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ARTICLE

Effects of Applying Direct Current on Microstructures and Properties of 7B04 Aluminum Alloy During Solid Solution and Artificial Ageing

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Abstract: The microstructures and mechanical properties of 7B04 aluminum alloy solid solutioned at 470 °C and aged at 100~140 °C without and with electric current were investigated by tensile test, scanning electron microscope (SEM) and transmission electron microscope (TEM). Compared with the 7B04 alloy treated by solid solution at 470 °C/30 min and ageing at 120 °C/24 h without electric current, the alloy with applied electric current of 500 A during solid solution and/or ageing treatment has the higher tensile strengths and improved elongation at the same ageing time. Applying electric current during solid solution can significantly shorten the time to the peak strength by 12 h in the subsequent ageing alloy. The alloy heat-treated at 470 °C/500 A+120 °C/500 A has the highest peak ultimate and yield strengths of 735 and 675 MPa, respectively, and the improved ductility of 10.9%, among all ageing conditions. The electric current can significantly increase the precipitation density of Guinier-Preston II (GPII) zones.

Key words: 7B04 alloy; artificial ageing; direct current; microstructures; properties

As an effective external field, the electromagnetic field, static magnetic field, current field, pulse current, electrostatic field, etc, were used for material preparations and property controls of ferromagnetic and non-ferromagnetic materials. Effects of electric and magnetic fields on phase transformations in metals and alloys were focused on solidification process[1], superplasticity[2], interfacial reactions in diffusion couples[3], annealing behavior and texture evolution[4-8], the dissolution and transformation of constituent phase during homogenization[9-11], precipitation process during ageing[12,13]. A high magnetic field of 12 T could promote the dissolutions of AlZnMgCu (T) and $CuMgAl_2$ (S) in Al-Zn-Mg-Cu alloy during homogenization[9,10]. Liu and his coworkers found that the applied electric field in Al-Li alloy could accelerate the dissolution of the non-equilibrium phases at grain boundaries as well as the removal of the interdendritic segregations during homogenization[11], while, it impeded the nucleation of δ' phase during artificial ageing[12]. They thought that a charged surface layer in materials created by the applied electric field interacted with the charged defects which approached to the surface, particularly vacancies, and thus the resulting additional defect flux in turn influenced the dislocation movement during heat treatment. Jung and Conrad thought[13] that the electron wind force, the chemical potential gradient and the internal stress produced together played the important roles in precipitation rate during the ageing process.

Zhang et al[14] revealed that the low density of electric pulse current significantly accelerated the ageing process and increased the peak hardness of 2219 aluminum alloy. Wang et al [15] pointed out that DC increased the electric conductivity and hardness of aged Cu-Cr-Zr alloy by promoting the diffusion of solute atom and the movement of vacancy. Applying electric field cannot generate current in sample. The effective detection distance is less than or equal to 50 μm. The effect of electric field is not caused by the flow of particles on macroscale. It can affect the chemical potential of alloy surface, and the resulting directional movement of the charge will influence the movement of particles. So, it can be

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deduced that the effect of electric current on the particle motion is greater than that of the electric field due to the direct Joule heating when a metal or alloy is directly connected to the electric current. Thus, due to a large Joule heating, and a discontinuous short pulse current will be used to prevent the damage of specimen, so the electric current is not suitable for the low temperature heat treatment of materials, such as the ageing of Al or Mg alloys. In the present work, DC was directly introduced into 7B04 aluminum alloy samples during solid solution and artificial ageing using our patent electric current heat treatment device[16], which can avoid the obvious Joule heating effect. 7B04 aluminum alloy has high strength and fracture toughness, low density and good corrosion resistance, and is widely used in aerospace industries. The evolutions of tensile properties and microstructures of 7B04 aluminum alloy with ageing time applying without or with electric current of 500 A were studied by tensile test, SEM and TEM.

