Phase evolution of a novel Al–Zn–Mg–Cu–Zr–Sm alloy during homogenization annealing treatment

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Abstract
The effects of pretreatment and homogenization on the microstructure of a new Al–6.7Zn–2.6Mg–2.0Cu–0.1Zr–0.3Sm alloy were investigated. The results show that severe dendritic segregation is visible at the grain boundaries of the as-cast microstructure, which consists of the η (Mg (Zn, Cu, Al))2, Al10Cu7Sm2 and Fe-rich phases. Besides, some small η and needle-shaped θ (Al3Cu) phases are distributed inside of the grains. After pretreatment at 400 °C for 10 h, the η phase and θ phase in the grains are dissolved, and Al3Zr particles are precipitated inside of the grains by homogeneous nucleation. However, dendritic segregation still exists at the grain boundaries. After homogenization annealing, the η phase present at the grain boundaries completely dissolves, and only a small number of Al10Cu7Sm2 and Fe-rich phases remain. The alloy microstructure becomes more uniform and the volume fraction of residual eutectic decreases to 0.70% after homogenization annealing. The optimal homogenization processing is determined as 400 °C/10 h + 470 °C/24 h, which is in perfect accordance with the results obtained via homogenizing kinetic analysis.

1. Introduction

Al–Zn–Mg–Cu–Zr alloys are widely used in the fields of transportation, construction, and weapon manufacturing owing to their tremendous strength, high corrosion resistance, and excellent processing performance [1–4]. However, the high requirements in the auto and aerospace industry, conventional Al–Zn–Mg–Cu–Zr alloys cannot satisfy the industrial requirements [5–8]. At present, adding rare earth elements into the alloy has become the most effective way of optimizing the alloy’s mechanical properties [9–11]. Nonetheless, a large number of non-equilibrium eutectic phases and coarse crystalline intermetallic particles can be precipitated from Al–Zn–Mg–Cu–Zr–RE alloys during the solidification process [12–14]. Moreover, Mg, Zn, Cu tend to cause serious microsegregation at grain boundaries. This sort of as-cast microstructure does not only affect the alloy’s subsequent deformation characteristics but also decreases the mechanical properties of subsequently processed products [15–17]. Therefore, homogenization heat processing is imperative to obtain an adequate service performance for the Al–Zn–Mg–Cu–Zr–RE alloys.

A multitude of research projects have been carried out on the microstructure characteristics evolution of Al–Zn–Mg–Cu–Zr (–RE) alloys during homogenization treatment. Xiao et al investigated the influences of homogenization treatment on the microstructure and mechanical properties of Al–Zn–Mg–Cu–Zr alloys. Their experimental results revealed that small-sized and hyperdense Al3Zr phases are obtained after heat treatment at 350 °C, which significantly boosts the mechanical properties of the alloys [18]. Liu et al studied the microstructure evolution of as-cast Al–8.35Zn–2.5Mg–2.25Cu alloy by homogenization annealing treatment. They noticed that the as-cast alloy consists of the α-Al phase, Fe-rich phase, and eutectic Mg (Zn, Cu, Al)2 phase. After homogenization at 470 °C/1 h, the content of the Mg(Zn, Cu, Al)2 phase decreased sharply, and the network-like structure of Mg (Zn, Cu, Al)2 gradually evolved into discontinuous Mg (Zn, Cu, Al)2 particles [19]. Jia et al conducted the single-stage homogenization treatment of 7050 aluminum alloy at different temperatures. They discovered that Zn began to diffuse when the heat treatment temperature reached 380 °C, while the...
diffusion of Mg and Cu started to accelerate as temperature was raised to 440 °C. When the temperature was raised to 450 °C, the alloy’s microstructure became uniform, and only the Al2CuMg phase existed in the α-Al matrix [20]. Wu et al found that after the new Al–Zn–Mg–Cu–Zr–Er alloy was heat treated through a two-stage homogenization annealing at 400 °C/10 h + 465 °C/24 h, not only could the microsegregation of the as-cast alloy be eliminated, but a large number of Al3Er(Zr, Zr) phases with an L12 structure could also be precipitated in the Al matrix [21]. Furthermore, Sm was also identified as a good refining element for aluminum alloys, since it is able to reduce the distance between secondary dendrite arms, refine the grain boundary phase of Al2Cu in aluminum alloys, and improve the mechanical properties of aluminum alloys [10, 22, 23]. However, only a few research projects have been reported on the application of Sm in Al–Zn–Mg–Cu–Zr alloys. The phase composition and homogenization process of Al–Zn–Mg–Cu–Zr–Sm alloys are not clear.

