Microstructure and properties of a new Al–Cu–Mg–Zn–Zr–Ti heat resistant aluminum alloy

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Abstract
The effects of adding Ti with 0, 0.47 and 0.89 (wt%) on the microstructure and properties of the new Al–2.87Cu–3.83Mg–2.91Zn–0.2Zr–xTi cold extruded heat-resistant aluminum alloy materials are studied. The results show that after cold extrusion, 480 °C × 2 h solution treatment and 120 °C × 12 h aging, with the increase of Ti content, the internal undissolved phase of the alloy increase, grain refine, room temperature strength and heat resistance also improved. The alloy with 0.89Ti has the highest strength, and its hardness and electrical conductivity are 160.8HV and 23.56% IACS, respectively. Compared with the alloy without Ti, the tensile strength at room temperature is increased by 73.08 MPa, reaching 471.55 MPa, and the elongation after fracture is 16.67%. The peak value of hot compression at 250 °C increase by 86.57 MPa, reaching 326.66 MPa. At the same time, the alloy also has good resistance to intergranular corrosion, and the maximum intergranular corrosion depth is 146.31 μm.

1. Introduction
Because of the excellent mechanical properties at room temperature (strength, plasticity and hardness, etc) and good mechanical properties at high temperature (high temperature resistance and diathermancy), 2xxx series aluminum alloys have been widely used in all kinds of aviation industry and modern automobile fields in the world [1–3]. Heat-resistant aluminum alloy can work at high temperature and stress for a certain time with the mechanical properties and oxidation resistance of the alloy do not decrease obviously. Because of the excellent properties of light weight, good machinability and especially the high strength at elevated temperature [4], the requirements of heat resistant aluminum alloy became more stringent. Most aluminum alloys were composed of the matrix and the second phase. The second relative phase precipitated in the alloy plays a decisive role in the property, both at room temperature and at high temperature. When the ambient temperature of the workpiece increases, the original characteristics of the internal matrix and the second phase will be changed, which will greatly affect the comprehensive properties of the material in all aspects. Therefore, scholars usually improve the heat resistance of the alloy by enhancing the high temperature resistance of the matrix itself and changing the proportion and quantity of the second phase in the matrix [5].

In 2xxx aluminum alloy, the main strengthening phases are θ phase (Al1Cu), S phase (Al1CuMg) and T phase [Mg17(Al, Zn)12], some β phases (Mg2Al4) will also be precipitated, among which S phase has the best strengthening effect, followed by θ phase, and S phase has high room temperature strength and high heat resistance [2, 6]. For T and β phase, β phase and aluminum alloy matrix are difficult to coherent, the size of precipitated phase is large and nucleation is difficult. When electrochemical corrosion is formed, the electrochemical potential is positive electrode, the alloy is negative electrode, electrochemical reaction occurs, which provides a necessary condition for intergranular corrosion, and finally affects the corrosion resistance of the material. However, the potential difference of T phase is similar to that of matrix Al, which can improve the corrosion resistance of alloy [7].

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Annealing is needed. In this article, the homogenization annealing system was used to cut the sample after the homogenization to a bar stock of 31.6 mm, the samples in this study were designed as Al-2.87Cu-3.83Mg-2.91Zn-0.2Zr-xTi, with x being the proportion of elements and selecting the best solid solution and aging process to change the proportion and quantity of aging precipitated phases. Based on these principles and combined with the previous research of Liu et al., the microstructure, mechanics and temperature resistance of aluminum alloys have been further studied, and the results showed that the addition of Ti can refine the grain structure and inhibit the dynamic recrystallization.

