Effect of solid solution heat treatment following age hardening on microstructure and mechanical properties of 7000 series power aluminum alloy

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Abstract
In this paper, Al-11.1Zn-2.7Mg-1.7Cu-1.4Ti-0.2Zr alloy material was prepared by powder metallurgy to study the effect of the different solid-solution aging systems on the microstructure and mechanical properties of this 7000 series aluminum alloy. The microstructure of the alloy under the three different solid-solution processes S1, S2, and S3 were observed using an optical microscope, scanning electron microscope, and x-ray diffraction analysis. The mechanical properties were tested to determine the best solid-solution process. The results show that under the S2 solid-solution process, the alloy has the best mechanical properties, with a compressive strength of 646.7(±6.5) MPa and a fracture strain of 5.9(±0.06)%.

1. Introduction
7000 series aluminum alloy is widely used in aerospace, automobile, railway transportation, and other fields due to its low density, high strength, and better processing performance [1–5]. At present, the most popular methods for preparing 7000 series aluminum alloy include casting [6], injection molding [7], and powder metallurgy [8]. Among them, powder metallurgy is widely used in the preparation of high-performance aluminum alloy materials [9] due to its low cost and simple processing route. It is also possible to control porosity and prepare homogeneous alloy materials by adjusting element composition and sintering parameters [10, 11]. Solution-aging treatment after powder metallurgy has a significant influence on the microstructure and mechanical properties of alloy materials. Time and temperature are important parameters in solution-aging treatment. Solution-aging treatment under reasonable parameters makes the mechanical properties of the sample better than the original sample [12–14]. Many scholars have studied the effect of the solution-aging system on the microstructure and mechanical properties of the alloy. Guo Fengbin et al [15] showed in the study of microstructure and mechanical properties of 7A56 aluminum alloy after solid-solution treatment that the best solid-solution process is 470 °C × 4 h. The conductivity after quenching was about 30.8%IACS, and the hardness was 204HV. Shang Baocnuan et al [16] studied the effect of solution-aging on the microstructure and properties of the 6082 alloy extruded bar. The results show that the tensile strength, yield strength, and elongation of 6082 alloy extruded bar were 367 MPa, 341 MPa, and 14.1% respectively after solution quenching at 545 °C × 50 min and aging at 170 °C × 8 h. Wang Weiwei et al [17] studied the effect of heat treatment on the mechanical properties of thixotropic 7A09 aluminum alloy. The results show that better mechanical properties...
are obtained after solid-solution at 475 °C × 12 h and aging at 133 °C × (20–22) h. The tensile strength was about 540 MPa, and elongation was more than 6.3%. However, solid-solution-aging has little research on the properties of aluminum alloy materials prepared by powder metallurgy. The purpose of this paper is to study the effects of three different solid-solution processes S1, S2, and S3 on the microstructure and mechanical properties of 7000 aluminum alloy prepared by powder metallurgy.

2. Materials and experimental methods

2.1. Preparation of Experimental Materials

The alloy composition designed in this experiment is Al-11.1Wt% Zn-2.7Wt% Mg-1.7Wt% Cu-1.4Wt% Ti-0.2Wt% Zr. The adopted powder metallurgy process flow is as follows: cold pressing forming of alloy powder → sintering under argon protection → hot extrusion → solid-solution and aging. Relevant parameters of the experimental process include; cold pressure of 740 MPa, sintering temperature of 550 °C under an argon atmosphere, hot extrusion ratio of 7:1, and extrusion temperature of 420 °C. In the experiment, the sample taken after the extrusion has a size of φ8 × 12 mm. The sample was then ground and polished: starting from 180# sandpaper to 2500#, followed with polishing on a polishing machine, and finally washed with alcohol and dried.

2.2. Experimental method

In the experiment, the hot extruded sample was analyzed using the DSC comprehensive thermal analyzer, and the approximate solution temperature of aluminum alloy in the resistance furnace determined according to the DSC curve. Experimental parameters are; heating rate 10 °C min⁻¹ and test temperature range 0 ~ 700 °C. The solid-solution process adopts the enhanced solid-solution process. The specific process was set according to the DSC thermal analysis curve of the alloy. The DSC test curve is, as shown in figure 1(a). There is an obvious endothermic peak when the temperature rises to 470 °C, which is very important for the formulation of the solid solution process. The solid-solution process of S1 was 450 °C × 2 h + 460 °C × 2 h, S2 was 450 °C × 2 h + 460 °C × 2 h + 470 °C × 2 h, and S3 was 450 °C × 2 h + 460 °C × 2 h + 470 °C × 2 h + 480 °C × 2 h as shown in figure 1(b). The aging temperature was 121 °C, and the hardness was measured at intervals to find the best aging time. This experiment was to test the microscopic structure and performance of the samples obtained.

