Effect of annealing time on bimodal microstructures and tensile properties of Mg–6Sn–3Al–1Zn alloy

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Abstract
Although the Mg–6Sn–3Al–1Zn alloy with bimodal grain size distribution has excellent mechanical properties, its mechanical properties can further be improved by adjusting or controlling its microstructures through annealing. In this study, the effect of annealing time on microstructures and mechanical properties of the Mg–6Sn–3Al–1Zn alloy was systematically studied. The Mg–6Sn–3Al–1Zn alloy with bimodal microstructures was prepared through pack-rolling with two passes at 450 °C (55 % thickness reduction per pass, and the total rolling deformation is 80 %). Then the alloy was annealed for 10 min, 20 min, 30 min and 40 min at 350 °C, respectively. The results showed that volume fractions of Mg2Sn phase and recrystallization grains increased with increasing of the annealing time. The multi-modal microstructures were formed in alloys after annealing. When the annealing time ≤30 min, the average grain size of the alloy increased with increasing of the annealing time, while it decreased after annealed for 40 min. The intensity of {0001} texture decreased with increasing of annealing time (<20 min), which caused by dynamic recovery. When the annealing time reached 30 min, the generation of recrystallization led to the enhancement of {0001} texture. However, the {0001} texture intensity reduced after the alloy was annealed for 40 min, which caused by further dynamic recrystallization. In Mg–6Sn–3Al–1Zn alloy annealed for 30 min, the volume fractions of Mg2Sn phase and recrystallization grains were respectively 1.29 % and 56 %, and the average grain size was 6.28 μm. Moreover, the mechanical properties of the alloy reached optimum, i.e., the tensile strength, yield strength and elongation were 336 MPa, 194 MPa and 26 %, respectively.

1. Introduction

The magnesium alloys as the lightest constructional material will be wildly used in many fields owing to its low density, high specific strength and specific stiffness. When the magnesium alloys were used on the rockets and satellites could reduce launch costs, and when it was used on airplane also cut down flying costs. The weight of transportation vehicles would be lightened after magnesium alloys were used, therefor fuel consumption and environmental pollution were reduced. When the magnesium alloys were used on 3C products, it could improve the appearance quality and lightened the weight.

The strength and ductility of the alloys are contradictory, both the strength and ductility cannot be enhanced rarely at the same time. How to improve the strength of the magnesium alloys while maintaining or even improving the ductility, is a hot topic in alloy research [1–5]. Grain refinement could improve the strength of alloy, for example, when the grain size of alloy decreases to ultrafine-grains or even nano-grains levels, the strength of alloy can reach to many times than their coarse-grains counterparts, but low ductility limits their applications. Bimodal microstructures include large grains and small grains that distribute uniformly in the alloy, the large grains provide plasticity, while small grains provide strength, developing which could simultaneously improve the strength and ductility of alloy [6–8]. There is a huge reported progress in the understanding of the strength-ductility balance of Mg alloys in recent studies, At present, high strength and
ductility has been obtained through bimodal structure in Ni [9, 10], Ti [11, 12], Zr [13, 14], Cu–Ag alloys [6, 7, 15, 16], Al–Mg alloys [8, 17–19], TiZr alloys [20] alloys as well as steels [21–23]. Conventional rolling required elevate temperatures and increased rolling passes owing to the poor plasticity of magnesium alloy. Pack-rolling not only effectively reduce temperature drop, but also reduce edge crack during the process of rolling. Bimodal microstructures of Mg–6Sn–3Al–1Zn alloy was obtained through pack-rolling at 450 °C with two passes, 55% rolling reduction per pass, and the total rolling deformation is 80%. The grain sizes of the pack-rolled Mg–6Sn–3Al–1Zn alloy are larger than bimodal 304 and 316 stainless steel [24, 25], but the coarser bimodal microstructures produced by pack-rolled also leads to a simultaneously high strength and ductility than casting Mg–6Sn–3Al–1Zn alloy.

Undoubtedly, the volume fractions of small grains and large grains, the grain size of small grains and big grains in the bimodal microstructures, and the rolling texture could influence the mechanical properties of Mg–6Sn–3Al–1Zn alloy. The present work thus aims to study the effects of annealing time on the bimodal microstructures and tensile properties of pack-rolled Mg–6Sn–3Al–1Zn alloy sheets. Annealing could adjust the microstructures of Mg–6Sn–3Al–1Zn alloy with bimodal gain size distribution (figure 1), which was produced by pack-rolling. When the alloy is annealed, some small grains of alloy in bimodal microstructures grows up, unrecrystallization big grains become recrystallization small grains, there is formed multi-modal microstructures. When the Mg–6Sn–3Al–1Zn alloy is annealed at 450 °C for 30 min, the yield strength of Mg–6Sn–3Al–1Zn alloy decreased a little, however, the elongation increased greatly. This paper will provide a new method for enhance the strength and elongation of magnesium alloys simultaneously.

