

Research Article

Effect of Deformation and Aging on Properties of Al-4.1%Cu-1.4%Mg Aluminum Alloy

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Deformation scheme and preheat treatment of Al-4.1%Cu-1.4%Mg aluminum alloy are chosen, homogenizing annealing at 430°C for 1.5 h, cooling to 250°C in furnace at a cooling rate of less than 30°C/h and then cooling to room temperature to make Al-4.1%Cu-1.4%Mg aluminum alloy annealed fully. Heat treatment tests of Al-4.1%Cu-1.4%Mg aluminum alloy mainly consisting of rolling and aging were conducted, and the optimum peak of aging mechanism is 190°C/12 h. Through comparison of microstructure and mechanical properties with different deformation rates and aging mechanisms, effect of deformation rates and aging mechanism on properties of Al-4.1%Cu-1.4%Mg aluminum alloy was analyzed, and the optimum double peak of aging mechanism is 135°C/7 h + 185°C/14 h. Orthogonal experiments were carried out to analyze mechanical and electrical properties of tested materials before and after deformation, and the effect of aging mechanism on Al-4.1%Cu-1.4%Mg Al alloy was analyzed, and the optimum regression of aging mechanism is 190°C/12 h + 240°C/40 min + 190°C/12 h. Aging scheme is closely related to corrosion resistance of Al-4.1%Cu-1.4%Mg aluminum alloy, and three different aging schemes can improve the corrosion resistance. The exfoliation corrosion evaluation results show that the aging effect on exfoliation corrosion ability order is RRA > two-step aging > peak aging.

1. Introduction

After a combination of solution treatment and aging, Al-Cu-Mg aluminum alloy possesses high strength, which makes it a most widely used aluminum alloy [1]. As a duralumin, Al-Cu-Mg aluminum alloy has a good molding ability and mechanical processing properties. Al-Cu-Mg aluminum alloy products mainly consist of plates, wires, and bars, which are mainly developed for the aircraft skin, bulkheads, wing ribs, engine components, and automobile industries [2, 3].

In order to develop aluminum alloy with high strength, high toughness, and high resistance to stress corrosion cracking, many trials and studies were carried out for decades, and there are mainly two effective methods, thermomechanical treatment (TMT) and retrogression and reaging (RRA) [4, 5]. TMT is a process combining both strain hardening after the plastic deformation and phase transformation strengthening after heat treatment [6]. The basic principle is to

increase the density of defects in the metal deformation and change its distribution; deformation defects generated by phase transition during heat treatment will affect nucleation and distribution of the new phases; at the same time, the formation of new phases will pin or block movement of dislocations defects, making defects stable and microstructure refined improving its strength and toughness [7]. Dislocation caused by deformation is often combined with a dislocation network through slipping or climbing movement to reduce energy; therefore, the microstructure of the treated Al-Cu-Mg aluminum alloy is with high dislocation density and substructures, and thermomechanical treatment is essentially substructure strengthening [8].

In 1974 B.M.Cina first proposed that after artificial aging is suitable for regression treatment, and later repeating the original artificial aging, which would effectively improve mechanical properties and corrosion resistance of aluminum alloy, and the heat treatment process is called

TABLE 1: Chemical composition of Al-4.1%Cu-1.4%Mg aluminum alloy (mass, %).

Elements	Cu	Mg	Mn	Fe	Si	Zn	Ti	Ni	Al
Mass, %	4.1	1.4	0.6	0.5	0.5	0.3	0.15	0.10	Bal

TABLE 2: Selection of levels in stage aging orthogonal design.

Level	First stage aging temperature/°C	First stage aging time/h	Second stage aging temperature/°C	Second stage aging time/h
1	105	4	180	14
2	115	6	190	16
3	125	8	200	18
4	135	10	210	20

RRA treatment [9]. Through peak aging, tensile strength of alloy could achieve ideal value, but the corrosion resistance decreases. In RRA, the temperature and time of second stage aging are critical to the whole aging process. However, to the best of the authors' knowledge, there have been few reports on deformation and aging treatment of Al-4.1%Cu-1.4%Mg aluminum alloy. The purpose of the present study is to investigate the effect of the deformation and different aging treatments on the microstructure and mechanical properties of Al-4.1%Cu-1.4%Mg aluminum alloy, providing reference for its various industrial applications.

