role in the production of aluminum alloy smelting and alloying, and play an important role in the development of super strong and tough aluminum alloy [7–9]. The addition of trace Sc in Al-Cu alloy can promote θ' precipitation in grain and inhibit θ' precipitation at grain boundary and inhibit the transformation of θ' into θ phase, there is a clear reinforcement effect[10–13]. The price of Sc is too high, even a small amount of addition will greatly increase the commercialization cost, and the lower price of Er also has a similar role of Sc element[9], becoming a potential rare earth modified element of 7055 aluminum alloy. The addition of Er will produce Al_3Er particles, which can inhibit the migration of subgrain boundaries during the solution aging process of aluminum alloy, so as to retain more LAGBs, the larger the proportion of LAGBs in the alloy, the better the corrosion resistance of the

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alloy[14,15]. The addition of Er, Cr and Zr can also enhance the recrystallization performance and stabilize the deformation recovery structure with many fine subgrain boundaries [9]. After adding Er to 7055 aluminum alloy, the synergistic effect of Si, Zr and Er elements inhibits the formation of hot cracks during pouring, and prevents the appearance of impurities and pores [16].

Heat treatment process has an important effect on improving the properties of 7055 aluminum alloy [17]. 7055 alloy as a kind of age hardening aluminum alloy, its excellent performance is mainly due to its uniform eutectic distribution Guinier-Preston zones (GP zones), semi-coherent η' phase and incoherent η phase. The precipitation sequence of these phases is [18]: Supersaturated solid solution zone (SSSS)- G.P. zones- η' phase (metastable $MgZn_2$)- η phase (equilibrium $MgZn_2$). Adjusting the size and quantity of the GP zone and η' phase formed in the early aging period is the key to improve the performance. At present, it is believed that the main strengthening effect of Al-Zn-Mg-Cu alloy is the joint action of GP region and η', and some scholars believe that the main strengthening phase is η' phase [19,20]. Precipitation hardening occurs in certain steps, such as solid solution treatment at the appropriate temperature and time, rapid cooling by quenching and aging treatment for a certain time to form a second phase precipitation inside the matrix [21]. Solution treatment is an important part of heat treatment, it is carried out at a relatively high temperature to dissolve the secondary phase formed during the solidification process, so that the alloying elements in the solid solution concentration is high and uniform. In general, for most of the as-cast 7055 aluminum alloy, the solution temperature is selected at 450 °C - 470 °C [22–24], while the Zn phase has a lower melting point (about 419.5 °C), higher solution temperatures can lead to overheating [25], thin and brittle oxide film at the banded interface[26]. Aging is the process of decomposition of supersaturated solid solution and precipitation of strengthened phase, according to the research [27], the as-cast 7xxx series aluminum alloy can reach the peak hardness of 189HV after aging at 120 °C for 156h, and the yield strength is increased by 240MPa compared with that of the as-cast aluminum alloy. After aging at 120 °C×1h+150 °C×4h, the time consumption is reduced and the hardness peak is reached at 184.7HV, the yield strength is increased by 235Mpa compared with the as-cast condition. When the aging temperature is increased to 150 °C, the growth of GP zone is accelerated, and the time to reach the peak hardness is shortened, and the corresponding mechanical properties are obtained. Higher aging temperature or longer aging time can increase the GP region volume fraction and the strength of the alloy, and the ductility of the alloy will not deteriorate significantly [28]. The single-stage aging temperature is generally 120 °C - 180 °C [29–31].