2. Experimental

2.1. Fabricated cast Mg–6Sn–3Al–1Zn alloy

(1) We configured 1.8 kg Mg–6Sn–3Al–1Zn alloy. The raw materials of the alloy were weighted by analytical balance according to weight ratio of the alloying elements, and then the surfaces of the Mg, Al, Sn and Zn ingot were polished with an angle grinder in order to remove oxide and dirt, and dried by muffle furnace at 150 °C.

(2) Residues of crucible, casting mould, slag spoon, stirrer and thermocouple were cleaned by an angle grinder, and then appropriate concentration talcum powder sodium silicate mixture liquid (100 g talcum powder was added into 200 ml water soluble silicate and was stirred with glass bar to ensure uniformity of the mixture liquid) was uniformly smeared to these tools. And they were dried in the sun until the solidification of mixture liquid, and then were further dried with heating furnace at 200 °C.

(3) We have weighted RJ-2 type refining agent 36 g, and dried it with heating furnace at 200 °C.

(4) The crucible was heated by crucible furnace to 500 °C, and then the dried Mg ingots were put into crucible. The mixed protective gas of CO2 and SF6 with a volume ratio of 99 : 1 is introduced into crucible and the
temperature of crucible furnace was set as 800 °C. Mg ingots was heated up until completed melting, then the Al, Zn and Sn ingot were added into the crucible in sequence. 

(5) When Mg, Al, Sn and Zn ingots were all melted, RJ-2 type refining agent was added into the melt and kept stirring 3 min. Then the impurities and residue oxides will be on top of the melt, and could be slagging-off by ladle.

(6) The melt will continue to be heated to 730 °C and held for 10 min. After the second slagging, the melt was poured into mold which was preheated to 200 °C. Protective gas was introduced during pouring process in order to prevent the alloy from oxidizing and blackening. Finally, we opened the mold, take out billet, and cooled it down in air.

2.2. Pack-rolling Mg–6Sn–3Al–1Zn alloys
The cylindrical alloy was machined to 42 × 42 × 110 mm, and executed solid solution for 8 h at 450 °C and followed water quenching. And then the alloy was cut by wire cutting to 30 × 50 × 3 mm. The exquisite sheets were packed using 6063 aluminium alloy after eliminating oxide layer on the surface of the sheets by 1500# sandpaper. The packed alloy was rolling at 450 °C with two passes, after rolling one pass, the sample was kept in furnace for 2 min, the amount of deformation per pass was 55 %, total rolling deformation was 80 %. After rolling, the samples were annealed at 350 °C with 10 min, 20 min, 30 min and 40 min, respectively, and followed water quenching, after that stripped aluminum alloy sleeve from sample after annealed.

2.3. Microstructure characterization and mechanical properties test
X- ray diffraction (XRD) analyze: cut 10 × 6 mm in the center of the annealed samples along the rolling direction, and cleaned with ultrasonic cleaners for 30 min after polished with 600# sandpaper, and then dried with hair dryer. Using the D/MAX-2400 type x-ray diffractometer for phase analyzing, scanning range was 20° ≤ 2θ ≤ 90°, scanning rate was 7° min⁻¹, scanning step was 0.02°, operate voltage and current was 40 KV and 40 mA, respectively.

Electron back scattering diffraction (EBSD) analyze: cut 5 × 3 mm in the center of the annealed samples along the rolling direction, and polished with 400#, 800#, 1200#, 2000#, 3000#, 5000# and 7000# sandpaper, and then electrochemical polishing with 7 % perchlorate alcohol solution, added liquid nitrogen and stirred constantly until the temperature of solution reached to −25 °C, polished voltage and current was 20 V and 0.2 μA, respectively, polished time was 2 min. Argon ion polishing was carried out after electropolishing, polished voltage was 3 kev, polished angle was 3°, polished time was 90 min, and then the sample was observed by the ZEISS MERLINCOMPACT type scanning electron microscope, operate voltage and step was 20 KV and 0.21 μm, respectively, {0001} basal plane, {11-20} and {10-10} cylindrical plane pole figures were tested.