2.3. Performance analysis

The polished sample was corroded for 15–30s in metallographic corrosive liquid. The metallographic structure morphology was then observed under an optical microscope (OM). The metallographic etching solution was the Graff Sargent solution (1 ml HF + 16 mL HNO₃ + 3g CrO₃ + 83 ml H₂O). Scanning electron microscope (Equipped with EDS), JSM-IT300 model, was used to test and analyze the surface structure of the material, by mainly analyzing the composition of the precipitated phase of particles and the changing trend of undisolved phase. The aluminum alloy was analyzed using a D8-ADVANCE x-ray diffractometer. The detailed test parameters were set as follows: Cu-K ray (λ = 0.15406 nm), scanning rate 5 °/min, and the scanning range 0 ~ 90°. XRD data were processed and analyzed by Jade software. The hardness of the sample was tested by a digital microhardness tester model HV–1000. Experimental parameters were: loading force 300gf and testing time 15s. Model 7051 eddy current conductivity meter was used to test the conductivity of the pattern.
compression experiment was conducted at room temperature using an electronic universal testing machine model DDL100. Experimental parameters were: compression rate 0.5 mm min\(^{-1}\) and a compression size of \(\phi 8 \times 12\) mm.

3. Results and discussion

3.1. Microstructural observations

The morphology of the sintered sample under the scanning electron microscope is as shown in figure 2(a). EDC energy spectrum collection carried out in figure 2(a), and the result is as shown in figure 2(b). Then, EDS energy spectrum collection also carried out on a small area selected from figure 2(a), and the result is as shown in figure 2(c). The energy spectrum collection results show that the two energy spectrum collections are similar, and the peak values of the curves are approximately the same. It can be concluded that the alloying degree of the sample was very high.

Figure 3 is the result of surface scanning of the localized region of aluminum alloy in the sintered state, and the distribution of several elements of Al, Zn, Mg, and Ti in the material as analyzed. From the scanning area, the Zn and Mg elements are uniformly distributed in the alloy, and there is no element segregation phenomenon, indicating that the metal alloying degree is high after sintering. However, the Ti was partially agglomerated, which may be due to the relatively high melting point of Ti. The low sintering temperature adopted in the experiment made the fluidity of Ti in the alloy weakened, thus agglomerated together. It may affect the precipitation strengthening effect of the precipitation phase in the later stage.

Because the sintered material has a loose structure and low density, to make the structure more compact, hot extrusion was carried out. Figure 4(a) shows the microstructure of the alloy after extrusion. By comparing the distribution of undissolved phases in the alloy before and after hot extrusion, it is found that the phase composition has not changed during hot extrusion. It can be seen that their microscopic morphology has a wide range of fine white particles, a few small black particles, and a partially coarse gray bar phase, which has effects on the distribution of the undissolved phase.

To study the composition of these phases, EDS analysis was carried out on the undissolved phase in figures 4(b)–(d). It was found that the gray block (position 1) in the figure was Ti-rich phase, the white particles with smaller size and more distribution (position 2) were Zn-rich, and Mg-rich phases precipitated in large quantities, and the black particles with a smaller number (position 3) were a few impurity phases. And from the line scanning results in figures 4(e) and (f), it can be seen that there are Al, Mg, Ti, Cu, Zn, and other elements distributed on the line. When passing through the gray zone, the Al element decreases and the Ti element increases greatly, further indicating that the gray zone is the Ti-rich phase. The undissolved phase belongs to a layered structure, the outer layer is mainly Al atoms, and the inner layer is Ti atoms.

The alloy was subjected to solid-solution treatment after extrusion. Figure 5 is an SEM image of the alloy under different heat treatments. As can be seen from the figures, with the increase of temperature, the number of precipitated particles produced by hot extrusion in the material decreases significantly. After the S1 process, the fine particle phase was basically dissolved and the number obviously reduced, leaving only some insoluble coarse phase. After the S2 process, the fine particle phase is further solid dissolved into the matrix, and the insoluble coarse phase is also completely solid dissolved. After the S3 process, the number of undissolved phases has not changed significantly, which may be that after S2 treatment, it is already close to the saturation degree of alloy
solid-solution, and S3 is to further increase the solid-solution temperature and time, based on S2. At the same time, the solid-solution effect was greatly reduced, and the growth of the second phase begins to occur in the alloy. The number becomes more due to the high solid-solution temperature and the long solid-solution time of S3.