1 Experiment

The chemical composition of 7B04 aluminum alloy used in present work is (in wt%): Al-6.4Zn-2.22Mg-2.24Cu-0.11Zr-0.04Si-0.06Fe-0.10Mn-0.04Cr-0.05Ni-0.06Ti, produced by semi-continuous cast. The ingots with size of 400 mm×1400 mm were homogenized at 470 °C for 24 h, followed by air cooling. The homogenized billets were preheated at 500 °C for 3 h and then extruded into aluminum sheet with size of 130 mm×10 mm×1200 mm at a speed of 4 mm/s and a ratio of 10, followed by air cooling. Then the extruded samples were treated by solid solution and artificial ageing with the application of a DC of 500 A. The schematic diagram of heat treatment device used in this work during solid solution and artificial ageing is shown in Fig.1. The heating rate was 5 °C/min. A wind bellowing device was used to maintain the temperature variation of sample within ±3 °C. The sample with size of 580 mm×20 mm×10 mm for electric current heat treatment was cut from the extruded sheet along the extruding direction. In order to investigate the effect of electric current on solid solution treatment, the extruded samples were treated at 410, 430, 450 and 470 °C for 30 min without or with current. In order to study the effect of electric current on artificial ageing, the extruded samples were solid solution treated at 470 °C for 30 min without or with current and then artificial aged at 100, 120 and 140 °C for different time without or with current. Four types of artificial ageing conditions performed in the present work are listed in Table 1: (1) solid solution and artificial ageing without electric current (470 °C/0 A+100~140 °C/0 A), (2) solid solution with electric current and artificial ageing without electric current (470 °C/500 A+100~140 °C/0 A), (3) solid solution without electric current and artificial ageing with electric current (470 °C/0 A+100~140 °C/500 A), (4) solid solution and artificial ageing with electric current (470 °C/500 A+100~140 °C/500 A).

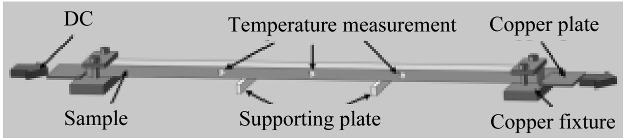

Fig.1 Schematic diagram of electric current heat treatment used in present work

Table 1 Artificial ageing treatments used in present work

Solid solution treatment	Ageing treatment
470 °C/0 A/30 min	100 °C/0 A/1 h, 2 h, 6 h, 12 h, 24 h, 48 h 120 °C/0 A/2 h, 6 h, 12 h, 24 h, 48 h 140 °C/0 A/2 h, 4 h, 8 h, 12 h, 24 h, 48 h
470 °C/500 A/30 min	100 °C/0 A/1 h, 2 h, 6 h, 12 h, 24 h, 48 h 120 °C/0 A/2 h, 6 h, 12 h, 24 h, 48 h 140 °C/0 A/2 h, 4 h, 8 h, 12 h, 24 h, 48 h
470 °C/0 A/30 min	100 °C/500 A/1 h, 2 h, 6 h, 12 h, 24 h, 48 h 120 °C/500 A/2 h, 6 h, 12 h, 24 h, 48 h 140 °C/500 A/2 h, 4 h, 8 h, 12 h, 24 h, 48 h
470 °C/500 A/30 min	100 °C/500 A/1 h, 2 h, 6 h, 12 h, 24 h, 48 h 120 °C/500 A/2 h, 6 h, 12 h, 24 h, 48 h 140 °C/500 A/2 h, 4 h, 8 h, 12 h, 24 h, 48 h

Tensile specimens with a gauge diameter of 8 mm and a length of 30 mm were machined from the artificial aged samples, and then tensile tested at room temperature using an Instron 1185 machine operating at a constant crosshead speed of 1.0 mm/min. The tensile properties under each condition were the average value of three specimens.