In order to refine the alloy structure and obtain the dispersed second phase to enhance the properties of 7055 aluminum alloy, considering that excessive temperature will lead to coarsening of the second phase size, in order to avoid this phenomenon and effectively improve the microstructure and mechanical properties of the material with rare earth phase, the solid solution treatment is set at 410 °C×1h. Combined with the above analysis, the aging treatment was set at 150 °C×12h. In this study, the process path of adding trace erbium element and heat treatment of as-cast 7055 aluminum alloy is proposed to further refine the grain structure of 7055 aluminum alloy and improve the strength and toughness of 7055 aluminum alloy. By adding different contents of Er to 7055 aluminum alloy, the microstructure and mechanical properties of four kinds of samples in as-cast 7055 aluminum alloy, as-cast heat treated 7055 aluminum alloy, as-cast 7055 aluminum alloy and as-cast heat treated 7055 aluminum alloy with Er were comparatively studied. The influence mechanism of Er and heat treatment process on as-cast 7055 aluminum alloy was discussed.

2. Materials and Methods

2.1. Preparation of Experimental Materials

7055 aluminum alloy and Er (99.99wt.%) as raw materials, 7055 aluminum alloy composition is Al-7.9Zn-2.5Cu-2.1Mg-0.1Zr-0.1Fe (wt.%). Using electronic balance (Shanghai Huachao Industrial Co., LTD., Shanghai, China) to weigh good raw materials according to the ratio of ingredients, and accurate to four decimal places. 7055 aluminum alloy was loaded into the alumina crucible at 780 °C and completely melted in the intelligent electric heating equipment (Xiangtan Samsung Instrument

Co., LTD., Xiangtan, China). Then added the rare earth element Er wrapped in aluminum foil and melt for 15min, stir with ceramic bar when adding, so that Er was more evenly integrated into the 7055 aluminum alloy, and then reduced to 740 °C for 15min, so that the gas in the solution is completely eliminated to prevent pores and slag inclusion in the casting, and poured the metal liquid into the graphite mold along the edge of the graphite mold. Let cool naturally to room temperature and remove from mold.

The sample was homogenized in a tube furnace (Xiangtan Samsung Instrument Co., LTD., Xiangtan, China) at 460 °C×3h. The as cast 7055-*x*Er (*x*=0wt.%, 0.2wt.%, 0.4wt.%, 0.6wt.%, 0.8wt.%) was obtained. The cast alloy was heat treated, the tube furnace was set at the solution temperature of 410 °C, the sample was immediately put into water to cool after holding for 1h, and then put into the tube furnace with the temperature set at 150 °C for 12h aging, and the heat treated 7055 -*x*Er aluminum alloy was obtained.

2.2.Data Acquisition and Analysis Methods

OM (ZEISS, ZEISS, Jena, Germany), SEM-EDS (ZEISS, EVO MA10, ZEISS, Jena, Germany), XRD (Ultima IV, Rigaku Co., Tokyo, Japan) grain size measurement and precipitated phase analysis were carried out respectively. Vickers hardness tester (SHYCHVT-30, Laizhou Huayin Hardness Meter Factory, Lai Zhou China) measures the hardness of the sample.

When measuring the size of the crystal phase, after grinding and polishing to a mirror surface, the corrosion was carried out with keller's etch (95ml water, 2.5mlHNO_3, 1.5mlHCL, 1.0mlHF). Metallographic photographs were taken at five different locations of each sample and the grain size was measured using the intercept method. Image-Pro Plus 6.0 software was used to process metallographic photos. First, a straight line segment was drawn in the metallographic photo, the length of the line segment was recorded, and then the number of grains passing through the line segment was counted. Then the length of the line segment was divided by the number of grains to obtain the average diameter of each grain. The average grain diameter in each metallographic photograph was measured 3 times. Finally, the average of the results obtained from all 5 metallographic photographs was taken as the grain diameter value of the sample.

Hardness measurement method: After pre-grinding and polishing, the Vickers hardness was measured, and the average value of the three points was taken. The test load was 5000g and the residence time of the indenter was 15s. The tensile test was carried out at room temperature using an electronic multifunctional testing machine (WDW-100C, jinan Fangyuan Instrument Co. Ltd., Jinan China). The specimen thickness was 1.5mm, the tensile rate was 0.1mm/min, and the tensile specimen was plate-like. The dimensions of the tensile specimen is shown in Figure 1.

