

Article

Effect of Electric Pulse Current Rapid Aging Treatment on Microstructure and Properties of Al-7Si-0.55Mg Alloy

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Received: 25 September 2019; Accepted: 11 October 2019; Published: 14 October 2019



Abstract: A rapid aging treatment method of Al-7Si-0.55Mg alloy using electric pulse, namely electric pulse aging treatment, is explored in this study. The effect of electric pulse assisted aging on microstructure and properties of Al-7Si-0.55Mg alloy are investigated by means of optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), tensile tests and hardness tests. The results show that the microstructure of the Al-7Si-0.55Mg alloy is effectively refined, and the morphology of eutectic Si changes from long and thin strip to a spherical shape or short rod. The elongation of the Al-7Si-0.55Mg alloy is significantly improved after electric pulse assisted aging, albeit it did slightly compromise the tensile strength. It is important to reduce the aging time by 3 h, saving energy. According to classical nucleation theory, the formula of the phase nucleation rate promoted by an electric pulse was determined. The application of an electric pulse can accelerate the nucleation of phase transformation by decreasing the thermodynamic energy barrier, which increases the nucleation rate and significantly improves the properties of the alloy. It provides an experimental and theoretical basis for the Al-7Si-0.55Mg alloy to obtain good mechanical properties and industrial applications.

Keywords: electric pulse; pulse current; pulse frequency; aging treatment; Al-7Si-0.55Mg alloy

1. Introduction

The Al-7Si-0.55Mg alloy has low density, high specific strength, good corrosion resistance and casting properties. It is widely used in aerospace, machinery manufacturing, transportation and other fields. With the development of modern industry, the demand for an as-cast aluminum alloy is increasing. Therefore, the requirements for various performance indices of as-cast aluminum alloys are also increasing [1–4]. The as-cast aluminum alloy needs heat treatment to improve its properties. However, traditional heat treatment has many problems such as low efficiency, large energy consumption, environmental pollution and serious surface oxidation of samples. Therefore, it is necessary to explore a new green heat treatment method to improve the microstructure and properties of the alloy and save energy.

Electric pulse processing technology has the advantages of no pollution, high energy utilization, flexible methods, convenient operation and remarkable effects. At present, as a new method to improve the microstructure and properties of materials, electric pulse processing technology has been widely studied [5]. Using the appropriate treatment process and applying electric pulses in a small area or between different phases, the technology can quickly prepare materials with excellent microstructure and properties, due to the characteristics of instantaneous heating and non-equilibrium treatment.

This is a new processing technology that is green, efficient, energy-saving and environment-friendly, effectively reducing the processing cost [6]. Troitskii [7] first proposed the electro-plastic effect of metals in 1963. In 1975, Golovin et al. [8] applied electric pulses to conductive thin plates, resulting in thermal concentration at the crack tip, and first proposed the possibility that crack propagation can be suppressed due to the thermal effect of pulse current. Misra [9,10] first used the current treatment technology in the solidification process of the hypereutectic alloy, Pb-15%Sb-7%Sn, and the hypoeutectic, Pb-10%Sb-3%Sn alloy. Pulsed current treatment could suppress the precipitation of the primary phase in the hypereutectic alloy, which obtains a uniform and fine solidified structure. Since then, a new technology for pulsed current refinement solidification has been developed. Conrad [11] suggested in his paper that when an electropulse is applied to metals, it may accelerate the atomic diffusion and dislocation movement, and lower the energy barrier of phase transformation. Zheng et al. [12] studied the electropulse-assisted solution treatment of the 6061 aluminum alloy. Compared with the samples treated with conventional heat treatment, the grain structure of the alloy was more uniform and fine, and the tensile strength and hardness were significantly improved. Therefore, the application of electric pulses to industrial production has broad prospects.

In this paper, the aging treatment of Al-7Si-0.55Mg alloy is studied. Further, a new green facile aging treatment method called electric pulse aging treatment is introduced. The Al-7Si-0.55Mg alloy was treated by solid solution at a constant temperature for a certain time. Then an electric pulse was applied during aging treatment that changed the pulse current with a constant pulse frequency and conduction time. The optimum pulse current was selected by changes of tensile properties and hardness of the alloy before and after aging and organization analysis of the Al-7Si-0.55Mg alloy, which determined that the aging treatment time of the Al-7Si-0.55Mg alloy was significantly reduced by using an electric pulse. It provides an experimental basis and theoretical basis for the Al-7Si-0.55Mg alloy to obtain good mechanical properties and industrial application.