The tensile fracture surfaces were observed on a SHIMADZU SSX-550 type scanning electronic microscope (SEM). Samples for characterizing the morphologies of the precipitates under different heat treatment conditions were prepared by twin-jet thinning electrolytically in a solution of 30 vol% nitric acid and 70 vol% methanol at –25 °C, and then observed on a JEM200CX type transmission electron microscopy (TEM).

2 Results and Discussion

2.1 Effects of electric current on solid solution

The relationship between the tensile properties and solid solution temperature of 7B04 alloy without or with electric current is demonstrated in Fig.2. With increasing temperature from 410 °C to 470 °C, the ultimate tensile strength and yield strength of alloy increase gradually, but elongation decreases slightly, when the alloy is solid solutioned without current. The strengths of alloy solid solutioned with current are higher than those of alloy solid solutioned without current at the same temperature. It should be noted that the ultimate strength of alloy solid solutioned at 450 °C with current (605 MPa) is equal to that of alloy solid solution at 470 °C without current (603 MPa).

2.2 Effects of electric current on artificial ageing

It can be seen that the curves of the mechanical properties

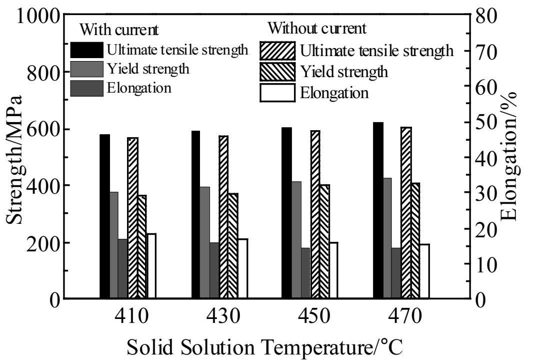

Fig.2 Relationship between the tensile properties and solution temperature of 7B04 alloy

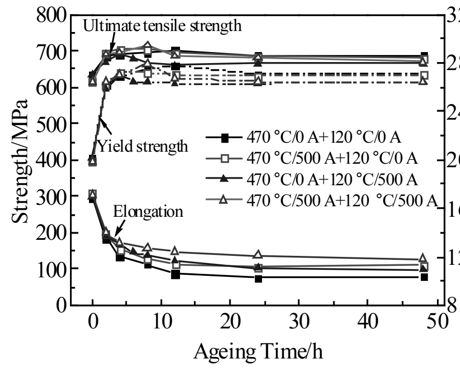

Fig.4 Curves of mechanical properties vs ageing time of 7B04 aluminum alloy aged at 120 °C

vs ageing time of 7B04 alloy aged at 100 °C without and with current have a similar changing tendency, as shown in Fig.3. The ultimate tensile/yield strengths of alloys heat-treated at 470 °C/0 A+100 °C/0 A, 470 °C/500 A+100 °C/0 A, 470 °C/0 A+100 °C/500 A and 470 °C/500 A+100 °C/500 A increase progressively with prolonging the aging time, and no peak values is obtained until 48 h, which are 675/605 MPa, 688/642 MPa, 694/649 MPa and 706/656 MPa, respectively. While, the elongation of alloy decreases with extending ageing time, and reaches the saturated value at 12~24 h in alloy heat-treated without or with current. It should be noted that the alloy heat-treated during solid solution or/and artificial ageing with current has higher strengths and elongation than the one heat-treated without current at the same ageing time.

Fig.4 displays the curves of the ultimate tensile strength, yield strength and elongation changing with ageing time of 7B04 alloy solid solutioned at 470 °C and aged at 120 °C without and with current. It can be seen that the age hardening curve of alloy aged at 120 °C has the similar shape to the alloy aged at 100 °C. The ultimate tensile strength, yield strength of alloy solid solutioned at 470 °C without current are 635 and 405 MPa, respectively. The ultimate tensile strength and yield