2. Experimental Materials and Method

2.1. Experimental Materials. The materials used in this research work were wrought heat treatable Al-4.1%Cu-1.4%Mg aluminum alloy, whose chemical compositions are given in Table 1.

2.2. Thermal Analysis. DSC thermal analysis is conducted in the EPM laboratory of Northeastern University with MDSCQ100 heat analyzer; samples are selected as a bulk sample of 5 mm side length cleaned with alcohol and acetone, respectively, before the test, with pure aluminum as a reference. The heating rate is 10°C/min, and the temperature range is selected between 300°C and 550°C.

2.3. Homogenization Treatment. Annealing temperature was selected as 430°C for 1.5 h, and the plate was cooled to less than 250°C with an air furnace cooling rate of 30°C/h and then air cooled to room temperature.

2.4. Rolling. The slab after homogenization at 450°C is rolled from 30 mm at 470°C to 6.2 mm (total deformation ratio of 79%), in which deformation ratios of 30%, 50%, and 70% were achieved. After solid solution at 490°C for 1 h, the slab was quenched in water immediately, and then it was cold-rolled to 1.4 mm sheet (deformation ratio of 77%) at room temperature for the subsequent experiments.

2.5. Aging. After rolling and solution, the alloy was subjected to aging treatment immediately, and aging treatment is divided into peak aging, stage aging, and RRA in this

experiment. For peak aging, the aging temperature was selected as 180°C, 190°C, and 200°C with aging times of 2 h, 6 h, and 12 h to determine the optimum aging temperature. Then, the optimum temperature aging time was selected from 2 h, 4 h, 6 h, 8 h, 12 h, 16 h, 20 h, and 24 h at the optimum aging temperature. In accordance with the characteristics of the stage aging, the first stage is a low-temperature preaging, generally 80°C lower than the peak aging temperature, and first stage aging time is usually about 6 h to ensure the stability of nucleation. The second stage aging is selected near the peak aging temperature, and the second stage aging time is generally about 16 h to ensure the effect of stabilizing. According to the above characteristics, the aging temperature and time range were selected as 105°C~135°C for 4 h~10 h in the first stage and 180°C~210°C for 14 h~20 h in the second stage, as shown in Table 2.

According to the experimental features of both peak aging parameters and RRA treatment, the first and third aging temperatures and times are the same as the peak aging temperature and time, and the second aging temperature was selected as 240°C and 280°C, and the aging time is 20 min, 40 min, and 60 min, respectively. The specific RRA trial program is shown in Table 3.

2.6. Hardness, Conductivity Test. Hardness experiments were conducted using 430/450SVDTM Vickers hardness measurement instrument with a load of 49 N for a dwell time of 15 s. FD102 digital eddy current conductivity meter with an accuracy of $\pm 0.1\%$ IACS was applied in conductivity measurement. The tensile specimens were got parallel to the rolling direction.

2.7. Corrosion Test. The qualitative analysis of the aluminum alloy was carried out according to ASTM G34-01. Plate sample size of interception is 15 mm \times 20 mm \times 2 mm. According to different surfaces of the sample, the samples are first washed with acetone, ground and polished, and finally washed with distilled water and dried naturally at room temperature. Corrosive medium is EXCO solution (4 mol/L NaCl and 0.5 mol/L KNO₃ + 0.1 mol/L HNO₃), the area ratio of the etched surface to corrosive media is 1500 mL/dm², and water bath temperature is maintained at $25 \pm 1^\circ\text{C}$. The prepared sample was soaked in the etching solution to

TABLE 3: Design of RRA tests.