2. Experiments

The experimental materials were metal mold casting Al-7Si-0.55Mg alloy plates (6.95 wt.% Si, 0.55 wt.% Mg, 0.12 wt.% Ti, 0.06 wt.% Be and balance Al) with a size of 50 mm × 10 mm × 2 mm. Al-7Si-0.55Mg alloy plates were treated by solution treatment in a box-type resistance furnace (SX-12-10, Harbin, China) preservation 10 h at 540 °C (according to GB/T 25745-2010 of China). Then the alloy plates were removed rapidly from the furnace and carried out by quenching in water at 60 °C. One group of three Al-7Si-0.55Mg alloy plates were treated by the conventional aging treatment in a box-type resistance furnace at 160 °C for 8 h. Then the basic principles of electric pulse aging treatment were applied using the SOYI-241000ML pulse power (SOYIPOWER, Shanghai, China) supply as shown in Figure 1. The two ends of copper wire were connected with the Al-7Si-0.55Mg alloy plate and the output end of the pulse power supply respectively. When the box type resistance furnace temperature was 160 °C, the alloy plates were put into the furnace for aging treatment. The pulse power supply was turned on and the corresponding parameters were set up. The time of electrical pulse assisted aging treatment was 5 h, in which the time of applying electric pulse was 30 s (pulse width of 100 µs). The Al-7Si-0.55Mg alloy plates held for the corresponding time and were removed rapidly from furnace for air cooling.

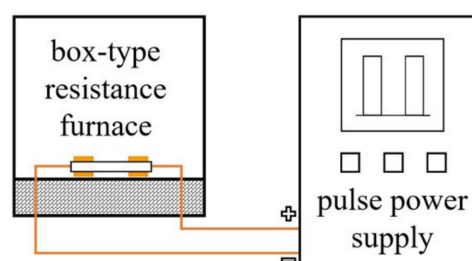


Figure 1. Schematic diagram of electric pulse assisted aging process.

The following specific experimental parameters were determined: Pulse frequencies of 10 Hz, 40 Hz and pulse currents of 300 A, 600 A, 900 A, 1200 A, respectively. The conventional heat treatment (CHT) was used as the control group. From the variation of hardness and tensile properties of the alloy before and after aging, the optimal parameters of the electric pulse aging treatment were selected. The tensile mechanical properties were tested by the MTS-E44304 electronic universal testing machine (MTS SYSTEMS, Shanghai, China) and standard tensile test plates were prepared according to the GB/T228-2010 of China, as shown in Figure 2. The microstructures were observed by an optical microscope (GX71, OLYMPUS, Tokyo, Japan). The transmission electron microscopy (JEM-2100, JEOL, Tokyo, Japan) was used to analyze the microstructure of the Al-7Si-0.55Mg alloy with conventional heat treatment (CHT) and the electric pulse aging after solid solution alloy.

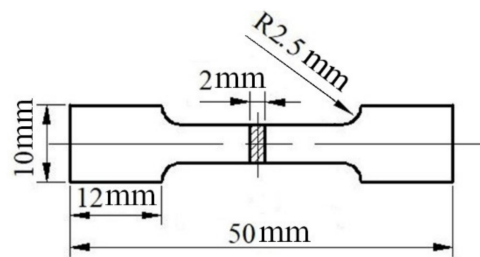


Figure 2. Standard tensile test plate of the Al-7Si-0.55Mg alloy.

3. Results

3.1. Microstructure

The microstructure of as-cast and conventional heat treatment Al-7Si-0.55Mg alloy are shown in Figure 3. As is well-known, the main microstructure of the as-cast Al-7Si-0.55Mg alloy is white, and the α -Al matrix and dark eutectics (α -Al + Si) combination is shown in Figure 3a. Due to segregation, the eutectics produced rich areas during solidification, which were unevenly distributed in the α -Al matrix. The morphology of eutectics in the alloy was mainly a long and thin strip. Tearing effects played a negative role in the matrix because of its sharp end, which limited the properties of as-cast alloys [13,14]. The microstructure of conventional heat treatment Al-7Si-0.55Mg alloy is shown in Figure 3b. The morphology of eutectic Si distributed along grain boundaries was spherical or a short rod. Compared with as-cast Al-7Si-0.55Mg alloy, the size of eutectic Si decreased and the distribution was more uniform. During the solid solution treatment, Si, Mg and other elements distributed along grain boundaries were re-dissolved into the α -Al matrix under the high temperature. During quenching, a large number of Si and Mg elements had no time to precipitate out due to the high cooling rate, which remained in the α -Al matrix with the supersaturated solid solution. Si and Mg elements diffused from the inside of the α -Al matrix to form grain boundaries, precipitating a large number of fine and dispersed Mg_2Si strengthening phase and a small Si phase, which improved the comprehensive mechanical properties of the alloy.