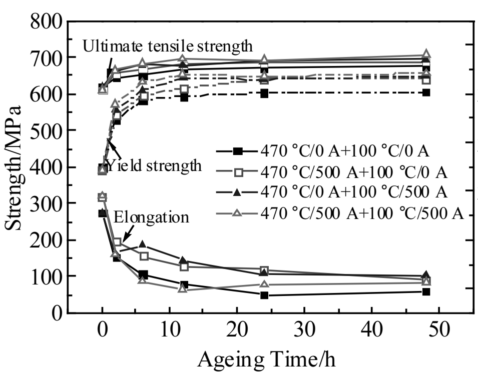

Fig.3 Curves of mechanical properties vs ageing time of 7B04 aluminum alloy aged at 100 °C

strength reach the peak values of 715 and 632 MPa, respectively, but elongation decreases to 9.5%, when the alloy treated at 470 °C/0 A+120 °C/0 A for 24 h.

The peaks of the ultimate tensile strength and yield strength of 7B04 alloy treated at 470 °C/500 A+120 °C/0 A appear at 12 h, which are 725 and 655 MPa, respectively, higher than those of alloy treated at 470 °C/0 A+120 °C/500 A for 24 h. It can be seen that only applying electric current in solid solution can significantly shorten the time to the age peak by 12 h, while both of strength and elongation at the age peak state increase.

The age peak state of 7B04 alloy treated at 470 °C/0 A+120 °C/500 A occurs at 24 h, the ultimate tensile strength, yield strength and elongation are 733 MPa, 673 MPa, 10.7%, respectively. Compared with the alloy treated at 470 °C/0 A+120 °C/0 A for 24 h, the increments of the ultimate tensile strength, yield strength and elongation are 18 MPa, 41 MPa and 1.2%, respectively. It should be noted that only applying electric current in artificial ageing cannot change the time to the age peak, but the strengths and elongation at the age peak state increase.

Applying electric current both in solid solution and ageing (470 °C/500 A+120 °C/500 A) can enhance the age hardening ability, while the ultimate tensile strength and yield strength increase to 680 and 570 MPa, respectively, in alloy aged for 2 h. The time to the age peak is shortened to 12 h, and the ultimate tensile strength, yield strength and elongation increase 20 MPa, 20 MPa and 1.4%, respectively.

Fig.5 exhibits the curves of the mechanical properties vs ageing time of 7B04 alloy aged at 140 °C without and with current. The time to the age peak of alloys heat-treated at 470 °C/0 A+140 °C/0 A, 470 °C/500 A+140 °C/0 A, 470 °C/0 A+140 °C/500 A and 470 °C/500 A+140 °C/500 A are 12, 4, 4 and 8 h, respectively, and the peak ultimate tensile strengths are 700, 705, 690 and 715 MPa, respectively.

It should be noted that the peak strengths of alloy aged at 140 °C are lower than those of alloy aged at 120 °C, but higher than those of alloy aged at 100 °C. The strengths of alloys

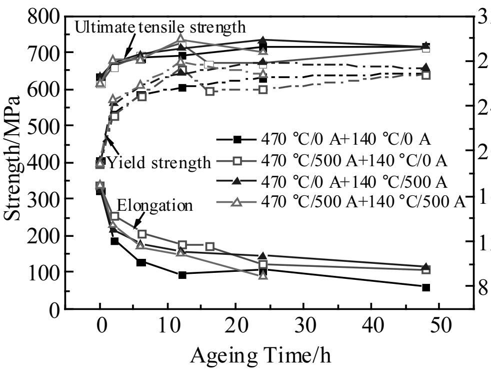

Fig.5 Curves of mechanical properties vs ageing time of 7B04 aluminum alloy aged at 140 °C

aged with current at 120 and 140 °C drop quickly after reaching the peak value, as seen in Fig.4 and Fig.5. Applying electric current can improve the ductility of 7B04 alloy. The time to the peak strength is shortened significantly when applying electric current during solid solution. While, the alloy heat-treated at 470 °C/500 A+120 °C/500 A has the highest peak strengths and good ductility.