3.2. XRD analysis

Figure 6 shows XRD patterns of the extruded alloy after S1, S2, and S3 processes (including unheated treatment). It was found that all the XRD patterns of the alloy showed Al peaks, and there were no Zn and Mg diffraction peaks, which indicated that the degree of alloying was very high and supersaturated solid-solution of Al matrix formed.

As can be seen from table 1, the half-peak width of the alloys treated with three solid-solutions was significantly lower than that of the non-treated alloys. The half-peak width of the alloys treated with solid-solutions was highest in S2 and lowest in S3. Table 1 also shows the fitting relationships between $(\delta^2 \theta)^2 / \tan^2 \theta_0$ and $\delta \theta / \tan \theta_0 \sin \theta_0$ of the PM aluminum alloy under different solid-solution processes.

According to the XRD analysis spectrum, the Hall-Williamson mathematical model can be used to calculate the lattice strain ($e^2$) and dislocation density ($\rho$) inside the material. Its main parameters, coherent diffraction zone size ($d$), lattice strain ($e^2$), angular position of main highest diffraction peak ($\theta_0$), half-peak width ($\delta \theta$), and XRD detection ray wavelength ($\lambda$) have a functional relationship as in equation (1) [18, 19]:

$$\frac{(\delta^2 \theta)^2}{\tan^2 \theta_0} = \frac{\lambda}{d} \left( \frac{\delta \theta}{\tan \theta_0 \sin \theta_0} \right) + 25 \langle e^2 \rangle^2$$  \hspace{1cm} (1)

After deep processing of the data, the linear relationship between $(\delta^2 \theta)^2 / \tan^2 \theta_0$ and $\delta \theta / \tan \theta_0 \sin \theta_0$ was obtained, as shown in figure 6. According to the intercept ($25 \langle e^2 \rangle$) and slope ($\lambda / d$) of the fitting straight line, the values of $d$ and $e^2$ can be obtained. The calculation of $\rho$ can adopt the mathematical formula in equation (2):

$$\rho = 2 \sqrt{3} \langle e^2 \rangle^{1/2} / (d \times b)$$  \hspace{1cm} (2)

The Berth’s vector (b) of aluminum is 0.286 nm. The dislocation strength $\sigma_p$ can be calculated by the mathematical formula in equation (3):

$$\sigma_p = M \alpha G b^{1/2}$$  \hspace{1cm} (3)

The values of M, $\alpha$ and G in the formula are 3.06, 0.24, and 26 GPa [20] respectively.
Table 2 shows the calculated values of the dislocation density and dislocation strength of the alloy. According to the data in the observation table, the dislocation density of the alloy after extrusion is the largest with a value of 1.3675 \times 10^{14} \text{ m}^{-2}. This is due to the change of grains in the material caused by hot extrusion and the appearance of many blocky grains, which introduces more dislocations. The strength provided by the dislocations is also highest, with a value of 63.861 \text{ MPa}. After S1, S2, and S3 treatments, the dislocation density decreased significantly, with the values of 0.5676 \times 10^{14} \text{ m}^{-2}, 0.5966 \times 10^{14} \text{ m}^{-2}, and 0.4012 \times 10^{14} \text{ m}^{-2}, respectively. The strength provided by dislocations was 41.142 \text{ MPa}, 42.181 \text{ MPa}, and 34.591 \text{ MPa}, respectively, which was also significantly lower than that of the unsolvable alloy. However, after solid-solution, the dislocation intensity changes as S2 > S1 > S3, as shown in figure 7.

3.3. Hardness and conductivity of the alloy
In this experiment, the effects of different solid-solution processes and different aging time on the hardness of the alloy at a single-stage aging temperature of 121 °C were studied. The measured hardness values are as shown in table 3, and the hardness changing trend is plotted as a curve, as shown in figure 8(a). The hardness of alloy in
Figure 5. SEM image of the alloy: (a) unheated treatment; (b) S1 treatment; (c) S2 treatment; (d) S3 treatment.