The fracture morphology of the as-cast and conventional heat treatment Al-7Si-0.55Mg alloys are shown in Figure 4. There were many tearing edges, dimples and bright white inclusions or second phase particles in the fracture morphology of the as-cast Al-7Si-0.55Mg alloy. Due to the large amount of dendritic segregation eutectic Si in the as-cast microstructure, the Si phase was prone to microcracks and gradually expanded outward when the alloy was subjected to external stress, which easily caused the alloy to fracture. After conventional heat treatment, the number of dimples in the fracture morphology of the alloy increased. There were smaller precipitated phases and some inclusions in the dimples, which reduced the stress concentration between the precipitated phases and the matrix at the grain boundary, and this prevented the generation and expansion of microcracks. It significantly improved the morphology of the eutectic Si phase and the comprehensive properties of the alloy.

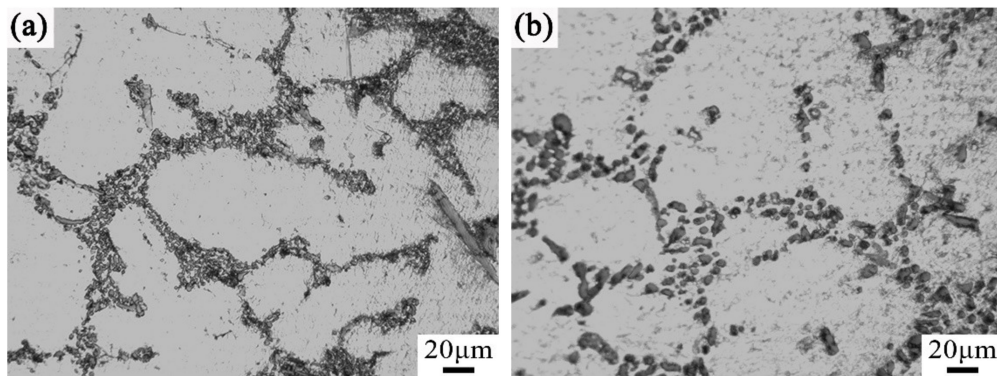


Figure 3. The microstructure of the Al-7Si-0.55Mg alloy: (a) As-cast and (b) with conventional heat treatment.

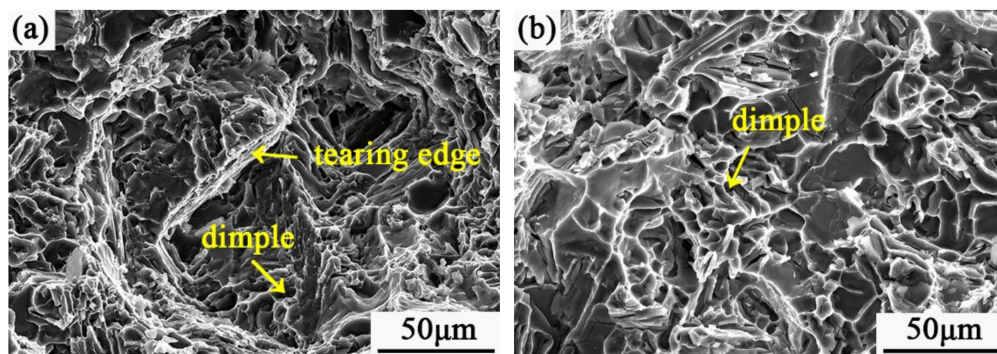


Figure 4. Fracture morphology of the Al-7Si-0.55Mg alloy: (a) As-cast and (b) with conventional heat treatment.

Figure 5 shows the microstructure of the Al-7Si-0.55Mg alloy after electric pulse assisted aging treatment (5 h), in which pulse frequency was 10 Hz, conduction time was 30 s, and pulse currents were 300 A, 600 A, 900 A, 1200 A, respectively. The microstructure of the Al-7Si-0.55Mg alloy after pulse current aging treatment was mainly composed of the α -Al matrix, eutectic Si and a small amount of β' (Mg_2Si). As shown in Figure 5, the morphology of eutectic Si distributed along the grain boundaries was spherical or as a short rod. There were some black impurity phases at the grain boundary. With the increase of pulse current, the electric pulse accelerated the nucleation of the phase transformation by decreasing the thermodynamic energy barrier. The thermodynamic energy barrier to be overcome in phase nucleation decreased, which increased the nucleation rate of the precipitated phase. As shown in Figure 5d, the morphology of eutectic Si was more round and evenly distributed in the α -Al matrix at a pulse current of 1200 A. Figure 6 shows the microstructure of the Al-7Si-0.55Mg alloy after electric pulse assisted aging treatment (5 h), in which pulse frequency was 40 Hz, conduction time was 30 s, and pulse current were 300 A, 600 A, 900 A, and 1200 A, respectively. It can be seen from Figure 6 that the microstructure changes of the Al-7Si-0.55Mg alloy after the electric pulse aging treatment was not obvious. The morphology of the eutectic Si distributed along the grain boundaries was spherical or as a short rod distributed in the α -Al matrix. With the increase of pulse frequency, the convection of atoms in the alloy increased, and the precipitation rate of Mg and Si elements from supersaturated solid solution increased. However, there was an optimal value for pulse frequency.