The typical tensile fracture surfaces of samples at the age peak state without and with current are illustrated in Fig.6. In general, the fracture surfaces display a wide range of dimples and cleavage feature as well as fracture modes in all ageing conditions. Compared with sample aged without electric current (Fig.6a), the samples aged with current exhibit more fine and deep dimples and less cleavage feature associated with fracture (Fig.6b, 6c and 6d).

Fig.7 reveals the TEM bright field images of samples at the age peak without and with current. Plenty of extremely fine dots with black contrast homogeneously distribute in the matrix of alloy heat-treated at 470 °C/0 A+120 °C/0 A/24 h (Fig.7a). The shape of these fine precipitates is not well defined since they are extremely fine, and the selected area electron diffraction (SAED) pattern only shows faint streaks. With the application of electric current (Fig.7b, 7c and 7d), the size of the precipitates increases slightly, while the density of them increases. It can be seen that the alloy heat-treated at 470 °C/500 A+120 °C/500 A/12 h has the highest density of precipitates among four ageing conditions.

From high resolution TEM (HRTEM) images of alloy heat-treated at 470 °C/0 A+120 °C/0 A/24 h (Fig.8a), in the $[110]_{Al}$ zone axis, most of dots seen in the low magnification image (Fig.7a) exhibit the plate-like morphology (labeled as arrows) tilted 26.5° to the electron beam direction; they are GP(II) zones[17-19], which precipitate as one or a few Zn-rich atomic layers on $\{111\}_{Al}$ over a wide temperature range from room temperature to about 140 °C[17]. These precipitates are up to about 2 nm thick and up to about 5 nm long. From present observations, the strengthening effect at the age-peak of the 7B04 alloy is produced predominantly by the GPII zones. The typical HRTEM image of alloy heat-treated at 470 °C/500 A+120 °C/500 A/12 h is shown in Fig.8b. It can be seen that the amount of GPII platelets increases, and they are up to 5 nm in thickness and up to 8 nm in length.

The effect of electric current on precipitation in alloy depends on: (a) the alloy composition, (b) the solid solution temperature and time, (c) the aging temperature and time and (d) the magnitude of the current density and its frequency, involving current effects of: (a) polarization, (b) electric

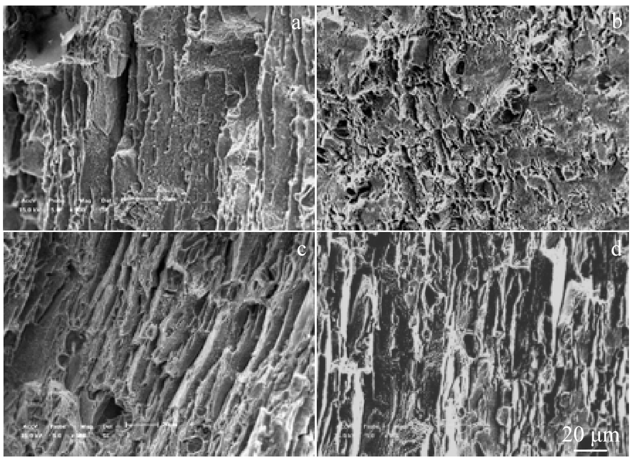

Fig.6 Typical tensile fracture surfaces of samples at the ageing peak without and with current: (a) 470 °C/0 A+120 °C/0 A/24 h; (b) 470 °C/500 A+120 °C/0 A/12 h; (c) 470 °C/0 A+120 °C/500 A/24 h; (d) 470 °C/500 A+120 °C /500 A/12 h