The phase composition of the sample was analyzed by XRD, the scanning rate was set to 10°/min, and the X-ray diffraction range was 10°-90°. When SEM-EDS analyses the chemical composition, the sample used for observation and analysis of the second phase was ground and polished similar to the preparation of the metallographic sample before observation, without metallographic corrosion.

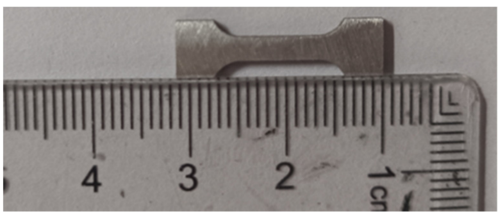

Figure 1. The tensile specimen.

3. Results

3.1. Microstructure and Mechanical Properties

Figure 2 shows the metallographic microstructure (OM) of as-cast 7055 aluminum alloy and as-cast 7055-*x*Er aluminum alloy. The dendrite coarsening state of the as-cast 7055 aluminum alloy was obviously improved after heat treatment, and the grain was refined. After heat treatment of 7055-0.2wt.%Er aluminum alloy, the grain was obviously refined and appeared as cell crystal. However, the content of Er element was increased to 0.4wt%, 0.6wt% and 0.8wt% after heat treatment, coarse dendrites appeared again. When the content of Er exceeds a certain value, since aluminum is a face-centered cubic structure, Er is a close-packed hexagonal structure, and the atomic radius of Er (0.176nm) is larger than that of Al (0.143nm), if Er enters the crystal lattice of Al, it will cause large lattice distortion and increase the system energy. In order to reduce the grain boundary energy, Er is enriched toward the grain boundary. During solidification, Er will gather at the front of the solid-liquid interface, and increasing the constitutional supercoiling [**Error! Reference source not found.**], then promoting nucleation and reducing grain size. Therefore, proper addition of Er can promote grain refinement.