As discussed above, the microstructure of the Al-7Si-0.55Mg alloy after electric pulse aging treatment consisted mainly of the α -Al matrix, eutectic Si and a small amount of β' (Mg_2Si). It was found that the electric pulse accelerated the nucleation of phase transformation by decreasing the thermodynamic energy barrier, which increased the nucleation rate of the precipitated phase. The internal atoms struck in two directions during the electric pulse assisted aging treatment of the Al-7Si-0.55Mg alloy because of the combination of the electric and the temperature field. The contents of silicon and magnesium were constant in the Al-7Si-0.55Mg alloy, so the number of precipitates were

continuously precipitating from the supersaturated solid solution. Electric pulse increased the atomic diffusion rate, the nucleation rate of eutectic and the precipitated phase (Mg_2Si), which was beneficial to the vacancy movement. It led to obtaining more uniform and finer precipitates.

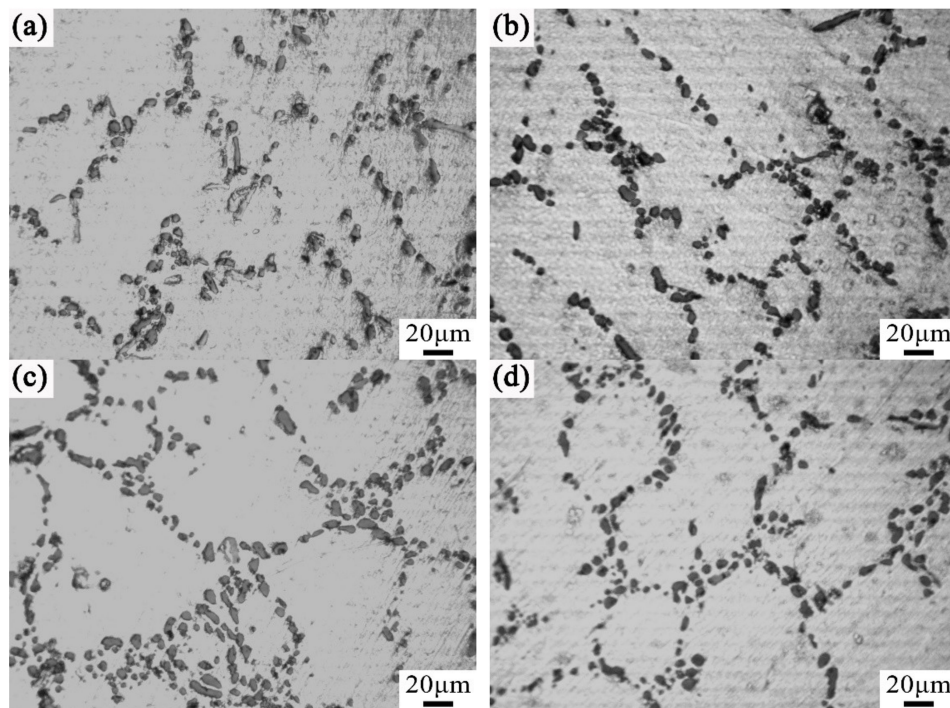


Figure 5. The microstructure of the Al-7Si-0.55Mg alloy with different pulse currents at a pulse frequency of 10 Hz: (a) 300 A, (b) 600 A, (c) 900 A and (d) 1200 A.