In this paper, the phase composition, phase evolution, and segregation characteristics of a novel Al-6.7Zn-2.6Mg-2.0Cu-0.1Zr-0.3Sm alloy were studied. The effects of different heat treatment temperatures and varying durations of homogenization on the microstructure of this new Al–Zn–Mg–Cu–Zr–Sm alloy were also investigated. Additionally, the composition and morphology of residual phases after heat treatment and the diffusion process of eutectic Mg(Zn, Cu, Al)2 were discussed. Finally, the kinetic analysis and optimum homogenization treatment process were presented.

2. Experimental procedure

2.1. Materials and processing

Pure Al, pure Zn, pure Mg and the intermediate alloys (Al-50% Cu, Al-5% Zr, and Al-20% Sm) were taken as the raw materials for smelting the Al-6.7Zn-2.6Mg-2.0Cu-0.1Zr-0.3Sm alloy. All the raw materials were purchased from Northeast Light Alloy Co., Ltd. Subsequently, the alloy was melted in a box-type high-temperature electrical-resistance furnace and cast into an iron mold (Φ60 mm × 100 mm). The melting temperature was set as 780 °C, while the refining temperature was 740 °C, and C2Cl6 was added for degassing before refining. Before smelting, all the raw materials and molds were preheated at 250 °C. Specimens with a size of 15 × 15 × 15 mm were cut from the ingot by wire-cut. The heat treatment procedure comprises two processes: pretreatment and homogenization. The as-cast specimens were pretreated at 400 °C for 10 h [24, 25]. The homogenization temperature range is set from 440 °C to 480 °C and the homogenization time range is configured from 2 h to 48 h.

3. Characterization

The specimens were ground and etched by the grid and Keller reagent, respectively. The morphology and microstructure of the as-cast, pretreated and homogenized alloy were analyzed by optical microscopy (OM, XD30M SDPTOP), scanning electron microscopy (SEM, Quanta 200, FEI) and scanning transmission electron microscopy (STEM, F200X, FEI) with an operating voltage of 200 kV. X-ray diffraction (XRD) patterns were acquired on a Philips X’Pert x-ray diffractometer using radiation of Cu Kα at a scanning rate of 4° min⁻¹. The evaluation method of the volume fraction of phases was reported in the previous work [21]. Ten SEM figures were used to assess the percentage composition of the alloy’s second phase. The standard deviation of these images with the average value was less than 0.08%.