Figure 6. (a) XRD patterns of unheated treatment; (b) XRD patterns of S1; (c) XRD patterns of S2; (d) XRD patterns of S3.
the extruded state without overheating treatment is 114.3 (±2.9) HV, but after S1, S2, and S3 treatment, the hardness is significantly improved, and the solid-solution strengthening effect is distinct. The hardness value under S2 treatment reaches the maximum value of 175.3 (±4.4) HV, which indicates that S2 treatment has the best solid-solution strengthening effect. After aging, the hardness of the alloy is obviously improved, which indicates that the aging hardening effect of the material is remarkable. The changing trend of hardness was

Table 1. The fitting relationships between $(\delta2\theta)^2/\tan^2\theta_0$ and $\delta2\theta/\tan\theta_0 \sin\theta_0$ and FWHM of PM Aluminum Alloy under Different Solid Solution Processes.

| Solid solution process | Half-peak width FWHM | The fitting relationships between $(\delta2\theta)^2/\tan^2\theta_0$ and $\delta2\theta/\tan\theta_0 \sin\theta_0$ |
|------------------------|-----------------------|-----------------------------------------------------------------------------------|
| Unsolved               | (Unsolved)            | (Unsolved)                                                                        |
| S1                     | (S1)                  | (S1)                                                                               |
| S2                     | (S2)                  | (S2)                                                                               |
| S3                     | (S3)                  | (S3)                                                                               |

The changing trend of hardness was...
observed. It found that the hardness of the alloy increased sharply after aging for 3 h under different solid-solution processes, and fluctuated within an interval after 6 h. A hardness peak value of 210.8 ($\pm$ 5.3) HV appears in the alloy after aging for 15 h after S1. The hardness peak value of 220.7 ($\pm$ 5.5) HV of the alloy appears after aging for 18 h after S2. The hardness of the alloy showed a decreasing trend after aging for 6 h by S3 and reached a peak value of 216.7 ($\pm$ 5.4) HV after aging for 18 h.

![Figure 7](image1.png)

**Figure 7.** Calculation of dislocation density and dislocation strength under different solution processes.

![Figure 8](image2.png)

**Figure 8.** (a) variation curve of alloy hardness with time after solution treatment; (b) conductivity of alloy under heat treatment process.

| Table 2. Parameters obtained from XRD data. |
|---------------------------------------------|
| Solid solution process | d/\text{nm} | $\langle c^2 \rangle^{1/2}$ | $\rho/(10^{14} \text{ m}^{-2})$ | $\sigma_p/\text{MPa}$ |
|-------------------------|-------------|----------------|-----------------|-------------------|
| Unsolved                | 79.824      | 9.012E-4       | 1.3675 $\pm$ 0.068 | 63.861 $\pm$ 3.193 |
| S1                      | 114.970     | 5.387E-4       | 0.5676 $\pm$ 0.028 | 41.142 $\pm$ 2.057 |
| S2                      | 63.925      | 3.149E-4       | 0.5966 $\pm$ 0.030 | 42.181 $\pm$ 2.109 |
| S3                      | 63.399      | 2.100E-4       | 0.4012 $\pm$ 0.020 | 34.591 $\pm$ 1.730 |

| Table 3. Compressive Properties of alloys with different heat treatments. |
|---------------------------------------------|
| Solid solution process | $R_{p0.2}$/MPa | $R_{mc}$/MPa | $\varepsilon_c$/% |
|-------------------------|----------------|--------------|-------------------|
| S1                      | 588.7 $\pm$ 5.9 | 643.1 $\pm$ 6.4 | 7.0 $\pm$ 0.07 |
| S2                      | 601.3 $\pm$ 6.0 | 646.7 $\pm$ 6.5 | 5.9 $\pm$ 0.06 |
| S3                      | 496.5 $\pm$ 5.0 | 586.2 $\pm$ 5.9 | 7.3 $\pm$ 0.07 |
Comparing the hardness of the three different solid-solution processes after aging, it found that the hardness of the alloy under the S2 solid-solution process is the largest after aging for 6 h. Because the number of solute atoms dissolved into the matrix in S1, S2, and S3 processes is different, it makes the number of strengthening phases produced after aging differently. The highest temperature of S1 treatment is obviously lower than at 474.7 °C, so the solid-solution effect is poor, the number of strengthening phases after aging is small, and the hardness is not high. S2 treatment has a suitable solid-solution temperature \( (470 °C) \) and a sufficient solid-solution time \( (6h) \), which makes the solid-solution degree high and the effect better. The main alloying elements are basically solid dissolved into Al, thus the number of hardened phases formed after aging is large, and the hardness value of the alloy is high. Due to the high solid-solution temperature and long solid-solution time of S3 treatment, local grain size may grow up obviously, which leads to the decrease of hardness and affects the performance of the material.

In this experiment, the conductivity of alloy under the 121 °C × 6 h aging system was tested, which was converted into the standard measurement value of international annealed copper, as shown in figure 8(b). It can be seen from the figure that the electrical conductivity of the alloy after aging in three different solid-solution processes is 22.663%IACS, 22.179%IACS, and 21.971%IACS, respectively, with a trend of gradual decrease. The change of electrical conductivity is related to the distortion degree in the material. The distortion degree determines the resistance value of the material, thus affecting the electrical conductivity. The higher the degree of solid-solution, the more solute atoms incorporated into the matrix, and the higher the distortion energy formed, thus increasing the probability of electron backscattering, resulting in increased resistivity and decreased conductivity. At the same time, the presence of some mesophase also leads to a decrease in electrical conductivity in the solid-solution process.