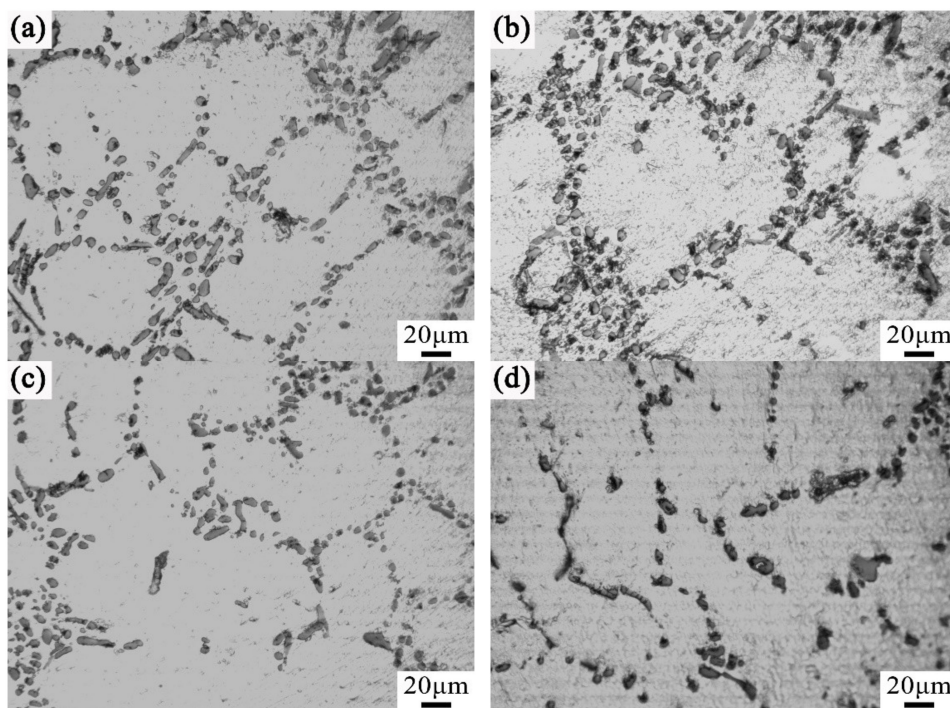


Figure 6. The microstructure of the Al-7Si-0.55Mg alloy with different pulse currents at a pulse frequency of 40 Hz: (a) 300 A, (b) 600 A, (c) 900 A and (d) 1200 A.

Figure 7 shows the bright field images and high-resolution transmission electron microscopy (HRTEM) images of the Al-7Si-0.55Mg alloy along the direction of $[110]_{\text{Al}}$ after conventional heat

treatment and aging treatment with different pulse parameters. Figure 7a–d shows the bright field images and HRTEM images of the Al-7Si-0.55Mg alloy after electric pulse aging treatment (160 °C/5 h) with 10 Hz and 40 Hz, 300 A and 1200 A, respectively. There are some large dark gray Si particles, spherical and short rod precipitates in Figure 7. The spherical precipitates were identified as GP zones, which were coherent with the α -Al matrix and the size of the GP zone was small. Owing to the subtle content of Mg in the Al-7Si-0.55Mg alloy, the precipitates of the GP zones or β' phase (Mg_2Si) were less after the aging treatment. As shown in Figure 7a, there were some spherical precipitates with a diameter of less than 6 nm and a few different lengths of rod precipitates about 15 nm–31 nm in length, 3 nm–6 nm in thickness. As shown in Figure 7b, the Si particles decreased obviously, and the rod precipitated phase of Mg_2Si increased gradually. The length and width of the precipitation phase (Mg_2Si) was about 11 nm–32 nm and 4 nm–7 nm. As shown in Figure 7c, d, Si particles were unevenly distributed in the α -Al matrix and the rod precipitated phase of Mg_2Si decreased, obviously. The morphology of precipitates and selective electron diffraction (SAED) at the conventional heat treatment are shown in Figure 7e, which had less spherical and rod precipitates. Compared with the conventional heat treatment, although the time of electric pulse assisted aging treatment was 3 h shorter than that of conventional aging, the spherical precipitates were more evenly distributed in the α -Al matrix. The pulse current promoted the nucleation and growth processes of precipitates, which provided priority conditions for the nucleation of the precipitates phase.