4. Results and discussion

4.1. Microstructure of the as-cast alloy

Figure 1 shows the morphology and microstructure of the as-cast Al–Zn–Mg–Cu–Zr–Sm alloy. The results indicate that the alloy as-cast microstructure is an obvious eutectic structure exhibiting severe dendrite segregation (figure 1(a)). As shown in figure 1(b), four significant diffraction peaks are observed at 38.5°, 44.5°, 65° and 78° corresponding with (111), (200), (220) and (311) in the Al matrix (JCPDS 04-0787). Also, there are seven identifiable diffraction peaks at 37°, 40.5°, 41.3°, 45.5°, 47°, 65.5°, and 68.7°, which correspond to the MgZn2 phase (JCPDS 34-0457), and one diffraction peak at around 40° corresponding to the Al10Cu2Sm2 phase (JCPDS 36-1276). To further verify the phase morphology and compositions, magnified SEM images and EDS analysis are shown in figure 1(c). A large number of lamellar and skeleton-like eutectic phases are distributed along the grain boundaries, and a small amount of insoluble intermetallic compounds are also present. According to the EDS analysis, the composition of the lamellar phase A is (in %) Al-66.64, Mg-10.23, Zn-12.67, and Cu-9.00, which is close to the η(Mg(Zn, Cu, Al)2) phase [19, 26]. The atomic ratio in the white blocky phase B is close to 10:7:2, and can be determined as the Al10Cu3Sm2 phases. Moreover, the light-grey blocky and needle
phases C are Fe-rich phases \((Al_{23}CuFe_4)\). The Fe-rich phases are formed by the reaction between Al and Cu during the smelting process due to the impurity of Fe in the raw material. Figure 2 shows the microstructure and composition distribution at the grain boundaries of the as-cast alloy. The elements Zn, Mg, Cu, and Sm are relatively abundant in the grain boundary, thus these elements are comparatively less distributed inside the grain. The Zr content is uniformly distributed in the as-cast alloy, and the impure Fe accumulates at the grain boundaries. On observation, small rod-like \(\eta\) phases (in figure 3(a), (A) and (B)) and needle \(\theta\) \((Al_2Cu)\) phases (in figure 3(a), (C) and (D)) were present in the grains, whereas no dispersed \(Al_2Zr\) phase was found. However, the grain size is strongly depended on the \(Al_2Zr\) precipitation due to the fact that these primary phases are potent grain refiners. Therefore, pretreatment is necessary to obtain the dispersion of \(Al_2Zr\), inhibiting the recrystallization ability of the alloy in order to prevent the growth of the grains [27].

5. Microstructure of the pretreated alloy

Figure 4 shows the morphology and microstructure of the Al–Zn–Mg–Cu–Zr–Sm alloy after pretreatment. Compared to the as-cast alloy, the content of the second phase at the grain boundary decreases (figures 4(a) and (b)). The \(\eta\) phase, \(Al_{10}Cu_5Sm_2\) phase, and \(Al_{23}CuFe_4\) phase can still be observed in the grain boundaries of the pretreated alloy (arrow A, B, and C in figure 4(c)). Figure 5 exhibits the dark field phase and element distribution of the alloy grain after pretreatment at 400 °C/10 h. Due to the diffusion of Mg, Zn, and Cu elements, the \(\eta\) and \(\theta\) phases dispersed in the grains are dissolved. Besides, some new granular phases of \(Al_2Zr\) appear inside of the grains. The formation of these \(Al_2Zr\) precipitates in the alloy is a process of desolvation and precipitation of a supersaturated solid solution. The nucleation rate of \(Al_2Zr\) is highly dependent on the supercooling degree of the phase transformation [28]. Consequently, the \(Al_2Zr\) phase with small size and high density is formed in the alloy due to the high degree of supercooling during low-temperature pretreatment [29].

As we know, the effectiveness of the precipitate to prevent recrystallization can be quantitatively expressed by formula (1) [30]:

\[
\text{Effectiveness} = \frac{\text{Precipitate Size}}{\text{Precipitate Density}}
\]
where $P_Z$ is the pinning force, $\gamma_{\text{GB}}$ represents the grain boundary energy; $f$ and $r$ are the volume fraction and average radius of the precipitated phase, respectively. According to formula (1), the $\text{Al}_3\text{Zr}$ (higher $f/r$) phases with smaller sizes and larger volume fractions would lead to a higher $P_Z$. These $\text{Al}_3\text{Zr}$ phases are capable of binding the grain boundary and affect the speed of recrystallization, which inhibits the growth of the grains. However, a large number of eutectic phases remain at the grain boundaries of the alloy, and further homogenization is needed.