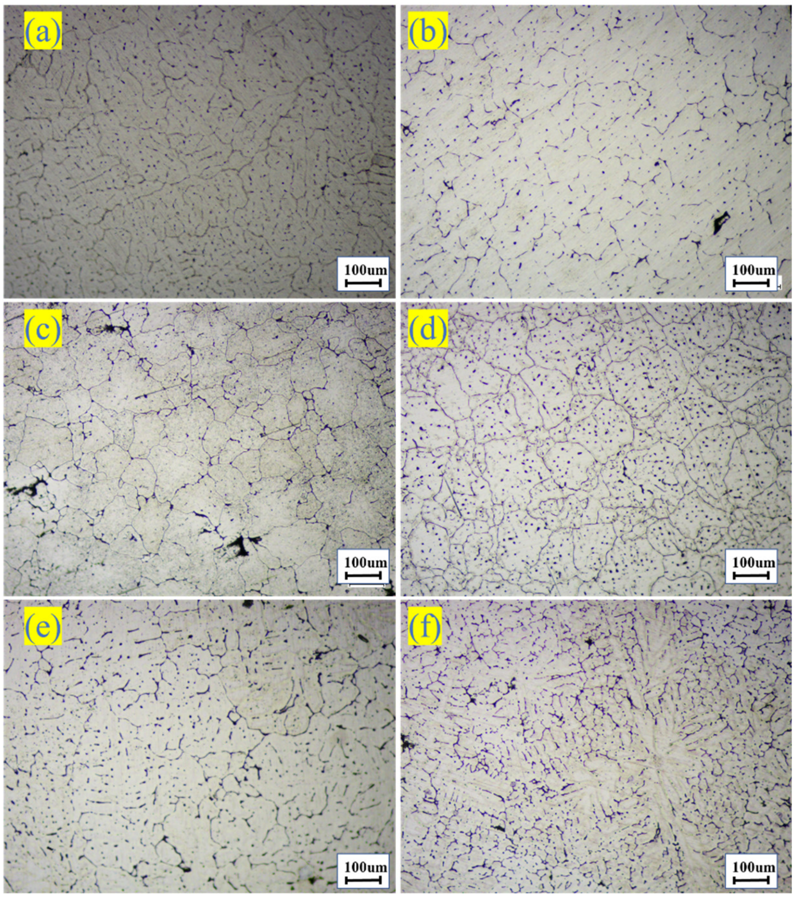

Figure 2. Optical microstructure: (a) as-cast 7055-0wt.%Er; (b) as-cast heat treatment 7055-0wt.%Er; (c) as-cast heat treatment 7055-0.2wt.%Er; (d) as-cast heat treatment 7055-0.4wt.%Er; (e) as-cast heat treatment 7055-0.6wt.%Er; (f) as-cast heat treatment 7055-0.8wt.%Er.

3.2. Phase Component Analysis

The phase composition of as-cast 7055 aluminum alloy, as-cast 7055-0.2wt.%Er and as-cast heat treatment 7055-0.2wt.%Er before and after the heat treatment was analyzed by XRD, as shown in Figure 3. The XRD pattern of the as-cast 7055 aluminum alloy is similar to that of the as-cast 7055 aluminum alloy, so it is not marked. The main precipitated phases of 7055 aluminum alloy before and after heat treatment are α-Al, η-$MgZn_2$, and the diffraction peak of Al_8Cu_4Er (PDF 33-0006) appears near 29° when Er was added after heat treatment (see Figure 1 (b)).

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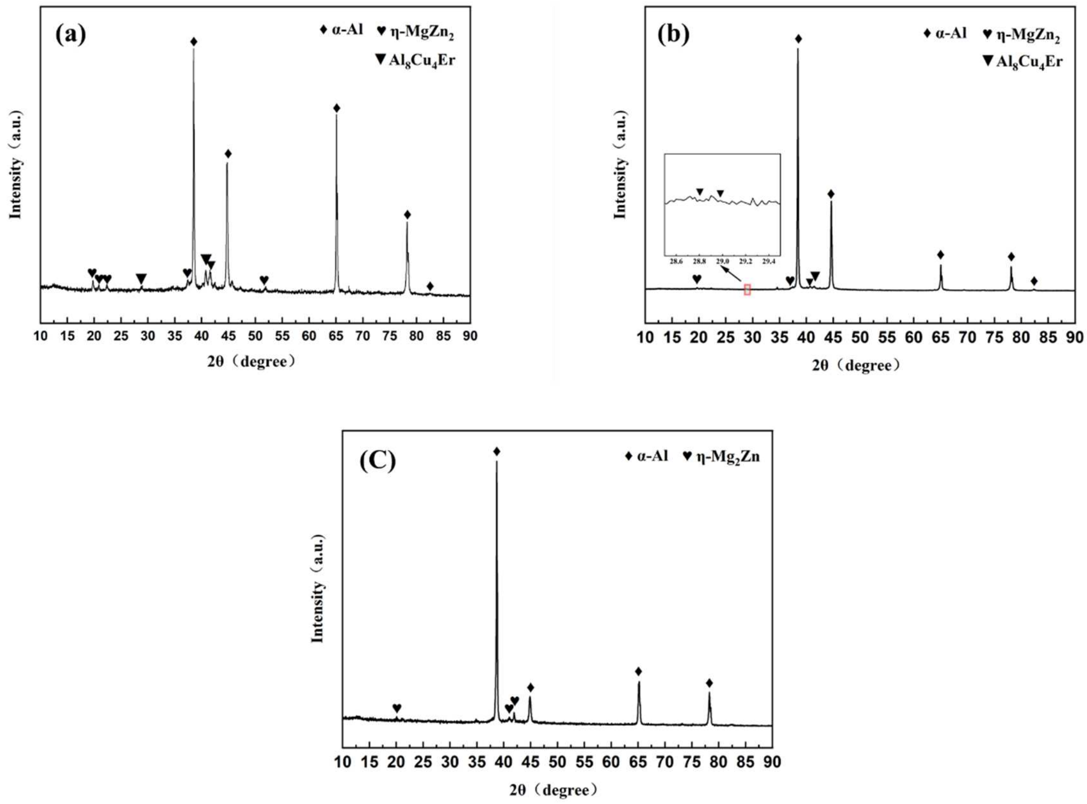


Figure 3. The XRD patterns: (a) as-cast treatment 7055-0.2wt.%Er; (b) as-cast heat treatment 7055-0.2wt.%Er; (c) as-cast 7055.