No.	1st stage aging temperature/°C	1st stage aging time/h	2nd stage aging temperature/°C	2nd stage aging time/min	3rd stage aging temperature/°C	3rd stage aging time/h
1	190	12	240	20	190	12
2	190	12	240	40	190	12
3	190	12	240	60	190	12
4	190	12	280	20	190	12
5	190	12	280	40	190	12
6	190	12	280	60	190	12

observe the surface variation of the specimen after different corrosion times. The specimens for observation are cleaned with 30% HNO₃ solution to remove surface corrosion products, and then etched sample surface was washed away the corrosion products and photographed in comparison with ASTM G34-01 standard, accomplish corrosion sample rating.

Corrosion levels and corresponding descriptions are as follows:

N-no appreciable attack: surface may be discolored or etched, but no evidence of pitting or exfoliation;

P-pitting: discrete pits, sometimes with a tendency for undermining and slight lifting of metal at the pit edges;

EA: (superficial) tiny blisters, thin slivers, flakes or powder, with only slight separation of metal;

EB: (moderate) notable layering and penetration into the metal;

EC: (severe) penetration to a considerable depth into the metal;

ED: (very severe) similar to EC except for much greater penetration and loss of metal.

3. Results and Discussion

3.1. Determination of Solution Temperature. Differential scanning calorimetry curve (DSC curve) is a technique of measurement for the energy changes between the sample and the reference material with variation of temperature and time. The curve is based on the endothermic and exothermic phenomena accompanied by the physical and chemical changes occurring in the heating process. At the program-controlled temperature, the function relationship of heat flow rate variation with temperatures was measured, which is commonly used to quantitatively measure melting point and hot melt. From Figure 1, we could find that the solution temperature is 523°C.

3.2. Effect of Rolling Reduction on Properties of Alloy. To study the deformation ratio on the properties of alloy performance, different reduction ratios were conducted, and both hardness testing and microstructure observation were carried out. As shown in Table 4, after cold rolling, the hardness of alloy increased apparently, especially to initial status.

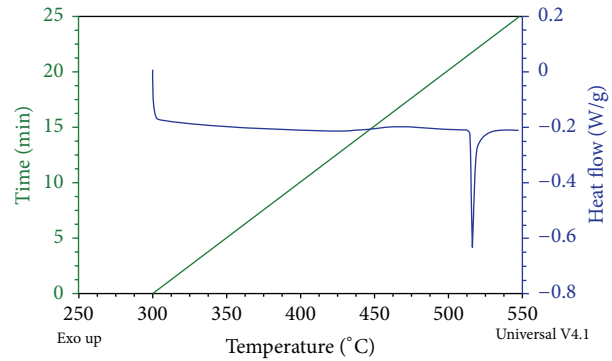


FIGURE 1: DSC curve of Al-4.1%Cu-1.4%Mg.

As shown in Figure 2, microstructure contrast of alloy with different reduction ratios shows that, with the increase of the deformation rate, the grain sizes become smaller, and their distribution is also more uniform.

3.3. Peak Aging. Mechanical properties after peak aging test at 180°C, 190°C, and 200°C for 2 h, 6 h, and 12 h are shown in Table 5. Variations of properties appear at three different aging temperatures. Mechanical properties of alloy aged at 190°C are better than 180°C and 200°C. Therefore, peak aging temperature of alloy is settled as 190°C.

Mechanical properties of alloy with different aging times at 190°C are shown in Table 6. The tensile strength and yield strength fluctuate with the rise of peak aging time and arrive at peak value after aging for 12 h, and there was not much variation in elongation. As discussed above, the optimum peak aging parameters are settled as 190°C for 12 h.

Microstructure of alloy after peak aging was observed, as shown in Figure 3. Initial coarse grains in the alloy are gradually refined and arrive at finest after aging for 12 h. As the aging time increased after peak aging time, the grain sizes grow further, and the grains become coarse again, which also explains why the alloy reached the highest hardness value at the peak aging time of 12 h.