6. Microstructure of the homogenization alloy

Figure 6 shows the SEM images of the alloy after 24 h of homogenization annealing holding time at different temperatures. As the homogenization temperatures rose from 440 °C to 460 °C, the size and the volume fraction of the second phase in the alloy gradually decreases. After being treated at 470 °C, only a small amount of insoluble block and strip-like second phase was left on the grain boundaries. In addition, the microstructure of the remelting ball and triangular grain boundary became apparent (figure 6(f)). The volume fractions of the second phases with different homogenization temperatures are laid out in figure 7. Which demonstrates that compared to the pretreated alloy (3.53%), the volume fraction of second phases after homogenization treatment at temperatures of 440 °C, 450 °C, 460 °C, 470 °C and 480 °C are 2.22%, 1.52%, 0.96%, 0.70% and 0.67%, respectively. The results suggest that the lamellar-like $\eta$ phase at the grain boundaries dissolve into the $\alpha$-Al matrix. When the homogenization temperature reaches 470 °C, only a small amount of insoluble block-like $\text{Al}_{10}\text{Cu}_2\text{Sm}_2$ and strip-like Fe-rich phases remain at the grain boundaries. The microstructure of the alloy became uniform and the dendrite segregation was eliminated. During the homogenization annealing treatment, various elements in the eutectic phase diffused into the $\alpha$-Al matrix, and the $\eta$ phase gradually dissolved. The higher the homogenization temperature, the faster each element diffuse, hence the greater the degree of phase dissolution. Notwithstanding, overburning defects occur when the homogenization temperature exceeds 480 °C. As a result, 470 °C is selected as the optimal homogenization temperature of this new Al–Zn–Mg–Cu–Zr–Sm alloy. This findings can also be supported by the XRD curves (figure 8). We noticed that the pretreated sample is mainly composed of the $\alpha$-Al phase and $\eta$ phase. With increase in the homogenization temperature, the diffraction peak intensity of the $\eta$ phase gradually decreases. When the homogenization temperature is 470 °C, the peak of the $\eta$ phase disappears, indicating that the $\eta$ phase has been dissolved into the $\alpha$-Al matrix. Nevertheless, the diffraction peak of the $\text{Al}_{10}\text{Cu}_2\text{Sm}_2$ phase changes slightly when the temperature is raised to
480 °C, suggesting that the melting point of the Al<sub>10</sub>Cu<sub>7</sub>Sm<sub>2</sub> phase is higher than that of the homogenization temperature.

Figure 9 shows the morphologies and EDS images of the Al–Zn–Mg–Cu–Zr–Sm alloy after being homogenized at 470 °C for various holding times. Compared to the pretreated alloy, the microstructure of the alloy is slightly altered after homogenization for 2 h and 4 h (figures 9(a) and (b)). Three types of phases (η, Al<sub>10</sub>Cu<sub>7</sub>Sm<sub>2</sub>, and Fe-rich phase) are still distributed on the grain boundaries in a continuous mesh pattern. Notwithstanding, the Cu content in the alloy’s η phase is higher than that of the pretreated alloy, which is attributed to the slow diffusion rate of Cu in the Al alloy [20, 31]. When the homogenization time increases to 8 h and 12 h, respectively, a large quantity of the second phase at the grain boundaries dissolves into the α-Al matrix. It can be perceived that the continuous lamellar-like η phase gradually decreases (figures 9(c) and (d)). Withal, the Al<sub>10</sub>Cu<sub>7</sub>Sm<sub>2</sub> phase and Fe-rich phase remain at the boundaries (figure 9(d)). When the homogenization time is set as 24 h, the η phase disappears completely and only a small amount of Al<sub>10</sub>Cu<sub>7</sub>Sm<sub>2</sub> phase and Al<sub>13</sub>CuFe<sub>4</sub> phase remains at the boundaries (figure 9(e)). With further increment in the homogenization time to 48 h, the microstructure of the alloy does not undergo any remarkable change. Moreover, the change in the second phase content at different homogenization times can be represented by statistical graphs (figure 10). The volume fractions of the second phase decrease sharply within the initial 8 h and then decline slightly with increase in the homogenization time.