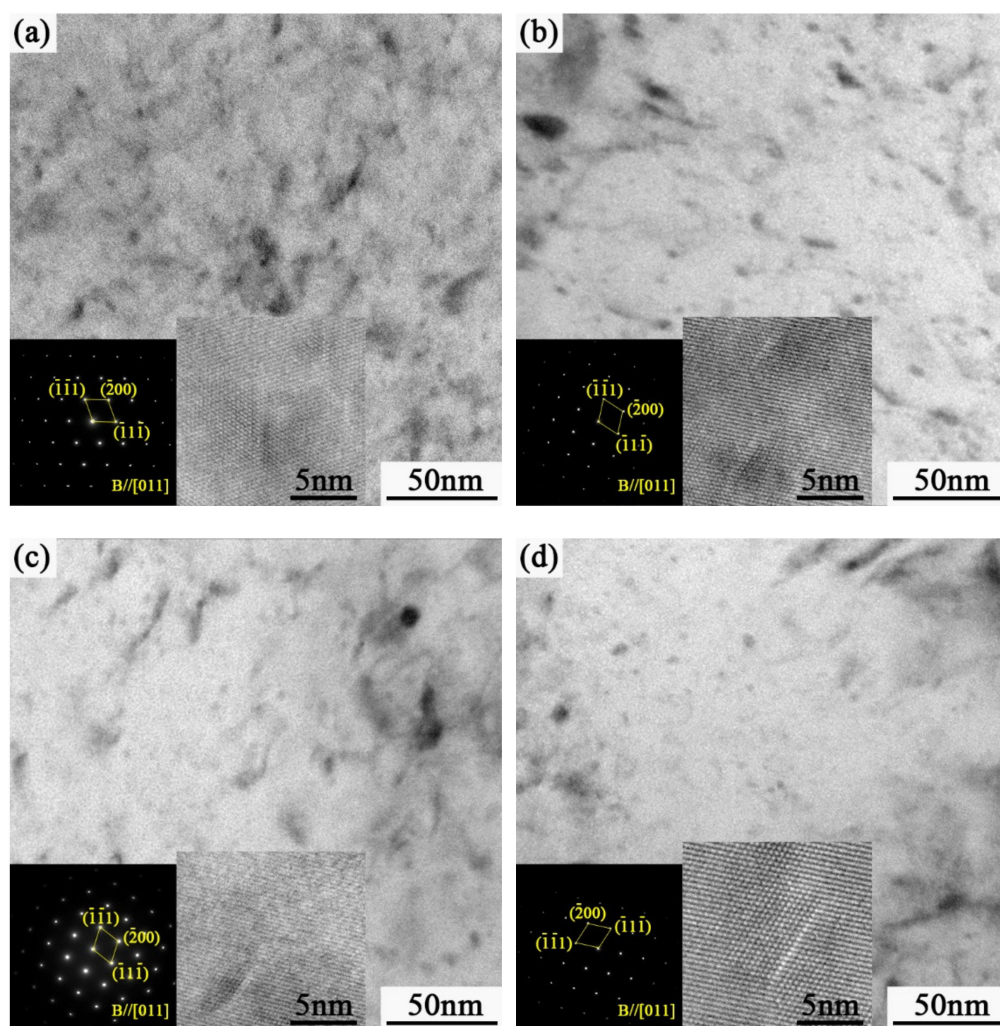


Figure 7. Cont.

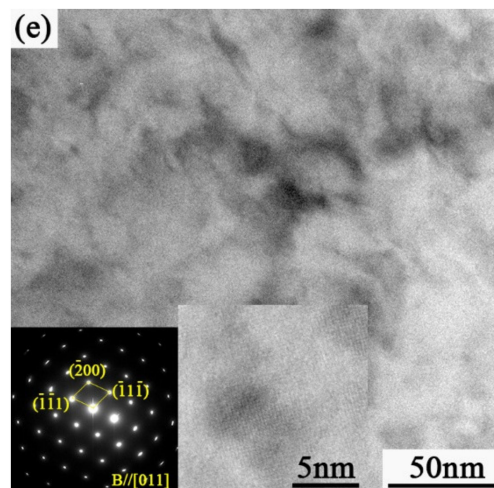


Figure 7. Bright field images and high-resolution transmission electron microscopy (HRTEM) images of the Al-7Si-0.55Mg alloy after aging treatment at different conditions: (a) 10 Hz, 300 A; (b) 10 Hz, 1200 A; (c) 40 Hz, 300 A; (d) 40 Hz, 1200 A and (e) conventional heat treatment.

3.2. Mechanical Properties

The tensile strength and elongation of the Al-7Si-0.55Mg alloy after aging treatment at different conditions are shown in Figure 8. The strength and elongation of the as-cast Al-7Si-0.55Mg alloy were 183 MPa and 6%, respectively. After conventional heat treatment, the tensile strength and elongation of the alloy were significantly increased to 279.1 MPa and 8.2%, respectively. The elongation of the Al-7Si-0.55Mg alloy was significantly improved after electric pulse assisted aging, which was higher than that after the conventional heat treatment. The strength of the Al-7Si-0.55Mg alloy after electric pulse assisted aging was similar to that of conventional heat treatment. With the increase of the pulse current, the electric pulse accelerated the nucleation of the phase transformation by decreasing the thermodynamic energy barrier, which increased the nucleation rate of the precipitated phase. When the pulse frequency and pulse current were 10 Hz and 1200 A, the strength and elongation of the alloy were the highest and the spherical precipitates were more evenly distributed in the α -Al matrix.