### 3.2. Compressive mechanical properties of the alloy

Table 4 shows the compression properties of the alloy after aging for 6 h, under different solid-solution processes. Figure 9(a) is a diagram of its compressive stress and strain. It can be seen from table 4 and figure 9(b) that alloy aged for 6 h and after S1, S2, and S3 treatment, has yield strengths \( R_{p0.2} \) is 588.7(±5.9) MPa, 601.3(±6.0) MPa, and 496.5(±5.0) MPa, respectively. It also has a compressive strength \( R_{mc} \) of 643.1(±6.4) MPa, 646.7(±6.5) MPa, and 586.2(±5.9) MPa, respectively and a fracture strain \( \varepsilon_c \) of 7.0(±0.07) %, 5.9(±0.06) %, and 7.3(±0.07) %, respectively. The compressive strength of PM aluminum alloy first increases and then decreases. After S2 solid-solution process, the maximum compressive strength is 646.7(±6.5) MPa. However, the compressive strength of the alloy decreased significantly under the S3 solid-solution process. The fracture strain of PM aluminum alloy is opposite to the changing trend of strength, reaching a maximum of 7.3(±0.07) % under the S3 solid-solution process. To sum up, comparing the compressive mechanical properties of the different heat treatments, the alloy treated by S2 solid-solution has better comprehensive properties, with the compressive strength 646.7(±6.5) MPa and the fracture strain 5.9(±0.06) %.

### 4. Conclusions

In this paper, the microstructure and related mechanical properties of 7000 series aluminum alloy under different solution and aging systems at 550 °C sintering temperature were studied. It can be seen that under

![Figure 9. (a) Compression stress-strain diagram of alloy after different heat treatments; (b) Compressive strength and fracture strain diagram of alloy after different heat treatments.](image-url)
Table 4. Hardness of alloys under different heat treatment processes.

| Aging time /h | 0     | 3     | 6     | 12    | 15    | 18    | 21    | 24    |
|---------------|-------|-------|-------|-------|-------|-------|-------|-------|
| S1 Solid solution hardness /HV | 167.6 ± 4.2 | 203.3 ± 5.1 | 210.7 ± 5.3 | 208.8 ± 5.2 | 210.8 ± 5.3 | 208.3 ± 5.2 | 207.6 ± 5.2 | 201.5 ± 5.0 |
| S2 Solid solution hardness /HV | 175.3 ± 4.4 | 205.8 ± 5.2 | 217.2 ± 5.4 | 219.2 ± 5.5 | 217.8 ± 5.5 | 220.7 ± 5.5 | 218.5 ± 5.5 | 212.1 ± 5.3 |
| S3 Solid solution hardness /HV | 173.8 ± 4.3 | 209.3 ± 5.2 | 215.8 ± 5.4 | 213.8 ± 5.4 | 214.3 ± 5.4 | 216.7 ± 5.4 | 213.3 ± 5.3 | 197.3 ± 4.9 |
different solid-solution systems, the evolution of some phases and the changes in mechanical properties occurred. The relevant conclusions are as follows:

(1) When prepared the aluminum alloy samples by powder metallurgy at a sintering temperature of 550 °C, the alloying degree of metallic elements was high, and there was no distinct element segregation.

(2) After S1 treatment, a large number of precipitated particles formed by hot extrusion were mostly solid-dissolved into the matrix. After S2 treatment, the solid-solution degree was close to saturation. The solid-solution strengthening effect was the best, and the dislocations contributed the most to the strength. After S3 treatment, the growth of the second phase began to appear in the alloy, and the number gradually increased. Therefore, the best solid-solution system was S2 treatment.

(3) After the 3 solid-solution treatments, the hardness increased rapidly after aging for 3 h and fluctuated at a specific value after aging for 6 h. The maximum hardness trend of the three solid-solution processes after aging was S2 > S3 > S1. The maximum hardness (220.7 ± 5.5) HV occurred after S2 treatment and 121 °C × 18 h aging.

(4) The compressive strength first increased and then decreased with the increase of solid-solution time and temperature. Compressive fracture strain had the opposite rule. Under S2 treatment, the alloy had better comprehensive properties. Its compressive strength was 646.7 (±6.3) MPa, and fracture strain was 5.9(±0.06) %.

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