XRD curves of the alloy at different durations of homogenization are exhibited in figure 11. Two diffraction peaks can be observed, corresponding to the η phase and Al<sub>10</sub>Cu<sub>7</sub>Sm<sub>2</sub> phase. With increase in the homogenization time, the diffraction peak intensity of the η phase progressively decreases. When the homogenization time reaches 24 h, the diffraction peak of the η phase completely disappears and the diffraction peak of the Al<sub>10</sub>Cu<sub>7</sub>Sm<sub>2</sub> phase changes lightly. Also, from the homogenized STEM pictures (figure 12), we can see that Al<sub>2</sub>Zr still has a spherical shape distribution in the matrix (at A), and its size does not vary significantly
from that of the pretreated alloy (both are 20–60 nm). During the homogenization process, the \( \eta \) phase is converted into the S (Al\(_2\)CuMg) phase. During further homogenization of the S phase, the elements’ diffusion gradually decline [14, 19]. B containing Cu and Mg is the remaining nano-scale S phase. Thence, 470 °C/24 h is selected as the optimal homogenization parameter for the Al–Zn–Mg–Cu–Zr–Sm alloy.

7. Homogenization kinetic analysis

Based on the element distribution diagram of the as-cast Al–6.7Zn–2.6Mg–2.0Cu–0.1Zr–0.3Sm alloy (figure 2), it can be seen that the main elements Zn, Mg, Cu and trace element Sm are abundant in the interdendritic region. Therefore, knowledge and application of the diffusion laws along the dendrite region are important to analyze elements distribution during the homogenization process [19, 21]. According to the laws of diffusion kinetics, the initial concentration of alloying elements along the dendritic regions can be expressed by Fourier series components in a cosine function [12, 32]:

\[
C(x) = \overline{C} + A_0 \cos \frac{2\pi x}{L}
\]  
(2)

where \( \overline{C} \) is the average concentration of the elements, \( L \) is the interdendritic space, and \( A_0 \) is the initial amplitude of the composition segregation, which can be defined by the following equation:

\[
A_0 = \frac{1}{2}(C_{\text{max}} - C_{\text{min}}) = \frac{1}{2} \Delta C_0
\]  
(3)

Applying boundary conditions to Fick’s second law, \( A(t) \) is given as [12, 31]:

\[
A(t) = A_0 \exp \left( -\frac{4\pi^2}{L^2} Dt \right)
\]  
(4)
Figure 5. Dark field phase and element distribution diagram of pretreatment alloy.

Figure 6. SEM images of the alloy after homogenization annealing at different temperatures for 24 h; (a) 440 °C, (b) 450 °C, (c) 460 °C, (d) 470 °C and (e) 480 °C.
The relationship between the diffusion coefficient and temperature can be written as [19]:

\[ D = D_0 \exp \left( \frac{-Q}{RT} \right) \]  

(5)

Substituting equation (5) in equation (4), the equation can be re-written as,

\[ A(t) = A_0 \exp \left[ -\frac{4\pi^2D_0t}{L^2} \exp \left( \frac{-Q}{RT} \right) \right] \]  

(6)

Assuming that the element distribution is homogenous when the composition segregation amplitude is reduced to 1% [33], that is,
At

\[ A(t) = \frac{A(0)}{100} \]  \tag{7}  

Then,

\[ \exp \left[ -\frac{4\pi^2 D_h t}{L^2} \exp \left( -\frac{Q}{RT} \right) \right] = \frac{1}{100} \]  \tag{8}
By taking natural logarithms of both sides, equation (8) can be expressed as:

\[
\frac{1}{T} = \frac{R}{Q} \ln \left( \frac{4\pi^2D_0t}{4.6L^2} \right)
\]

where \(D_0\) is the diffusion coefficient, \(Q\) represents the diffusion activation energy, \(R\) denotes the gas constant, \(T\) and \(t\) are the homogenization temperature and time, respectively.