Statistics on the volume fraction of Mg2Sn phase: the white phase was Mg2Sn phase, and the matrix was α-Mg phase in the SEM images. Statistic works were performed on the total area of Mg2Sn phase, and then the area of the SEM image was divided, therefore, the volume fraction of Mg2Sn phase was obtained.

Tensile properties test: the sample was cut by wire cutting into bone shape along the rolling direction, and polished with 800# sandpaper. the dimensions of the tensile specimen were shown in figure 2, tensile properties was tested by an universal testing machine (Shimadzu AGS-X-50 kN), the tensile rate was 0.2 mm min⁻¹, and the test was repeated 3 times, the average value was as the final value of the tensile tests.

Figure 2. The sizes of tensile sample.
3. Results

Figure 3 shows that the XRD patterns of Mg–6Sn–3Al–1Zn alloys without and with annealing at 350 °C for different time. As can be seen, all the alloys are composed of Mg₂Sn phase and α-Mg phase, indicating that annealing process does not change the phases composition of the alloys.

Figure 4 shows that the SEM images of Mg–6Sn–3Al–1Zn alloys with annealing at 350 °C for different time. The white dots shape and strips shape in microstructure of alloys are Mg₂Sn phase, and the gray phase is α-Mg. The volume fraction of Mg₂Sn phase increases with increasing of the annealing time, the volume fraction of Mg₂Sn phase is 1.07 %, 1.18 %, 1.29 % and 1.49 % when the annealing time is 10 min, 20 min, 30 min and 40 min, respectively.

Figure 5 shows that the EBSD images of grains of recrystallized, substructured and deformed grains of Mg–6Sn–3Al–1Zn alloys with annealing at 350 °C for different time, the blue area, yellow area and red area represent...
recrystallized regions, substructure regions and deformed regions, respectively. The EBSD technique which used to distinguish the recrystallized, substructured and deformed grains has been reported in [26]. The volume fractions of the recrystallized, substructured and deformed grains of Mg–6Sn–3Al–1Zn with annealing at 350 °C for different time are show in figure 6, which is statistics from figure 5. The volume fraction of recrystallized grains increases with increasing of the annealing time, when the annealing time was 10 min, 20 min, 30 min and 40 min, the volume fraction of recrystallized grains is 10 %, 17 %, 56 % and 66 %. The volume fraction of substructured grains decreases with increasing of the annealing time, when the annealing time was 10 min, 20 min, 30 min and 40 min, the volume fraction of substructured grains is 88 %, 80 %, 31 % and 24 %, respectively.

Figure 7 shows the EBSD maps of the Mg–6Sn–3Al–1Zn alloys with annealing at 350 °C for different time. Grain size distribution diagrams of Mg–6Sn–3Al–1Zn alloys with annealing at 350 °C for different time are shown in figure 8, which is statistics from figure 7. We can see from figure 7, there is formed multi-modal gain distribution microstructures after annealed. With increasing of the annealing time, unrecrystallization big grains become recrystallization small grains, the grain size of big grains decreasing and the grain size of small grains increasing. We can see from figure 8, when the annealing time is less than or equal to 30 min, the average grain size of alloy increases with increasing of the annealing time, the average grain size of alloy is 5.16 μm, 5.43 μm and 6.28 μm after the alloy is annealed with 10 min, 20 min and 30 min, respectively. After the alloy is annealed for 40 min, the average grain size of alloy decreases to 5.81 μm.

Figure 9 shows low angle boundary distribution diagrams and corresponding local misorientation angle distribution diagrams of the Mg–6Sn–3Al–1Zn alloys with annealing at 350 °C for different time. As can be seen from figures 9(a), (c), (e) and (g), the low angle boundary occurs in big grains. When the alloy is annealed with 20 min, both the number of big grains and the grain size of big grains increase, the volume fraction of low angle boundary increases, after the alloy is annealed with 30 min, both the number of big grains and the grain size of big grains unchanged, the volume fraction of low angle boundary unchanged, however, after the alloy is annealed with 40 min, the grain size of big grains decreases, the volume fraction of low angle boundary decreases. The evolution of average local misorientation angle with increasing of the annealing time consist with the evolution of volume fraction of low angle boundary with increasing of the annealing time, when the alloy is annealed with 10 min, 20 min, 30 min and 40 min, the average local misorientation angle is 0.624 °, 0.705 °, 0.703 ° and 0.590 °.