Aging hardening characteristics appear in Al-Cu-Mg aluminum alloy: with the extension of the increase in the aging temperature and aging time, the strength of the alloy increased gradually and declined after the peak aging. Aging intensity variation is caused by the following factors: (1) solid solution strengthening; (2) substrate's recovery and recrystallization; and (3) new phase precipitation [10]. The first

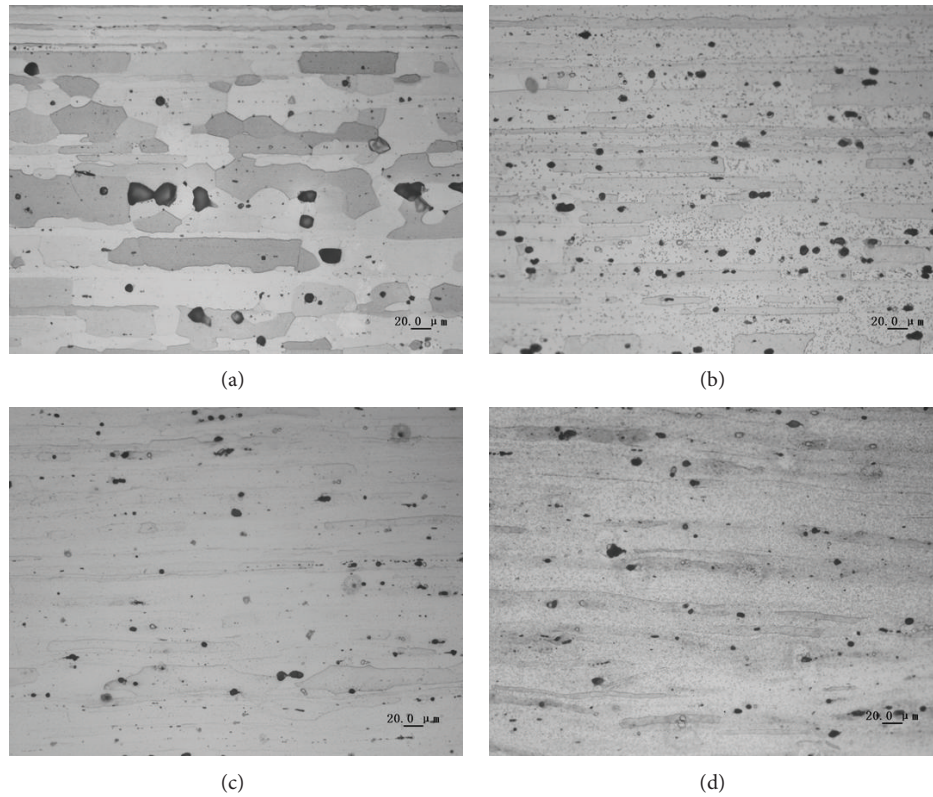


FIGURE 2: Comparison of microstructure of alloys after different deformation ratios: (a) without deformation, (b) 30%, (c) 50%, and (d) 70%.

TABLE 4: Hardness of alloy before and after different deformation ratios.

Treatment	Initial status	After homogenization	30% cold-rolling	50% cold-rolling	70% cold-rolling
Hardness (HV)	90.1	98.6	103.8	108.2	111.7

two factors could make strength decrease with the increasing aging time, while the third factor makes the strength increase, but the strength will fall when the coherent relationship of precipitation phase and the parent phase is destroyed, and the precipitated phase becomes coarse [11]. In the preaging, dispersion precipitates of phase strengthening outweigh softening caused by the other two factors, and therefore the strength is rising and arriving at the peak value. In the later aging, the hardening caused by the precipitation phase is less than the softening caused by the other two factors, therefore, leading to a decreased strength. Some literature also points out that the transition phase S phase (Al_2CuMg) is the main aging precipitation strengthening phase of Al-Cu-Mg alloy [12–14]. The strength variation of the alloy during aging is mainly caused by the interaction between the S phase and matrix dislocations [4].