2xxx series aluminum alloy can improve the comprehensive properties through technology. Therefore, the high temperature resistance and the mechanical properties of the alloy can be improved by optimizing the proportion of elements and selecting the best solid solution and aging process to change the proportion and quantity of aging precipitated phases. Based on these principles and combined with the previous research of 2219 aluminum alloy studied by Fu as well as the phase analysis of aluminum alloy, the composition is designed as Al-2.8 ~ 3Cu-3.6 ~ 4Mg-2.8 ~ 3Zn-0.18 ~ 0.21Zr-xTi. On the basis of the composition in this paper, the microstructure, mechanics and temperature resistance of aluminum alloy have been further studied, which will provide a theoretical basis for the development of aluminum alloy with higher temperature resistance and stronger corrosion resistance as well as higher tensile strength.

### 2. Experimental

The samples of the three alloys adopted in this article were self-designed components, and EDS and spectrometers have been used to measure the actual components of the alloy samples. The contents of the components are shown in Table 1. For convenience, the samples were marked as C1, C2 and C3. No. Cu Mg Zn Zr Sr Ti Al

| No. | Cu    | Mg    | Zn    | Zr    | Sr    | Ti    | Al   |
|-----|-------|-------|-------|-------|-------|-------|------|
| C1  | 2.85  | 3.84  | 2.89  | 0.20  | 0.11  | 0     | Bal. |
| C2  | 2.84  | 3.83  | 2.95  | 0.18  | 0.10  | 0.47  | Bal. |
| C3  | 2.92  | 3.81  | 2.91  | 0.21  | 0.10  | 0.89  | Bal. |

Table 1. Actual composition of C1 ~ C3 alloys (wt%)
diameter of 12 mm was obtained after the extrusion is successful. Considering the high degree of alloying of Mg and Zn in this alloy, the solution temperature of the alloy was lower than that of the other 2xxx aluminum alloy, and the solution treatment system selected as 480 °C for 2h, and the aging time is about 12h at 120 °C.

The microstructure of the specimens and dislocation enhancement were observed on a scanning electron microscope (SEM) and x-ray diffraction (XRD) respectively to study the grain boundaries and adjacent regions as well as the phases of the three samples. Hardness tests were carried out on an HV-1000 hardness tester with a load of 0.2 kg at the sample with a size of 10 mm length × 10 mm width × 5 mm thickness, the dwell time was 25s and 3 ~ 5 measurements were made for each sample to obtain an average value. Tensile properties were tested at room temperature at a strain rate of 0.5 mm min⁻¹, and three measurements were tested for each sample. An optical metallurgical microscope was used to obtain the metallographs. The metallographic corrosive solution was composed of 2 ml HF + 5 ml HNO₃ + 3 ml HCL + 190 ml H₂O in order to study the grain structure. The intergranular corrosion solution was composed of NaCl 57 g L⁻¹ + H₂O₂ 10 ml L⁻¹.

The samples of the hot compression test were the samples after solution aging. The size of the sample was a cylinder with a diameter of 10 mm and a height of 15mm. In the process of machining, it was necessary to keep the two circular surfaces parallel and ensured that the height: the diameter was 3 : 2. Then the hole with diameter 0.5mm was machined by EDM in the middle of the height direction of the samples, and the depth of the hole was ≥0.5 d (d is the diameter of the bottom of the sample). After the sample was processed, it will be put on the thermal simulation test equipment to carry out the experiment. The deformation temperature was 250 °Cand the deformation amount was 50%. The deformation rate was 1s and the temperature was about 50 s, the heat preservation used for compression was 4 ~ 6 min, and the water cooling is carried out after the end. The device model is Gleeble-3500.

The electrical conductivity was measured on the 3 specimen for each condition for 4 ~ 6 times. The conductivity measured was present in the unit of % IACS(International Standard,1% IACS = 0.58 MS⁻¹, Mean value was calculated to describe the conductivity of the alloy.

3. Results and discussion

3.1. SEM analysis

The transverse SEM images of the C1, C2 and C3 alloys were shown in figure 1, and the three samples were solution treated samples. It was found that there were some unsoluble phases in the three pictures, while the proportion of undissolved phases in the figure 1(a) was the least. With the increase of the Ti content, the proportion of undissolved phases in the matrix increased, and some of the elongated, white, coarse phases (figures 1(b) and (c)) showed that the solid solubility of Ti in this series of aluminum alloys was slowly saturated.