The grain distribution diagrams and the volume fraction of three types grain size of Mg–6Sn–3Al–1Zn alloys with annealing at 350 °C for different time are shown in figure 10 and summarized in table 1. When annealing...
time \leq 30 \text{ min}, with increasing of the annealing time, the volume fraction of small grains (\text{grain size} < 10 \mu m) decreases, the volume fraction of middle grains (10 \mu m < \text{grain size} < 30 \mu m) increases, the volume fraction of big grains (\text{grain size} > 30 \mu m) almost does not changes, when the alloy is annealed with 10 min, 20 min and 30 min, the volume fraction of small grains is 44.1\%, 37.7\% and 31.4\%, the volume fraction of middle grains is 21.9\%, 31.5\% and 36.9\%, the volume fraction of big grains is 34.0\%, 30.8\% and 31.7\%. When the sample is annealed for 40 min, the volume fraction of grain size which is less than 30 \mu m increases, the volume fraction of grain size which is larger than 30 \mu m decreases, the average grain size of alloy decreases.

Figure 11 shows the pole figure of \{0001\} basal plane, \{11\_20\} and \{10\_10\} cylindrical planes of Mg–6Sn–3Al–1Zn alloys with annealing at 350 °C for different time. \{0001\} basal texture form in the alloys after packed rolling, and texture types do not change after annealed, texture intensity is 12.95, 9.24, 12.56 and 8.13 after annealed for 10 min, 20 min, 30 min and 40 min, respectively. When the annealing time is less than or equal to 20 min, the volume fraction of recrystallized grains of alloy is little, the texture intensity decreases with increasing of the annealing time, which caused by dynamic recovery. When the annealing time is 30 min, the volume fraction of recrystallized grains of alloy increases sharply, the recrystallized texture lead to the texture intensity increases. When the annealing time is 40 min, the texture intensity decreases, which caused by further dynamic recrystallization.

The tensile stress-strain curves, the variation of tensile strength, yield strength and elongation of the Mg–6Sn–3Al–1Zn alloys with annealing at 350 °C for different time are shown in figure 12. When the alloy is annealed for 10 min, the tensile strength decreases from 326 MPa of the as-rolled alloy to 285 MPa, and yield strength decreases from 212 MPa of the as-rolled alloy to 156 MPa, while the elongation does not change. When the alloy is annealed with 20 min, the tensile strength, yield strength and elongation increase to 310 MPa, 172 MPa and 22\%, respectively. When the alloy is annealed with 30 min, the tensile strength, yield strength and elongation increase to 336 MPa, 188 MPa and 26\%, respectively. When the alloy is annealed with 40 min, the tensile strength and elongation decreases to 325 MPa and 23.6\%, respectively, but the yield strength does not change.
Figure 7. The EBSD maps of the Mg–6Sn–3Al–1Zn alloys with annealing at 350 °C for 10 min (a), 20 min (b), 30 min (c) and 40 min (d), respectively.

Figure 8. Grain size statistical diagram of Mg–6Sn–3Al–1Zn alloys with annealing at 350 °C for 10 min (a), 20 min (b), 30 min (c) and 40 min (d), respectively.
Figure 9. Low angle boundary distribution diagrams (a), (c), (e), (g) and corresponding local misorientation angle distribution diagrams (b), (d), (f), (h) of the Mg–6Sn–3Al–1Zn alloys with annealing at 350 °C for 10 min (a), (b), 20 min (c), (d), 30 min (e), (f) and 40 min (g), (h).
4. Discussion

4.1. The effect of annealing time on the volume fraction of Mg$_2$Sn phase

The volume fraction of Mg$_2$Sn phase increase with increasing of the annealing time. It was because the alloy is solid solution before rolling, which lead to Mg$_2$Sn phase solid solute into $\alpha$-Mg matrix, after annealed at 350 °C, the Mg$_2$Sn phase aging precipitation from as-rolled alloys. The longer annealing time, the more volume fraction of Mg$_2$Sn phase.

| Table 1. The volume fractions of three types grain sizes of Mg-6Sn-3Al-1Zn alloys with different annealing time. |
|---------------------------------|
| Grain size | Annealed for 10 min | Annealed for 20 min | Annealed for 30 min | Annealed for 40 min |
| $<10$ µm | 44.1% | 37.7% | 31.4% | 41.1% |
| $10$ µm | 21.9% | 31.5% | 36.9% | 46.2% |
| $>30$ µm | 34.0% | 30.8% | 31.7% | 12.7% |

Figure 10. Grain distribution diagram of Mg–6Sn–3Al–1Zn alloys with three types grain size come from EBSD images with annealing at 350 °C for 10 min (a)–(c), 20 min (d)–(f), 30 min (g)–(i) and 40 min (j)–(l), grain size $<10$ µm (a), (d), (g), (j), 10 µm $<\text{grain size}<30$ µm (b), (e), (h), (k), grain size $>30$ µm (c), (f), (i), (l).
4.2. The effect of annealing time on volume fraction of recrystallized grain