3.4. Stage Aging Test Results. Stage aged aluminum alloy performance test results with four influencing factors are shown in Table 7. The maximum tensile strength is 483.6 MPa, while the minimum is 325.1 MPa, and the gap between them is 158.5 MPa, which signifies that the effect of different influencing factors on tensile strength is apparent. The yield strength

and elongation also show such apparent influence. Different influencing factors have some effects on both hardness and conductivity. For conductivity, the maximum conductivity is 43.8% IACS, and the minimum is 39.4% IACS with a difference of 4.4% IACS.

In this paper we use the consolidated balance multi-index method and range analysis of each indicator in turn and then find a more satisfactory level combination. The effect of first stage aging temperature on mechanical properties is significant. There is some variation in tensile strength and yield strength, but elongation at 105°C and 135°C is relatively higher, for hardness and conductivity; properties at 135°C are better than those at 105°C. In summary, the optimum first stage aging temperature is 135°C. Six h or 8 h is more appropriate for first stage aging time to ensure better mechanical properties. Through range analysis, secondary aging temperature is extremely significant to mechanical properties among four influencing factors. Indicating that in the stabilization stage, the aging temperature is essential to mechanical properties. At 180°C, strength, elongation, and hardness could be guaranteed, while at 190°C the alloy shows a better conductivity. Therefore, 185°C is chosen as optimum second stage aging temperature. The alloy achieves optimum

TABLE 5: Mechanical properties of alloy at different peak aging temperatures.

Aging temperature/time	Tensile strength/MPa	Yield strength/MPa	Elongation/%
180°C/2 h	422.6	383.6	5.2
180°C/6 h	428.4	387.5	5.1
180°C/12 h	444.1	400.4	5.0
190°C/2 h	435.0	389.9	6.9
190°C/6 h	426.8	373.0	5.9
190°C/12 h	490.4	444.8	6.0
200°C/2 h	430.9	379.4	5.3
200°C/6 h	442.1	382.7	5.1
200°C/12 h	465.8	398.7	5.7

TABLE 6: Mechanical properties of alloy aged at 190°C for different aging times.

Peak aging time/h	Tensile strength/MPa	Yield strength/MPa	Elongation/%	Hardness/HV	Conductivity/%IACS
0	433.2	382.7	5.1	129.7	28.4
2	435.0	389.9	6.9	140.4	38.9
4	457.8	387.7	8.2	141.0	39.6
6	426.8	373.0	5.9	137.4	41.5
8	393.8	319.6	5.1	155.2	43.2
12	490.4	444.8	5.9	147.5	42.5
16	436.4	398.0	5.8	149.7	41.6
20	401.4	355.9	4.7	146.3	41.4
24	443.8	405.7	6.1	160.0	41.2

TABLE 7: Results of stage aging orthogonal tests.

No.	A	B	C	D	Tensile strength/MPa	Yield strength/MPa	Elongation/%	Hardness/HV	Conductivity/%IACS
1	1	1	1	1	462.8	399.0	11.4	149.7	39.4
2	1	2	2	2	438.6	385.3	6.1	146.2	40.7
3	1	3	3	3	398.0	350.1	7.0	132.0	41.7
4	1	4	4	4	329.3	289.9	26.4	104.9	43.8
5	2	1	2	3	429.2	380.1	9.8	141.1	40.9
6	2	2	1	4	450.2	393.9	11.4	152.7	39.7
7	2	3	4	1	356.3	320.4	6.9	120.7	42.6
8	2	4	3	2	383.9	329.8	6.8	128.7	42.0
9	3	1	3	4	389.6	349.7	3.2	126.6	42.2
10	3	2	4	3	364.0	330.2	7.9	116.3	43.0
11	3	3	1	2	466.3	370.0	9.8	142.2	40.9
12	3	4	2	1	430.3	381.9	13.2	140.1	41.5
13	4	1	4	2	325.1	280.0	19.7	107.4	43.6
14	4	2	3	1	402.9	380.1	14.4	134.3	41.5
15	4	3	2	4	397.2	344.6	6.7	141.7	40.7
16	4	4	1	3	483.6	434.8	9.7	150.7	40.0

mechanical properties for second stage aging time of 14 h or 18 h, so 14 h is chosen for second stage aging time. Overall, optimum stage aging process is 135°C/7 h + 185°C/14 h.