Figure 4 shows the SEM backscattering diagram of 7055 aluminum alloy with different components after cast and heat treatment, and the EDS measurement results of the corresponding intermetallic compounds on the diagram are shown in Table 1. Intermetallic compounds appear brighter in the image because their atomic number is higher than that of the Al matrix. The as-cast Al-Mg-Zn-Cu alloys are generally composed of α(Al) matrix and non-equilibrium eutectic structures, including α-Al, η-Mg $(Zn,Al,Cu)_2$, S-$CuMgAl_2$, Al_7Cu_2Fe and Mg_2Si[33–35]. EDS analysis showed that high-density lamellar eutectic η-$Mg(Zn,Al,Cu)_2$ existed in all the four samples (Figure 4 point e, f, h, j). In 7055-0.2%Er aluminum alloy, a new phase Al_8Cu_4Er (Figure 4 point g, i) appeared in both the as-cast and solid-solution aging states, which in the form of polygonal blocks. In addition, Al_3Er may also exist, but due to the small content, not detected by XRD.

Table 1. Chemical composition of the points in Figure 4 determined by EDS analysis (at. %).

Point	Al	Mg	Cu	Zn	Er	Fe	Phase
Spots A	58.98	15.89	21.75	15.21	0	0	α-Al+η-$Mg(Zn,Al,Cu)_2$
Spots B	59.52	18.27	7.95	14.27	0	0	α-Al+η-$Mg(Zn,Al,Cu)_2$
Spots C	58.78	3.39	28.45	6.01	3.36	0	Al_8Cu_4Er
Spots D	82.45	1.33	5.25	1.84	0	9.14	α-Al+η-$Mg(Zn,Al,Cu)_2$
Spots E	58.27	1.57	27.92	6.28	4.98	0.98	Al_8Cu_4Er
Spots F	25.90	32.61	16.78	24.70	0	0	α-Al+η-$Mg(Zn,Al,Cu)_2$