The volume fraction of recrystallized grains not only is related to annealing temperature, but also is connected with annealing time. The higher annealing temperature, the shorter annealing time is needed to complete dynamic recrystallization. The stress of as-rolled alloy, annealing time and annealing temperature provide drive force for recrystallization. When the annealing time of alloy is 20 min, the volume fraction of recrystallized grains is 17 %, it was because the alloy belongs to close-packed hexagonal structure, which has high fault energy, and the dynamic recovery is obvious at the initial stage of annealing, then lead to the absence of dynamic recrystallization. When the annealing time of alloy is 30 min, the volume fraction of recrystallized grains increases to 56 %, the dynamic recovery has been completed, and the dynamic recrystallization phenomenon is rather distinct owing to long annealing time. When the annealing time is 40 min, the volume fraction of recrystallized grains slowly increases to 66 %, due to that the recrystallization drive force of deformed alloy almost disappear, the only remaining driving force is annealing temperature and annealing time, the drive force of recrystallization

![Figure 11](image-url). The pole figures of (0001) basal plane, (11-20) and (10-10) cylindrical planes of Mg–6Sn–3Al–1Zn alloys with annealing at 350 °C for 10 min (a), 20 min (b), 30 min (c) and 40 min (d).
decreases, thus the volume fraction of recrystallized grains increase inconspicuously compared to the alloy with annealing for 30 min.

4.3. The effect of annealing time on grain size of alloy
The volume fraction of recrystallized grains of alloy after annealed for 20 min is still low compared to the alloy after annealed for 10 min, which can be seen from figure 5. The microstructure of alloy is dominated by dynamic recovery, the average grain size of alloy almost does not change. When the alloy is annealed for 30 min, owing to the volume fraction of recrystallized grains of alloy increasing, which lead to the grain size of big grains decreasing. however, the grain size of small grains increases due to long time annealing, finally, the average gain size of alloy increases. When the alloy is annealed for 40 min, the volume fraction of recrystallized grains of alloy increases, the grain size of big grains decreases sharply, but the grain size of small grains increases slowly, finally, the average gain size of alloy decreases.

4.4. The effect of annealing time on mechanical properties
After the alloy is annealed for 10 min, the tensile strength and yield strength of Mg–6Sn–3Al–1Zn alloy decreases obviously compared to as-rolled alloy, because occurring dynamic recovery after annealed, lead to the work hardening phenomenon is eliminated. When 10 min ≤ annealing time ≤ 30 min, tensile strength and yield strength increases with the increasing of annealing time, owing to the volume fraction of Mg2Sn phase increases. When annealing time ≤ 30 min, the volume fraction of recrystallization increases, and average grain size of alloys increases leads to the elongation increases. After the alloy is annealed for 40 min, the average grain size of grains decreases, the volume fraction of Mg2Sn phase increases, which lead to the tensile strength increases, basal texture decrease lead to the tensile strength decreases, finally, the tensile strength decreases a little. In the multi-modal gain distribution microstructures, large grains provide plasticity, small grains provide strength, however, after the alloy is annealed for 40 min, the average grain size of the unrecrystallized large grain decreases, lead to the elongation of alloy decreasing seriousness.

5. Conclusions
The alloy with bimodal microstructures was annealed for 10 min, 20 min, 30 min and 40 min at 350 °C, respectively. The results showed that:

(1) With the annealing time increasing, aging precipitation lead to the volume fraction of Mg2Sn phase increasing. The volume fraction of recrystallized grains increases. When the annealing time is less than or equal to 20 min, the microstructure was dominated by dynamic recovery, and the intensity of {0001} texture decreased. However, when the alloy was annealed for 30 min, the microstructure was dominated by dynamic recrystallization, and the intensity of {0001} texture increased. The {0001} texture intensity was reduced after annealing for 40 min, which was led by further dynamic recrystallization.

(2) Multi-modal microstructures composed of small (grain size <10 μm), middle (10 μm < grain size <30 μm), and big grains (grain size >30 μm) were formed in the annealed alloy.
(3) When the alloy was annealed for 30 min, the mechanical properties reached optimum, the tensile strength, yield strength and elongation was 336 MPa, 194 MPa and 26%, respectively.

Acknowledgments

This work is supported by the Natural Science Foundation of Qinghai (Grant No. 2020-ZJ-907).

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