In the present work, the space \((L)\) of the \(\eta\) phase can be used to describe the distribution in the pretreated alloy by the section line method. More than 97% of the spacing \(L\) is larger than 30 \(\mu m\), with the maximum \(L\) value being about 110 \(\mu m\) (figure 13). Moreover, the second phase between dendrites consists of \(Zn\), \(Mg\), and \(Cu\) elements, and the diffusion coefficient of \(Cu\) in the Al alloy is much lower than that of \(Mg\) and \(Zn\) at the same temperature \((D_{Cu} < D_{Mg} < D_{Zn})\). Consequently, the kinetics of the entire homogenization process can be
Figure 12. Dark field phase and element distribution diagram of homogenized alloy.

Figure 13. Distribution of η interphase distance L in pretreated alloy.
controlled by Cu, $D_0(Cu) = 0.084 \text{ cm}^2 \text{ s}^{-1}$, $Q(Cu) = 136.8 \text{ kJ/mol}$, and $R = 8.31 \text{ kJ/(mol·K)}$ [34, 35]. The homogenization kinetic curves of the $\eta$ phase with different $L$ values can be obtained by substituting these parameters into equation (5) (figure 14). It is obvious that with the increase in the $\eta$ phase spacing ($L$), higher temperatures and longer times are needed for complete homogenization. Withal, according to the present results (figure 6), the homogenization temperature should not exceed 470 °C. When the homogenization temperature is 470 °C, the homogenization time for the $\eta$ phase with a space ($L$ value) of 110 $\mu$m is 23.2 h. This points out that the segregation along the Al grain boundaries is almost eliminated. Ultimately, the alloy’s optimal homogenization time at 470 °C is determined as 23.2 h, which is consistent with the experimental results in this work.

8. Conclusions

(1) The as-cast Al–Zn–Mg–Cu–Zr–Sm alloy demonstrated severe segregation. The primary phases at the grain boundaries are the $\eta$ phase, $\text{Al}_{10}\text{Cu}_7\text{Sm}_2$ phase, and $\text{Al}_{23}\text{CuFe}_4$ phase.

(2) After pretreatment at 400 °C for 10 h, the second phase in the grain boundary does not exhibit any remarkable change. The intragranular $\eta$ phase and $\theta(\text{Al}_2\text{Cu})$ phase disappeared, and a new granular precipitate phase $\text{Al}_3\text{Zr}$ appears in the grains of the alloy.

(3) After the pretreated alloy is homogenized at 470 °C for 24 h, the $\eta$ phase completely dissolves in the Al matrix, but the melting points of the $\text{Al}_{10}\text{Cu}_7\text{Sm}_2$ and $\text{Al}_{23}\text{CuFe}_4$ phases are higher than the homogenization temperature, thus these phases could not completely dissolve in the Al matrix. Following a thorough analysis, the optimized homogenization scheme of the Al–Zn–Mg–Cu–Zr–Sm alloy is determined as (400 °C, 10 h) + (470 °C, 24 h).

(4) A homogenization kinetic equation derived from the Gauss diffusion mathematical model is established and used to verify the accuracy of the optimum homogenization parameter.