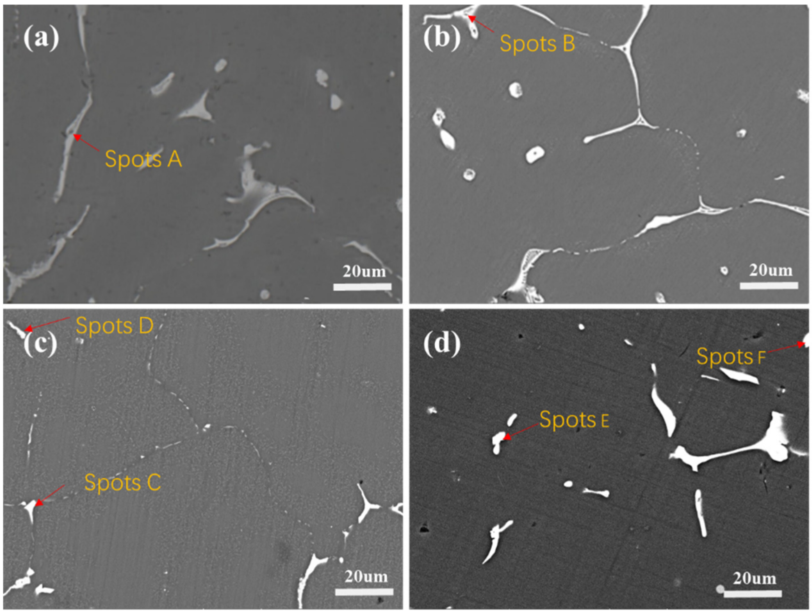
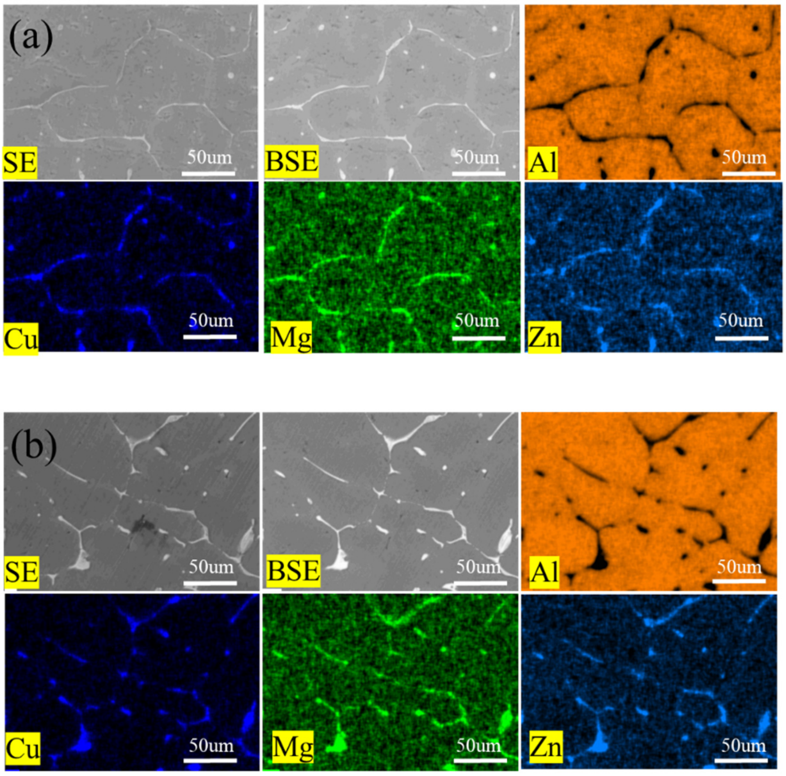
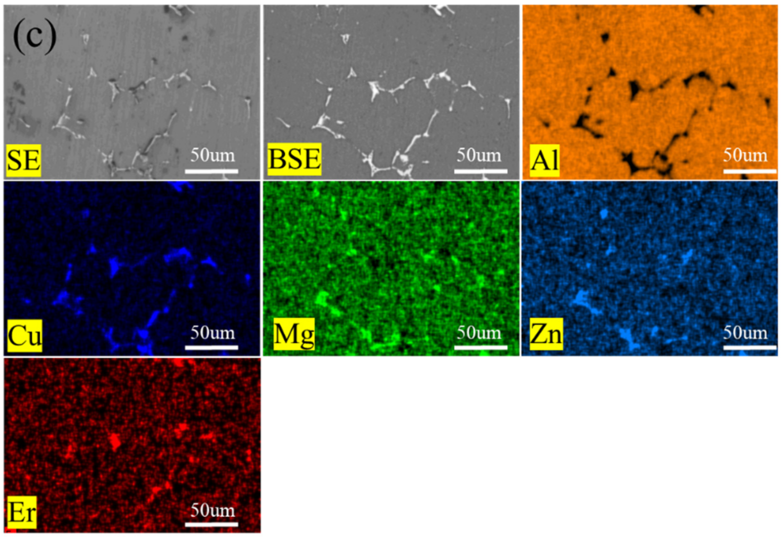


Figure 4. SEM microstructure images: (a) as-cast 7055; (b) as-cast heat treatment 7055; (c) as-cast 7055-0.2wt.%Er; (d) as-cast heat treatment 7055-0.2wt.%Er.