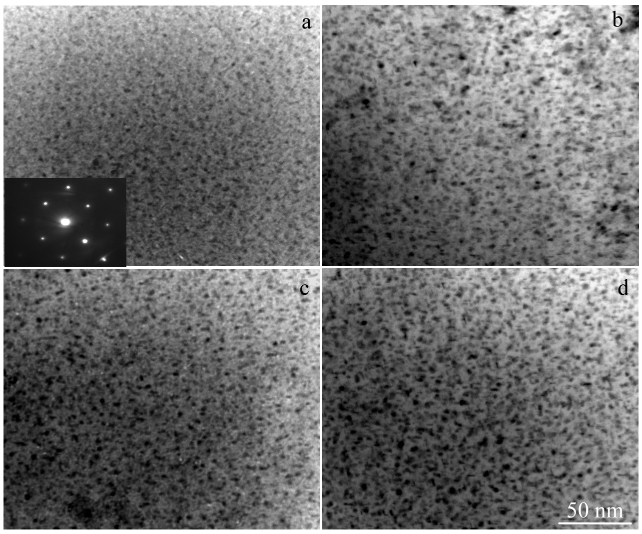

Fig.7 TEM bright field images of samples at ageing peak without and with current: (a) 470 °C/0 A+120 °C/0 A/24 h; (b) 470 °C/500 A+120 °C/0 A/12 h; (c) 470 °C/0 A+120 °C/500 A/24 h; (d) 470 °C/500 A+120 °C/500 A/12 h

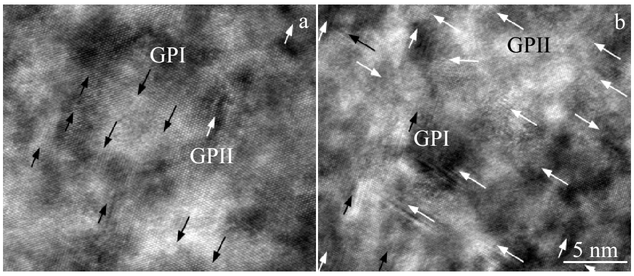

Fig.8 HRTEM images of $[110]_{Al}$ of alloy heat treated at 470 °C/0 A +120 °C /0 A/24 h (a) and 470 °C/500 A+120 °C/500 A/12 h (b)

charge force, (c) electron wind force, (d) electrochemical potential, (e) electrostatic force and current pinch force, and (f) Joule heating. Thereby, an electric field or current can alter the equilibrium state or the kinetics of phase transformations.

The phase transformations here are all considered to be diffusion-controlled, and so the atomic drift flux which occurs with application of an electric potential to a metal is given by[4]:

$$\varphi_i = \frac{N_i D_i}{kT}\left(kT\frac{\partial \ln N_i}{2x} - \Omega\frac{\partial \sigma}{\partial x} + Z^* e\rho j\right) \tag{1}$$

where N is the atomic density, D is the pertinent diffusion coefficient, k is the Boltzmann constant, T is the absolute