The undissolved phase of the matrix above was analyzed by EDS. The results in figure 2 showed that the insoluble phase in region A is fine circular gray, and the main element is Al, Cu and Mg. It can be calculate from the ratio of the element that the undissolved phase is S phase (Al₂CuMg) and S phase is high temperature strengthening phase, which can improve the heat resistance of the alloy. The gray-white region B was insoluble rich Ti phase (such as Al₃Ti). These coarse insoluble phases in the matrix were easy to cause stress concentration and affect the mechanical properties.

3.2. Metallographic structure analysis

Figure 3 was a metallographic corrosion diagram of C1, C2 and C3 alloys. After the solution treatment at 480 °C for 2h, samples were immersed in the metallographic etching solution for 40s and then observed by the metallographic microscope.

It could be found from the diagram that when Ti is not added, the grains (figure 3(a)) of the alloy express irregular shape and interlaced with each other, and the recrystallizing is not obvious, but some of the grains also grow abnormally. With the increase of Ti, it was found that the grain refinement of the alloy is more obvious, and the grain size in figure 3(c) is the smallest, which is about 133 μm (figure 4). However, when the content of Ti increased, the coarse black insoluble second phase of the alloy also increased, and the particles of coarse Al₂Ti appeared on the surface (figure 3(c)) after corrosion, which would affect the mechanical properties of the alloy, indicating that the content of Ti should not be added too high in aluminum alloy. Therefore, if the content of Ti was further increased on the basis of this design component, the effect of grain refinement was not too obvious, and the undissolved phase in the alloy will be further increased, thus it would have an opposite effect of the alloy on the properties [16].

3.3. XRD analysis and dislocation enhancement

Figure 5 showed the XRD and half-peak width of C1, C2 and C3 alloys after 480 °C × 2 h solid solution, figures 5(a), (c), (e) were the analysis maps of C1, C2 and C3 respectively. Figures 5(b), (d), (f) were the
corresponding half-peak widths of each alloy. The XRD spectra of C1, C2 and C3 alloys were compared with those of pure Al (figure 6). It was found that the three alloys were quite different from pure Al, indicating that the texture ratio of C1, C2 and C3 alloys increased after Mg, Zn alloying. Compared with the half peak width of the three alloys, the half peak width of C1 alloy was the smallest, which indicates that Ti has a certain effect on the texture and lattice strain of the series of alloys.

According to the above data, the dislocation density and the corresponding dislocation strengthening contribution value of the three materials in this article can be obtained. The coherence diffraction region size (d), the lattice strain (\(\varepsilon^2\)) and the half-peak width (FWHM: obtained from the XRD data) (\(\delta2\theta\)), the angular position of the highest peak of the diffraction peak (\(\theta_0\)), the x-ray wavelength (\(\lambda\)), relationships of the physical
The value of \( \theta_0 \) and FWHM (\( \delta 2\theta \)) can be obtained according to figure 5, and these data are then used to fit the functional diagram of the three alloys of figure 7. The coherence diffraction region size (d) and the mean lattice strain (\( \langle e^2 \rangle^{1/2} \)) of the XRD are calculated according to the slope of the straight line in figure 7 and the intercept parameter at the time of the intersection with the Y-axis. As can be known from the following equation, the greater the lattice strain, the greater the dislocation density in the alloy [19].

\[
\rho = 2\sqrt{3} \langle e^2 \rangle^{1/2} (d \times b)
\]

(2)

The berber vector (b) of aluminum alloy in this formula is 0.286 nm. Thus, the dislocation density (\( \rho \)) of the alloy is obtained. The dislocation strengthening (\( \sigma_s \)) can be calculated from the following equation [20]:

\[
\sigma_s = MGb/\rho^{1/2}
\]

(3)

In the formula, M is a Taylor position factor, the value is 3.06. \( \alpha \) represents the numerical factor and the value is 0.24; G represents the shear modulus and the value is 26 GPa. The related dislocation parameters are calculated through the above three equations in table 2.