Figure 5 shows the mapping scanning results of 7055 aluminum alloy with different components after casting and heat treatment. Compared with the original as-cast 7055 aluminum alloy, the Mg and Zn segregation of the 7055 aluminum alloy with Er element was relatively weakened (Figure 5a,b and Figure 5c,d). After heat treatment, the Er element in 7055-0.2 wt.%Er aluminum alloy was more evenly dispersed than that in the as-cast 7055-0.2% Er aluminum alloy.





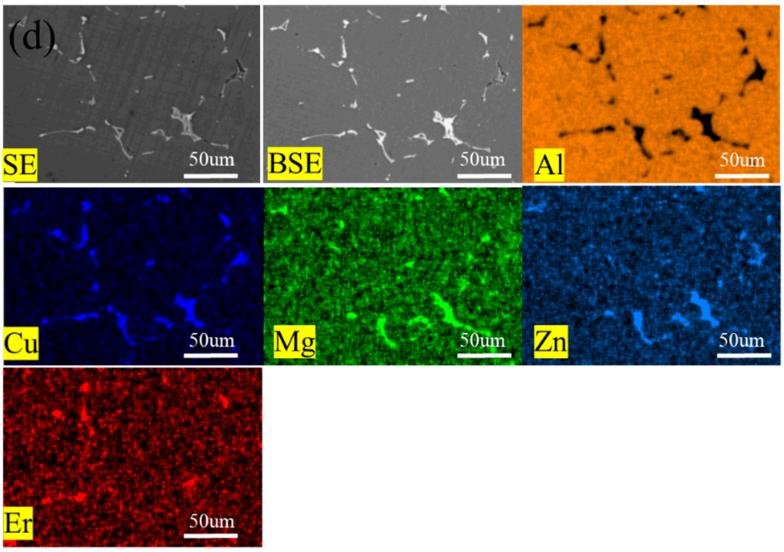


Figure 5. EDS mapping scanning results of alloy samples under different states: (a) as-cast 7055; (b) as-cast heat treatment 7055; (c) as-cast 7055-0.2wt.%Er; (d) as-cast heat treatment 7055-0.2wt.%Er.

3.3. Mechanical Property

Figure 6 shows the Vickers hardness values of 7055-xEr aluminum alloy treated as cast and heat treatment. The hardness of the as-cast 7055 aluminum alloy with the addition of Er was higher than that of the original as-cast 7055 aluminum alloy, and the hardness of the corresponding component of 7055 aluminum alloy with the addition of 0.6wt.%Er was improved after solution aging, and the hardness of the 7055 aluminum alloy with the addition of 0.2wt.%Er after heat treatment reached the maximum value 168.4HV. Compared with the original cast 7055 aluminum alloy hardness value increased by 60.4HV.

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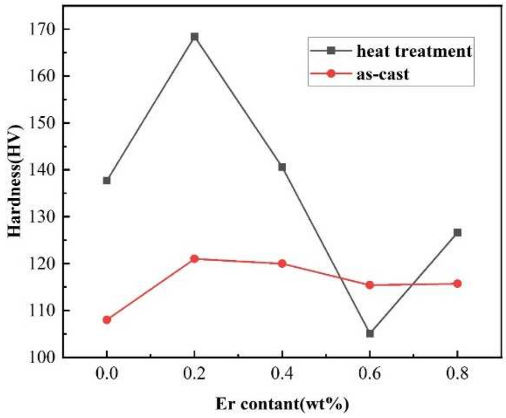

Figure 6. Hardness value curves of 7055 aluminum alloy with different Er content before and after heat treatment.

Figure 7 shows the tensile curves of 7055 aluminum alloy samples with different Er content before and after heat treatment. The Figure 7a,c show that the elongation of 7055-0.2wt.%Er in as-cast state is the maximum value 10.54%. The maximum yield strength and tensile strength of 7055-0.2wt.%Er in as-cast state heat treatment are 542.12MPa and 577.67MPa, respectively. Except that the tensile properties of the heat treatment decreased when the Er content was 0.6wt%, the tensile properties of the other contents were improved after heat treatment (Figure 7b,d).