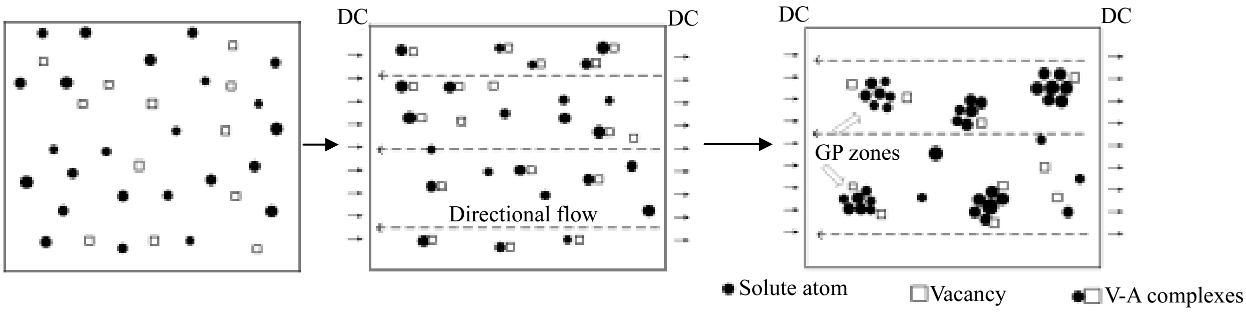

Fig.9 Mechanism of the electrical current influencing the precipitation

temperature, Ω is the atomic volume, Z^* is an effective valence, which for many metals is of the order of 10, e is the charge on an electron, ρ is the resistivity and j is the current density. It can be seen that the electron wind force $Z^*e\rho j$ plays an important role on precipitation. Yang et al[8] found that a pulsed electric current could innovatively be used to tailor grain boundary and improve the plasticity of pure Ti through an enhancement in long-range atomic diffusion. In the present work, we also discover that the electric current improves the ductility of alloy at the age-peak state. Liu et al[11] reported that applying an electric field during solid solution treatment of Al-Li alloy influenced the size and distribution of the subsequent ageing precipitates, which is in agreement with the results of the present work. They discovered that an electric field of E=2 kV/cm could reduce the activation energy from 0.71 eV to 0.53 eV. This indicated that an electric field changed the solute diffusion mechanism to the precipitation of the solute- vacancy complexes. Esmaeili et al[20] pointed out that the contribution of the yield stress in the age-peak of aluminum alloy was proportional to the volume fraction of precipitates. Our previous works[9] proved that the electric current obviously accelerated the diffusion of Zn and Cu in 7B04 alloy during homogenization. In the present work, it can be seen that only applying electric current alternatively during solid solution can significantly shorten the time to the age-peak state, besides enhancing strength. Thus, it can be deduced that the electric current promotes the dissolution of residual phases, such as T and S, during solid solution, and so there are more Zn, Mg and Cu entering Al matrix than those of alloy solid-solution without electric current. So, the ultimate strength of alloy solid solution at 450 °C with current is equal to that of alloy solid solutioned at 470 °C without current. Jung and Conrad[13] also found that the electric field during solid solution increased the yield and tensile strengths of the subsequent natural ageing alloy, and this effect was equivalent to a reduction of 10~25 °C in the solid solution temperature. From TEM observations (Fig.7 and Fig.8), it can be found that the size of precipitates is larger slightly in alloys heat-treated with electric current during solid solution and/or artificial ageing than that in alloy at traditional T6 condition. The mechanism of the electric current influencing the precipitation is shown in Fig.9. The electric current promotes the directional movements of vacancies and solute atom, and accelerates the formation of vacancy-solute atom complexes, so increases the precipitation density of strengthening phase-GP zones in the alloys heat-treated with electric current.

3 Conclusions

1) With increasing solution temperature from 410 °C to 470 °C, the ultimate tensile strength and yield strength of 7B04 alloy increase gradually, but elongation decreases slightly. The strengths of alloy solid solutioned with current are higher than

that of the alloy solid solution treated without current at the same temperature.

2) The ultimate strength of alloy solid solutioned at 450 °C with current is equal to that alloy solid solutioned at 470 °C without current.

3) Applying electric current alternatively during solid solution or artificial ageing increases both strength and elongation, due to the high precipitation density of GPII zones. The alloy heat-treated at 470 °C/500 A+120 °C/500 A/12 h has the highest peak strength.

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施加直流电对 7B04 铝合金固溶与时效组织和性能的影响

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摘　要：采用拉伸试验、扫描电子显微镜（SEM）和透射电子显微镜（TEM）等手段，研究了 7B04 铝合金在施加和不施加直流电条件下，经 470 ℃固溶和 100~140 ℃时效处理后的组织和力学性能。与传统的 T6 处理（470 ℃/30 min+120 ℃/24 h）相比，固溶和/或时效处理时施加 500 A 的电流，可显著提高 GPII 区的析出密度，进而提高峰时效状态下的抗拉强度和延伸率。固溶过程中施加直流电可将随后时效合金达到峰值强度的时间明显缩短 12 h。

关键词：7B04 合金；人工时效；直流电；组织；性能

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