Through verification by experiment, after stage aging 135°C/7 h + 185°C/14 h, the alloy's mechanical properties are as follows: tensile strength 520.7 MPa, yield strength 429.6 MPa, elongation 12.5%, the hardness 151.8 HV, and conductivity 41.8% IACS. The test results show that the orthogonal design is reasonable and appropriate.

3.5. RRA Treatment. While the second stage aging temperature is selected at 240°C, tensile strength and yield strength achieved the maximum value. Elongation reaches a good level after aging for 40 min. As shown in Table 8, a combination of comprehensive properties that could be achieved after the second stage of RRA is 240°C/40 min is as follows: tensile strength 501.6 MPa, yield strength 453.7 MPa, elongation 13.1%, hardness 152.3 HV, and conductivity 44.7% IACS.

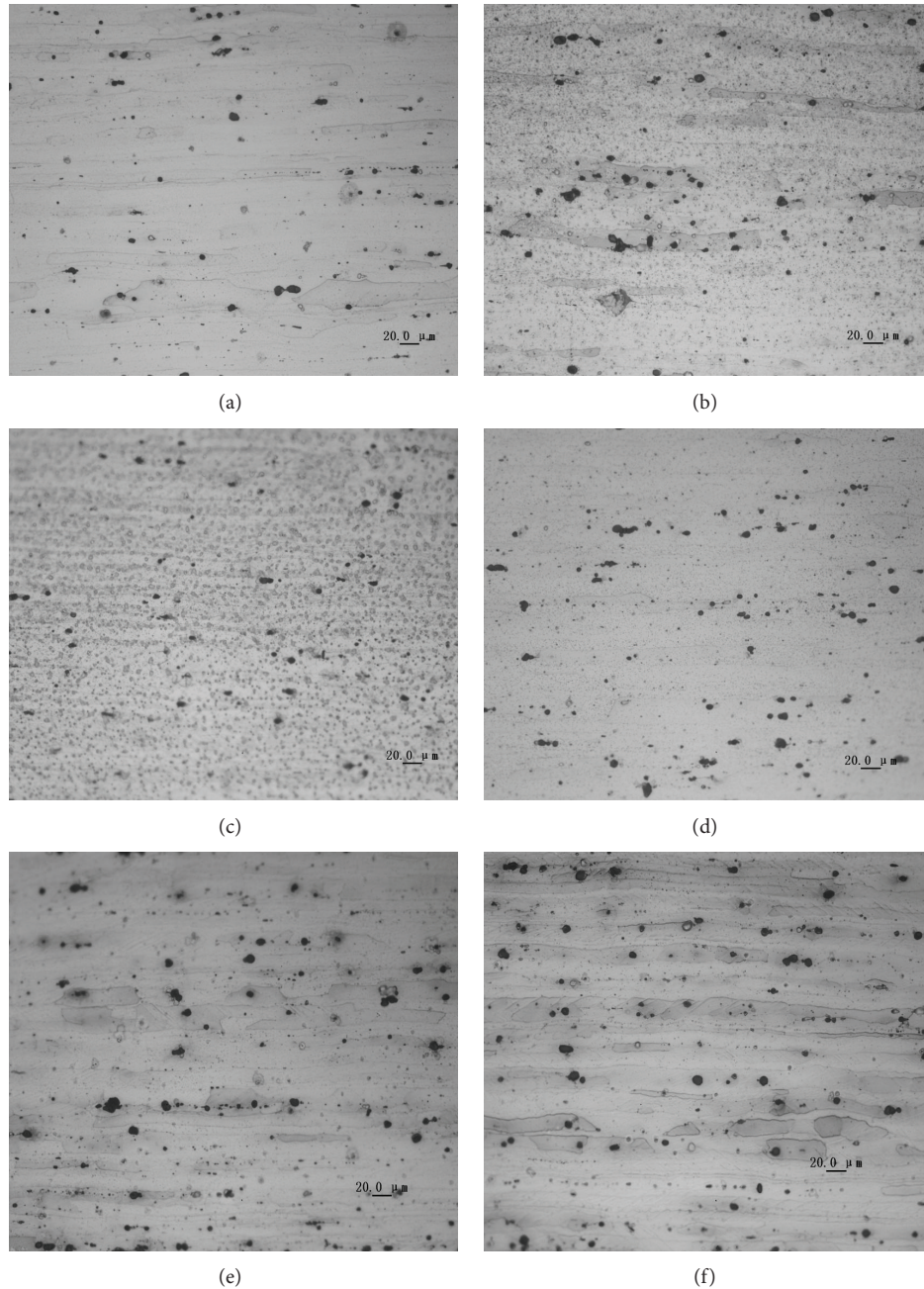


FIGURE 3: Effects of different aging times on alloys' microstructure at 190°C for (a) 4 h, (b) 8 h, (c) 12 h, (d) 16 h, (e) 20 h, and (f) 24 h.

3.6. Corrosion Test Results. While the second stage aging temperature is selected at 240°C, tensile strength and yield strength achieved the maximum value. Elongation reaches a good level after aging for 40 min. A combination of comprehensive properties that could be achieved after the second stage of RRA is 240°C/40 min is as follows: tensile strength 501.6 MPa, yield strength 453.7 MPa, elongation 13.1%, hardness 152.3 HV, and conductivity 44.7% IACS.

The sample soaking time is 96 h. Figure 4 shows corrosion morphology after surface cleaning under different aging conditions. As can be seen from Figure 4, different aging schemes affect alloy's corrosion seriously. As immersion time in the EXCO solution increases, exfoliation corrosion