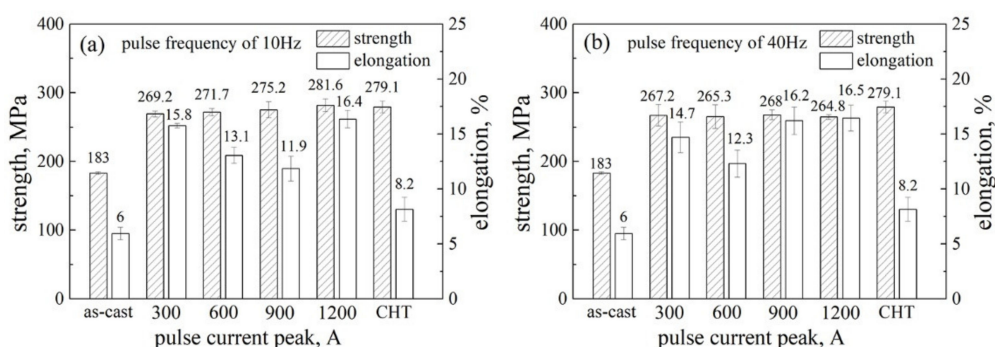


Figure 8. Tensile strength and elongation of the Al-7Si-0.55Mg alloy after aging treatment at different conditions: (a) 10 Hz; (b) 40 Hz.

The hardness of the Al-7Si-0.55Mg alloy after aging treatment at different conditions is shown in Figure 9. The hardness of the as-cast Al-7Si-0.55Mg alloy was 98 HV, which increased to 135 HV after conventional heat treatment. The hardness of the Al-7Si-0.55Mg alloy after conventional heat treatment was slightly higher than that of the electric pulse assisted aging treatment. When the pulse current was 1200 A and the pulse frequency was 10 Hz and 40 Hz, the hardness of the alloy was 137 HV and 133 HV, respectively.

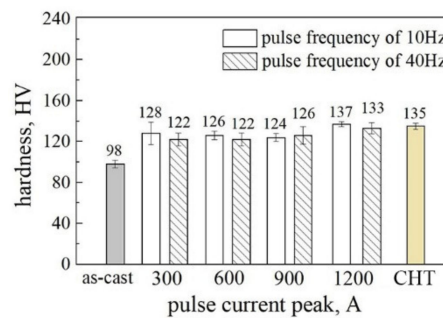


Figure 9. The hardness of the Al-7Si-0.55Mg alloy after aging treatment at different conditions.

4. Discussion

The precipitation sequence of the aging process for the Al-Si-Mg alloy is generally considered to be as follows: Supersaturated solid solution $\rightarrow$ GP zone $\rightarrow$ metastable phase β' (Mg_2Si) $\rightarrow$ stable phase β (Mg_2Si) [15]. At the beginning of the aging process, the atoms of Si and Mg recombine in the α -Al matrix. Due to the uneven distribution of components, the atomic segregation zone is formed, which is the GP zone. The GP zone has very fine particles with sizes in the range of 1~3 nm. The GP zone is spherical and coherent with the α -Al matrix. With the extension of aging time, the GP zone gradually changes to the β' phase. The β' phase is semi-coherent with the α -Al matrix and has a shape of fine needles, and is considered to be the most effective strengthening phase of the alloys. When the temperature and time of aging treatment increase, the metastable phase β' (Mg_2Si) gradually changes to the stable phase β (Mg_2Si). The phase is non-coherent with the α -Al matrix and has a shape of a rod, and their sizes are much larger than the β' phase. The lattice distortion in the alloy decreases, so that the strength and hardness of the alloy slightly decreases, while the ductility and toughness increase significantly.

The aging treatment of the alloy is a solid phase transition process in which the second phase particles precipitate from the supersaturated solid solution into nucleation and growth. During phase transition, nucleation and growth of the new phase require a large amount of energy. Electric pulse processing technology can directly transfer energy to atoms in a short time, making the atoms in the alloy migrate rapidly. High-velocity atoms overcome the energy barrier required for phase transition to achieve an efficient use of energy. It overcomes the bottleneck that advanced manufacturing technology can develop rapidly [16]. The energy introduced by the electric pulse includes two aspects. On the one hand, a large number of electrons move toward metal atoms to cause an increase in the internal energy of the material, which is called the thermal effect, and its macroscopic expression is an increase in the temperature of the alloy. On the other hand, the pulse current interacts with defects in the alloy to improve its mobility, so that the precipitated phase can be rapidly precipitated. This part of energy is called non-thermal effect. The main effect of this experiment is the non-thermal due to the short conduction time during the aging treatment of the electric pulse assisted Al-7Si-0.55Mg alloy. Previous studies [17,18] indicated that the effect of electric pulse enhanced the nucleation of phase transformation by decreasing the thermodynamic energy barrier. This effect of the electric pulse on the phase transformation can be described with Equation (1):