From the data in figure 7 and table 2, it could be seen that the dislocation parameters after adding 0.49% Ti were lower than that without Ti. With the increase of Ti element, the strengthening effect of dislocation density decreased first and then increased, which indicates that the dislocation contribution value was decreased when the alloy sample was treated with solid solution [21]. When the content of Ti was further increased, the dislocation density increased by 0.89 \( \times \) 10\(^{-4} \)%. The strength contribution value is 45.08 MPa, which indicates that the consumption of dislocation in solid solution treatment will be suppressed when the content of Ti was added to a certain range. In addition, because the Ti content of C3 alloy was too high, the lattice strain was the largest, and the hardenability of the alloy reduced.

### 3.4. Hardness and conductivity

For 2xxx aluminum alloy, mechanical properties are one of the most important properties, such as hardness, conductivity, yield strength, tensile strength and extension after break. In order to explore the mechanical
properties of the new heat-resistant alloy after Mg and Zn alloying, it is necessary to explore its aging system. According to the phase-temperature simulation analysis of the software and the research results on 7xxx, 6xxx, 5xxx and 2xxx, it is preliminarily determined that the aging system of the alloy adopts 120 °C × 12 h, 155 °C × 12 h, 190 °C × 12 h, respectively to select the optimum aging system. Figure 8 showed the hardness change curve of the C1, C2 and C3 alloys under the three aging systems, and table 3 showed the hardness of the three alloy aging systems and the values of the conductivity after the peak aging.

It could be found from table 3 that the hardness of the three alloys increased with the increase of Ti content under the three aging conditions, which was consistent with the test results of grain size in figure 3 that the smaller the grain size, the better the mechanical properties of the alloy, the higher the hardness value. After aging at 120 °C for 12 h, the hardness of the alloy increased the most, more than 15%, and the increase of C2 was about 24%, which was 155.63 HV. After the aging treatment of the alloy at 155 °C for 12 h and 190 °C for 12 h, the hardness of the alloys increased slowly and the overaging occurred due to the high degree of alloying of the Mg and Zn of the C1 to C3 alloys. Precipitation of MgZn2 separated out at the time of aging at 120 °C, so that the hardness was increased faster [22]. Thus, the aging system of this series of alloys was selected to be 120 °C for 12 h.

The higher the conductivity, the better the stress corrosion resistance. After the Ti element was added, the difference of conductivity was not obvious, only slightly decreases, the conductivity of the C3 alloy was only reduced by 0.49% IACS, which was due to the high Zn content of the alloy, the stress corrosion resistance of the alloy has been greatly improved [12].

3.5. Room temperature tensile properties and tensile fracture analysis

The yield strength, tensile strength and elongation at break of C1, C2 and C3 alloys at 120 °C for 12 h were measured in this article, and the specific values of the mechanical properties were shown in table 4.

The data in table 4 found that after the alloying of Mg and Zn, the strength of the series of aluminum alloys compared with the other types of 2xxx aluminum alloy was improved, which is due to the addition of Mg and Zn boosts the produce of the phases like MgZn2, 5(Al3CuMg) and 7[Mg54(Al, Zn)69], so the strength was increased. Both the yield strength and the tensile strength increased with the increase of the Ti content, which was in accordance with the test results of the hardness values in table 3, where the highest strength was the C3 alloy with

![Figure 4. Grain size distributions and average grain size (a) C1; (b) C2; (c) C3.](image)
Figure 5. XRD spectrum: (a) C1; (c) C2; (e) C3; FWHM: (b) C1; (d) C2; (f) C3.