becomes more severe. There are varying degrees of exfoliation corrosion at 180°C, 190°C, and 200°C peak aging temperatures, with obvious characteristics of exfoliation corrosion. We selected three two-step aged samples and six regression reaged samples for comparison. Six regression reaged samples in the corrosion test do not show obvious corrosion tendency, but only slight pitting. The cleaned samples were assessed in accordance with ASTM G34-01, and the corrosion level assessment results are shown in Table 9. As can be seen from Table 9, aging scheme of Al-4.1%Cu-1.4%Mg aluminum alloy relates closely to. After peak aging treatment, the alloy's corrosion resistance was enhanced, which is still at a low level. Two-step aging improved corrosion resistance at a higher

TABLE 8: Results of RRA tests.

No.	Tensile strength/MPa	Yield strength/MPa	Elongation/%	Hardness/HV	Conductivity/%IACS
1	515.1	472.3	12.1	155.9	42.8
2	506.7	474.6	13.1	152.3	44.7
3	497.7	451.8	13.3	148.2	44.1
4	483.6	445.0	14.2	139.6	44.5
5	463.5	421.4	15.5	134.0	45.6
6	462.2	418.9	14.1	136.4	45.9

TABLE 9: Evaluation of exfoliation corrosion results.

Aging scheme	Level	Aging scheme	Level
180°C/12 h	ED	190°C/12 h + 240°C 20 min + 190°C/12 h	P
190°C/12 h	EC	190°C/12 h + 240°C 20 min + 190°C/12 h	P
200°C/12 h	ED	190°C/12 h + 240°C 20 min + 190°C/12 h	P
105°C/4 h + 180°C/14 h	EB	190°C/12 h + 240°C 20 min + 190°C/12 h	P
115°C/10 h + 200°C/16 h	EA	190°C/12 h + 240°C 20 min + 190°C/12 h	N
135°C/8 h + 190°C/12 h	EA	190°C/12 h + 240°C 20 min + 190°C/12 h	N