$$\Delta G^{\text{EPT}} = \Delta G_0 + \Delta G_e \quad (1)$$

$$\Delta G_e = \mu g \xi(\sigma_0, \sigma_1) j^2(t) \Delta V \quad (2)$$

where ΔG^{EPT} is the free energy change of the system for the phase transformation in electric pulse assisted aging treatment, ΔG_0 is the free energy change of the system for the phase transformation in conventional aging treatment, and ΔG_e is the free energy change of the system in nucleation due to the change of pulse current. μ is the magnetic permeability in vacuum, g is a positive geometric factor for coarse grained materials, $j(t)$ is the current density, ΔV is the volume of the new formed

phase, and $\xi(\sigma_0, \sigma_1)$ is a factor which depends on the electrical properties of the parent phase and the new phase, $\xi(\sigma_0, \sigma_1) = \sigma_1 - \sigma_0/\sigma_0 + 2\sigma_1$, σ_0 and σ_1 are the electric conductivities of α phase and β phase, respectively [19–22]. During the phase transformation in electric pulse assisted aging treatment, $\sigma_1 < \sigma_0$ results in $\xi(\sigma_0, \sigma_1) < 0$, therefore, $\Delta G_e < 0$ according to Equation (2).

According to the classical nucleation theory, the precipitation phase will grow up only when the radius of the precipitated phase nucleation is larger than the critical nucleation radius. The critical nucleation number of the precipitated phase in the parent phase during electric pulse assisted aging treatment is [23]:

$$N^* = N_0 \exp\left(\frac{-G_c}{kT}\right) \quad (3)$$

where G_c is the thermodynamic energy barriers to be overcome in phase nucleation, $G_c = \max\{\Delta G^{\text{EPT}}\} = \max\{\Delta G_0 + \Delta G_e\}$, N_0 is the number of nucleation in the parent phase, k is the Boltzmann constant, and T is the phase transformation temperature.

This implies that an electric pulse can accelerate the nucleation of phase transformation by decreasing the thermodynamic energy barrier. The thermodynamic energy barriers to be overcome in phase nucleation decrease, which increases the nucleation rate of the β' phase. Compared with conventional heat treatment, although the time of electric pulse assisted aging treatment is 3 h shorter than that of conventional aging, the spherical precipitates are more evenly distributed in the α -Al matrix. The elongation of the alloy is significantly improved after electric pulse assisted aging treatment. The pulse current promotes the nucleation and growth process of precipitates, which provides priority conditions for the nucleation of precipitates phase.

5. Conclusions

- (1) The microstructure of the as-cast Al-7Si-0.55Mg alloy is mainly composed of the α -Al matrix and eutectic (α -Al + Si). The morphology of eutectics in the alloy is mainly a long and thin strip. Tearing effects play a negative role in the matrix because of its sharp end, which limit the properties of as-cast alloys. During the solid solution treatment of the Al-7Si-0.55Mg alloy, the long strip Si phases were fused and spheroidized into short strips or spheres. Compared with the as-cast alloy, the morphology of eutectic Si distributed along grain boundaries was spherical or as a short rod after heat treatment.
- (2) The strength and elongation of the as-cast Al-7Si-0.55Mg alloy were 183 MPa and 6%, respectively. After conventional heat treatment, the tensile strength and elongation of the alloy were significantly increased to 279.1 MPa and 8.2%. The elongation of the Al-7Si-0.55Mg alloy was significantly improved after electric pulse assisted aging, which was higher than that after conventional heat treatment. The strength of the Al-7Si-0.55Mg alloy after electric pulse assisted aging was similar to that of conventional heat treatment. When the pulse frequency and pulse current were 10 Hz and 1200 A, the strength and elongation of the alloy were the highest. Compared with conventional heat treatment, although the time of electric pulse assisted aging treatment was 3 h shorter than that of conventional aging, the spherical precipitates were more evenly distributed in the α -Al matrix. The pulse current promoted the nucleation and growth process of precipitates, which provided priority conditions for the nucleation of the precipitates phase.

Author Contributions: Conceptualization, D.L. and D.S.; Data curation, K.K.; Formal analysis, K.K.; Investigation, A.W.; Software, G.G.; Writing—original draft, K.K.; Writing—review & editing, K.K.

Funding: The research was financially supported by the Research and Development Project of Application Technology in Harbin, Heilongjiang Province of China (2017RAYXJ017).

Conflicts of Interest: The authors declare no conflict of interest.

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