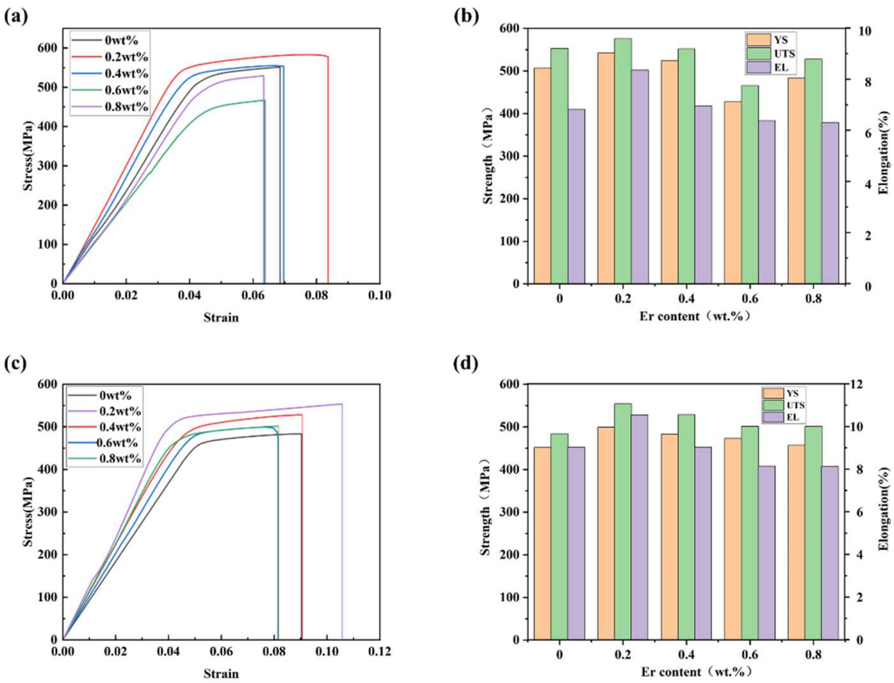

Figure 7. The tensile properties: (a) Tensile curve of 7055-*x*Er heat treatment in as-cast state; (b) Column chart of yield strength, tensile strength and elongation of 7055-*x*Er for as-cast heat treatment; (c) Tensile curve of 7055-*x*Er as-cast; (d) Column chart of yield strength, tensile strength and elongation of 7055-*x*Er as-cast.

4. Conclusions

In this paper, the changes of microstructure, phase composition, hardness and mechanical properties of 7055 aluminum alloy with different Er content before and after heat treatment were studied. The conclusions are as follows:

(1). 7055-0.2wt.%Er aluminum alloy after heat treatment, the grain refinement effect was the most obvious, the grain size was 72μm.

(2). Al_8Cu_4Er was formed after the addition of Er to 7055 aluminum alloy, and the high-density lamellar eutectic *η*-$Mg(Zn,Al,Cu)_2$ became thinner. After adding 0.2wt.%Er, the distribution segregation phenomenon of Mg and Zn was reduced, and the Er element in 7055-0.2wt %Er aluminum alloy after heat treatment was more evenly dispersed than that in the as-cast 7055-0.2wt.%Er aluminum alloy.

(3). At solution 410 °C×1h and aging 150 °C×12h, the mechanical properties of as-cast 7055-0.2wt %Er aluminum alloy after heat treatment increased the fastest, and the hardness reached 168.8HV, which was 60.4HV higher than that of 7055 as-cast aluminum alloy. The maximum elongation of 7055-0.2wt.%Er as cast was 10.54%, and the yield strength, tensile strength and elongation of 7055-0.2wt.%Er as cast heat treatment were 542.12MPa, 577.67MPa and 8.36%, respectively.

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Conflicts of Interest: The authors declare no conflicts of interest.

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