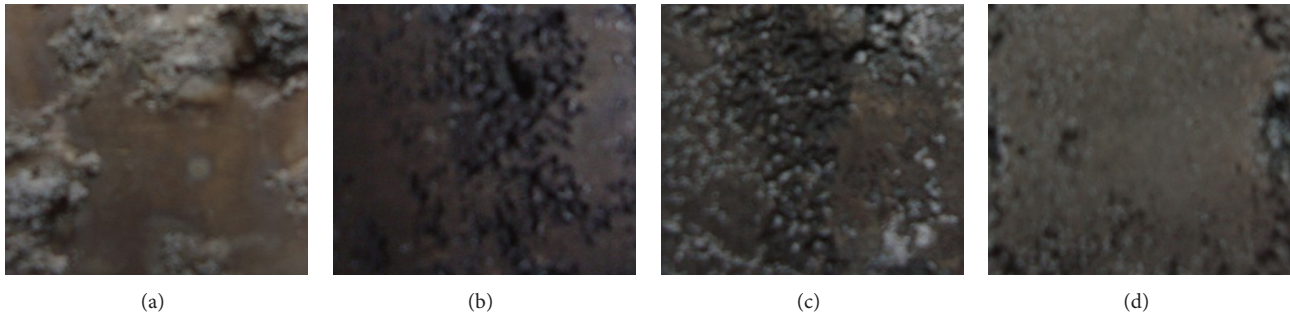


FIGURE 4: Macrophotograph of exfoliation corrosion: (a) without aging, (b) peak aging, (c) two-step aging, and (d) RRA aging.

level, and RRA significantly improves the alloy's corrosion resistance.

4. Conclusions

In this paper, rolling processing and aging heat treatment of Al-4.1%Cu-1.4%Mg aluminum alloy were carried out. Through comparison of microstructures and mechanical properties, effect of rolling reduction ratios and aging process on alloys was analyzed. Orthogonal experiment was applied in analyzing mechanical and electrical properties, and aging process at certain deformation treatment of alloy was settled, which provides reference for various applications of alloy for future industry. The conclusions are as follows.

(1) Deformation scheme and preheat treatment of Al-4.1%Cu-1.4%Mg aluminum alloy are chosen, homogenizing annealing at 430°C for 1.5 h, cooling to 250°C in furnace at a cooling rate of less than 30°C/h and then cooling to room temperature to make alloy fully annealed. Solid solution temperature of Al-4.1%Cu-1.4%Mg aluminum alloy is 495°C. Feasible rolling process is that the alloy is hot-rolled at 470°C from 30 mm to 6.2 mm, and then immediately solid solution at 495°C for 1 h, then cold-rolled to 1.4 mm sheet at room temperature.

(2) After tested deformation, the optimum peak aging process of Al-4.1%Cu-1.4%Mg aluminum alloy is determined: 190°C/12 h. Properties after the above peak aging process are as follows: tensile strength 490.4 MPa, yield strength 444.8 MPa, elongation 5.95%, hardness 147.5 HV, and conductivity 42.5% IACS. Optimum strength with low elongation was obtained after peak aging treatment.

(3) After tested deformation, the optimum stage aging process of Al-4.1%Cu-1.4%Mg aluminum alloy is determined: 135°C/7 h + 185°C/14 h. Properties after the above stage aging process are as follows: tensile strength 520.7 MPa, yield strength 429.6 MPa, elongation 12.5%, hardness 151.8 HV, and conductivity 41.8% IACS. Both strength and elongation are improved after stage aging.

(4) After tested deformation, the optimum RRA process of Al-4.1%Cu-1.4%Mg aluminum alloy is determined: 190°C/12 h + 240°C/40 min + 190°C/12 h. Properties after the above stage aging process are as follows: tensile strength 501.6 MPa, yield strength 453.7 MPa, elongation 13.1%, hardness 152.3 HV, and conductivity 44.7% IACS. Both mechanical properties and conductivity were enhanced after RRA treatment.

(5) Aging scheme is closely related to corrosion resistance of Al-4.1%Cu-1.4%Mg aluminum alloy, and three different

aging schemes can improve the corrosion resistance. The exfoliation corrosion evaluation results show that the aging effect on exfoliation corrosion ability order is RRA > two-step aging > peak aging.

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