Figure 6. XRD spectrum for pure Al.
Figure 7. $(\delta\theta)^2 / \tan^2 \theta_d$ and $\delta\theta / (\tan \theta_d \sin \theta_d)$ linear fit relationship: (g) C1; (h) C2; (i) C3.

Table 2. Related parameters of dislocation.

| Alloy No. | $d$ (nm) | $(\varepsilon^2)^{1/2}$ (%) | $\rho$ ($10^{14} \text{m}^{-2}$) | $\sigma$ (MPa) |
|-----------|----------|-----------------------------|-------------------------------|----------------|
| C1        | 49.22    | 4.51E-04                    | 1.11                          | 57.55          |
| C2        | 47.70    | 1.73E-04                    | 0.44                          | 36.21          |
| C3        | 46.54    | 2.62E-04                    | 0.68                          | 45.08          |

Figure 8. Hardness curve of three aging states.
a yield strength of 365.03 MPa and a tensile strength of 471.55 MPa. Although the coarse particles which are difficult to dissolve increase with the increase of Ti element and thus affect the mechanical property, but the effect of refining crystal grains is very obvious (figure 3), and the final performance showed that the strength is slightly improved (figure 9). In addition, for this series of aluminum alloys, the plasticity is excellent. The extension at break of C1 alloy has reached 26%. Although the addition of Ti will affect the extension at break of the alloy, the extension at break of C3 alloy with the highest Ti content is also above 16%.

Figure 10 showed the tensile fracture analysis of C1, C2 and C3 alloys. Figures 10(a), (c), (e) were 500 times fracture morphologies that of C1, C2 and C3 samples under SEM, respectively, and figures 10(b), (d), (f) were 1000 times fracture morphologies that of C1, C2 and C3 samples under SEM, respectively. It could be seen from the diagram that the fracture surface of C1, C2 and C3 alloys is smooth and the dimples are obvious. The morphology of C1, C2 and C3 alloys was mainly composed of large dimples, small dimples and a small amount of tear edges. Compared with the three morphologies, it could be found that the fracture morphology of C1 alloy has many dimples and only a very small amount of tear edges, so the plasticity was the best. There were some small circular dimples around each of the larger dimples, which indicated that the alloy has a tendency of recrystallizing. With the increase of Ti content, the morphology dimples in figures 10(c) and (d) became less, and the average size of the dimples became larger and the plasticity decreased. The size of the morphology dimple of the C3 alloy was further enlarged, the tearing edge becomes more, the fracture surface was flat and the plasticity is further reduced. It can be concluded that with the increase of Ti content, the coarse phase transformation and plasticity inside the matrix decrease, which was consistent with the results of the mechanical properties of table 4.
3.6. Thermal compression thermodynamic simulation test

Figure 11 was the thermal simulation test curve of three kinds of alloys. The samples after solid solution were tested by 250 °C, the deformation was 50%, and the strain rate was 1s⁻¹. Table 5 showed the stress peaks of the three alloys after cold extrusion.

It could be seen from the figure 11 that when the strain stage of the three alloys occurs at the beginning of the strain stage, the true stress increases significantly, and after reaching the peak value, with the increase of the strain, the stress value no longer rises, and all of them have a slight downward trend, which shows that the dynamic softening of the alloy is stronger than that of the working hardening strengthening effect in the process of strain after the peak value. From the data in the table 5, it was known that the heat resistance of this series of aluminum alloys is better than that of 2219 aluminum alloy. Because of the higher degree of Mg alloying, it promoted the matrix to produce more temperature-resistant S phase, so it exhibited better temperature-resistant performance. Compared with the three alloys, it was found that with the increase of Ti content, the peak value of the alloy also increases, and the best heat resistance was C3 alloy, the peak value increase was 36.1%, which was 326.66 MPa, indicating that Ti can improve the heat resistance of this series of aluminum alloys.

![Figure 10. Tensile fracture analysis of alloys: (a), (b): C1; (c), (d): C2; (e), (f): C3.](image-url)
Figure 11. True stress-strain diagram of the alloys.