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References

[1] Cao F H, Zheng J X, Jiang Y, Chen B, Wang Y R and Hu T 2019 Acta Mater. 164 207–19
[2] Liu L, Jia Y Y, Jiang J T, Zhang B, Li G A, Shao W Z and Zhen L 2019 J. Alloys Compd. 799 1–14
[3] Dong P X, Chen S Y and Chen K H 2019 J. Alloys Compd. 788 329–37
[4] Chen L, Chen G J, Tang J W, Zhao G Q and Zhang C S 2019 Mater. Charact. 158 109953
[5] Li Z M, Jiang H C, Wang Y L, Zhang D, Yan D S and Rong L J 2018 J. Mater. Sci. Technol. 34 1172–9
[6] Fang H C, Chao H and Chen K H 2014 Mater. Sci. Eng. A 610 10–6
[7] Zhang X G, Mei F Q, Zhang H Y, Wang S H, Fang C F and Hao H 2012 Mater. Sci. Eng. A 552 230–5
[8] Fang H C, Chen K H, Chen X, Huang L P, Peng G S and Huang B Y 2011 Mater. Sci. Eng. A 528 7606–15
[9] Alexopoulos N D, Tiryakioğlu M, Vasilakos A N and Kourkoulis S K 2014 Mater. Sci. Eng. A 604 40–5
[10] Qiu H X, Yan H and Hu Z 2013 J. Alloys Compd. 567 77–81
[11] Chen Z W, Chen P and Ma C Y 2012 Rare Met. 31 352–5
[12] Deng Y, Yin Z M and Cong F G 2012 Intermetallics 26 114–21
[13] Yoganjaneyulu G, Babu K A, Siva G V, Vigneshwaran S and Narayanans C S 2019 Mater. Lett. 253 18–21
[14] Mondal C and Mukhopadhyay A K 2005 Mater. Sci. Eng. A 391 367–76
[15] Yuan S, Chen L, Tang J, Zhao G, Zhang C and Yu J 2019 J. Mater. Sci. 54 9843–56
[16] Bita P, Hamed M and Masoud E 2017 J. Mater. Res. 32 4186–95
[17] Wong T W, Hadadzadeh A, Benoit M J and Wells M A 2018 J. Mater. Process. Tech. 254 238–47
[18] Xiao T, Deng Y L, Ye L Y, Lin H Q, Shan C J and Qian P W 2016 Mater. Sci. Eng. A 675 280–8
[19] Liu Y, Jiang D M, Xie W L, Hu J and Ma B R 2014 Mater. Charact. 93 173–83
[20] Jia P F, Cao Y H, Geng Y D, He L Z, Xiao N and Cai J Z 2014 Mater. Sci. Eng. A 612 335–42
[21] Wu H, Wen S P, Lu J T, Mi Z P, Zeng X L, Huang H and Nie Z R 2017 T. Nonferr. Metal. Soc. China 27 1476–82
[22] Hu Z, Hong Yan H and Rao Y S 2013 T. Nonferr. Metal. Soc. China 23 3228–34
[23] Li Z, Hu Z and Yan H 2016 J. Wuhan Univ. Technol. 31 624–9
[24] Guo Z, Zhao G and Chen X 2016 Mater. Charact. 114 79–87
[25] Guo Z, Zhao G and Chen X 2015 Mater. Charact. 102 122–30
[26] Fan X G, Jiang D M, Meng Q C, Li N K and Sun Z X 2006 T. Nonferr. Metal. Soc. China 16 1247–1250.
[27] Robson J D and Prangnell P B 2003 Mater. Sci. Eng. A 352 240–50
[28] Deng Y L, Zhang Y Y, Wan L, Zhu A A and Zhang X M 2013 Metall. Mater. Trans. A 44 2470–7
[29] Knipling K E, Dunsand D C and Seidman D N 2007 Metall. Mater. Trans. A 38 2552–63
[30] Humphreys F J 1997 Acta Mater. 45 5031–9
[31] Li W B, Pan Q L, Xiao Y P, He Y B and Liu X Y 2011 T. Nonferr. Metal. Soc. China 21 2127–33
[32] Zhang F, Shen J, Yan X D, Sun J L, Sun X L and Yang Y 2013 Rare Met. 33 28–36
[33] Liu X Y, Pan Q L, Fan X, Hu Y B, Li W B and Liang W J 2009 J. Alloys Compd. 484 790–4
[34] Xu L, Li Z H, Wang G J, Li X W, Lv X Y, Zhang Y A, Fan Y Q and Xiong B Q 2017 Mater. Charact. 131 285–97
[35] Wang H J, Xu J, Kang Y L, Tang M G and Zhang Z F 2014 J. Alloys Compd. 585 19–24