Table 5. True stress peaks of alloys.

| Alloy No. | True stress peaks/MPa |
|-----------|-----------------------|
| C1        | 240.09                |
| C2        | 302.46                |
| C3        | 326.66                |

Figure 12. Intergranular corrosion diagram of alloys: (a) C1; (b) C2; (c) C3.
alloys. Due to the higher solid solubility of Ti in this series of aluminum alloys, the proportion of Al₃Ti strengthening phase in this series of aluminum alloys was also higher, so the C3 alloy with the highest temperature resistance with the highest Ti content.

3.7. Resistance to intergranular corrosion

The intergranular corrosion diagrams after soaking in intergranular corrosion solution for 6 h of C1, C2 and C3 alloys were shown in figure 12. Table 6 was the evaluation for intergranular corrosion grade.

It could be concluded from the diagram that the maximum corrosion depth of C1 and C3 alloys is in the range of 100 ~ 300 μm, the grade is grade 4, the corrosion depth of C2 alloy is less than 100 μm, and the grade is increased to grade 3. The corrosion depth of 2xxx aluminum alloy after Mg, Zn alloying is small, which was due to the fact that Zn element can promote the formation of T phase, the potential difference of T phase is similar to that in matrix Al, and it is not easy to form electrochemical corrosion, which can improve the resistance to intergranular corrosion and stress corrosion. Compared with the intergranular corrosion diagrams of the three alloys, the corrosion morphology of the three alloys was similar, there was no large area of grain corrosion and shedding, and the outer layer of the alloy was smooth, which is a sign of excellent intergranular corrosion resistance. Among them, the corrosion depth of the alloy in figure 12(b) was the smallest 95.19 μm. C3 alloy was the highest, the corrosion resistance has penetrated into the internal grain, and the corrosion resistance is the worst. Only adding a small amount of Ti content can improve the intergranular corrosion properties of the alloy. When the content of Ti is in a certain range, it could promote the corrosion resistance of the alloy. However, with the increase of Ti content, the coarse insoluble ratio of the alloy will increase, and it was easy to form corrosion channels, resulting in a greatly decrease of corrosion resistance in the aluminum alloy.

4. Conclusion

In this article, three new heat-resistant aluminum alloys marked as C1, C2 and C3 were designed. The Ti content gradients of C1, C2 and C3 alloys were 0, 0.47 and 0.89 (wt%), respectively. The sample preparation process was as follows: melting casting, homogenization annealing, cold extrusion, solid solution aging treatment. A series of microstructure analysis and performance tests were adopted. The effect of Ti content on the microstructure and properties of a new type of Al-Cu-Mg-Zn-Zr-Ti cold extruded heat-resistant aluminum alloy was studied. the main conclusions were as follows:

(1) After solid solution treatment, with the increase of Ti content, the content of fine undissolved phases in the alloy increased further.

(2) The three alloys were aged at 120 °C × 12 h, 155 °C × 12 h and 190 °C × 12 h, respectively. After aging at 120 °C × 12 h, the three alloys reached peak aging of 140.75HV, 155.63 HV and 160.80 HV respectively. With the increase of Ti content, the conductivity decreased from 24.05% IACS to 23.56% IACS.

(3) With the increase of Ti content, the strength of room temperature increased and the plastic decreased. The alloy with 0.89% Ti has the highest strength. Compared with the Ti-containing alloy, the tensile strength of room temperature increased by 23.32 MPa, reached 471.55 MPa, and the elongation at break was 16.67%. At the same time, the alloy had good intergranular corrosion resistance and the maximum intercrystalline corrosion depth was 146.31 μm.

(4) With the increase of Ti element, the heat resistance of the alloy is improved. Among them, the heat resistance of the alloy added with 0.89Ti was the best, and the peak value of true stress in hot compression at 250 °C was 36.1% higher than that of the alloy without Ti, reaching 326.66 MPa.
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