Indian Journal of Engineering & Materials Sciences
Vol. 27, June 2020, pp. 573-578

Microstructure and mechanical properties of directionally solidified alloys of Mg-9Zn-2Y

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Received:16 February 2016 ; Accepted:02 April 2018

Samples of directionally solidified Mg-9Zn-2Y alloys have been obtained using vacuum induced directional solidification equipment. The metallographic structures and mechanical properties at room and high temperatures of the directionally solidified alloys have been investigated and compared with those of conventional casting alloys. The structures have consisted of columnar crystals with a specific orientation, and the parallel phases near the grain boundaries and inside the grains have quasi-crystals of I-Mg_3Zn_6Y. At room temperature, the maximum compressive resistance and section expansion ratio of the directionally solidified alloys have been 54 and 31% higher, respectively, than those of the conventional casting alloys. The peak stress of the directionally solidified alloys has found 238 MPa, which is 57% higher than that of the conventional casting alloys (152 MPa) at 200 °C with a strain rate of 0.1 s^{-1}. In addition, the peak stress was 140 MPa at 300 °C with a strain rate of 0.1s^{-1}, while it was only 84 MPa for the conventional casting alloys. Therefore, the directionally solidified alloys has higher strength and plasticity at room temperature and improved mechanical properties at a high temperature.

Keywords: Directional solidification, Mg-Zn-Y alloys, Columnar crystals, Mechanical properties

1 Introduction

Numerous studies have evaluated magnesium alloys owing to its light structure. Studies have evaluated improving the mechanical properties of magnesium alloys at a high temperature. Various studies have shown that it is effective to restrict the dislocation motion and strengthen the grain boundaries[1,2].

Recently, there has been great interest in Mg–Zn–Y system alloys containing an icosahedral quasicrystal phase (I-phase) owing to their increased strength and ductility[3-6]. The quasi-crystal I-Mg_3Zn_6Y in Mg-Zn-Y alloys, similar to that discovered in as-cast MgZnRE alloys by Luo and others in 1993, is a very efficient strengthened phase with a high melting point. The icosahedral quasicrystal phase is more stable than the crystalline phase and has a strong interface relationship with the matrix phase. Furthermore, because of its pinning force on the grain boundaries and distortion motion, the magnesium alloy properties at high temperatures can be improved[7,8]. However, as reported[9,10], conventional casting methods often bring coarse and eutectic quasi-crystals with a network distribution that cannot reinforce the mechanical properties of the alloys efficiently.

The columnar crystals with a specific orientation, obtained by directional solidification, delays the formation of cracks and increases the creep life. Thus, it improves the properties at a high temperature[11,12]. Although, Zou and others have found that AZ31 magnesium alloys with columnar crystals, obtained by directional solidification, have a high strength and plasticity at room and high temperatures[13]. Zhao and others have optimized the process of directional solidification AZ91 magnesium alloys[14]. Minimal research has been performed on the directional solidification of Mg-Zn-Y alloys. The purpose of this study was to experimentally investigate the mechanical properties of directionally solidified Mg-9Zn-2Y alloys at a high temperature.

2 Experimental Procedure

The significant raw materials of Mg-Zn-Y alloys were magnesium billets, zinc billets, and Mg-30Y (30wt%) master alloys. A 1-kg directional solidification vacuum melting furnace was employed, and the melting temperature was set to 760 °C. The melting liquid was poured into a graphite cavity. The bottom of the cavity was equipped with a chilling unit draw bar cooled by circulating water. The drawing speed was 10 mm/min. Then, a bar of a directionally solidified sample was obtained. The diameter of the bar was 20 mm, and the height was 100 mm. A vacuum resistance furnace was applied to obtain a conventional casting bar. The melting temperature was also set to 760 °C. Then, the molten alloy was poured into the preheated metal mold. The

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*Corresponding author (E-mail: lxping3588@163.com) conventional casting alloys and the directionally solidified alloys were both cooled by circulating water. The temperature of circulating water was set to 5 °C. The ICAP6300 phase spectrum instrument was used to analyze the final chemical composition (wt %) of the directionally solidified and conventional casting alloys. The chemical composition of the directionally solidified alloys was 9.05 wt% of Zn and 2.20 wt% of Y, and the balance was magnesium. The chemical composition of the conventional casting alloys was 8.75 wt% of Zn and 2.35 wt% of Y, and the balance was magnesium. A spark cutting machine was used to cut the sample from the bottom to obtain samples with a diameter of 10 mm and a height of 15 mm. The compression experiment at room temperature was conducted on a WDW3100-type microcomputer control electron universal testing machine with a strain rate of 0.2 s^{-1}. The mechanical properties at high temperatures were obtained on a Gleeble-3500D thermal simulated test machine at 200, 250 and 300 °C with strain rates of 1.0 to 1×10^{-3} s^{-1}. During the compression process, argon protection was used to prevent oxidation of the sample. The compression direction was parallel to the growth direction of the columnar crystals. Three samples were taken for each compression experiment. The experimental results were averaged to increase the experimental reliability. The microstructures of the experimental alloys were observed and analyzed by an Scanning electron microscopy (LEO JSM 5400). X-ray diffraction (A 2500/PC) with a scanning step of 0.3° was used to analyze the phase constituent. The scanning range was 20° to 90°(2θ).

3 Results and Discussion

3.1 Structures of the Directionally Solidified Alloys

As shown in Fig 1a, after directional solidification, columnar crystals were obtained with a parallel

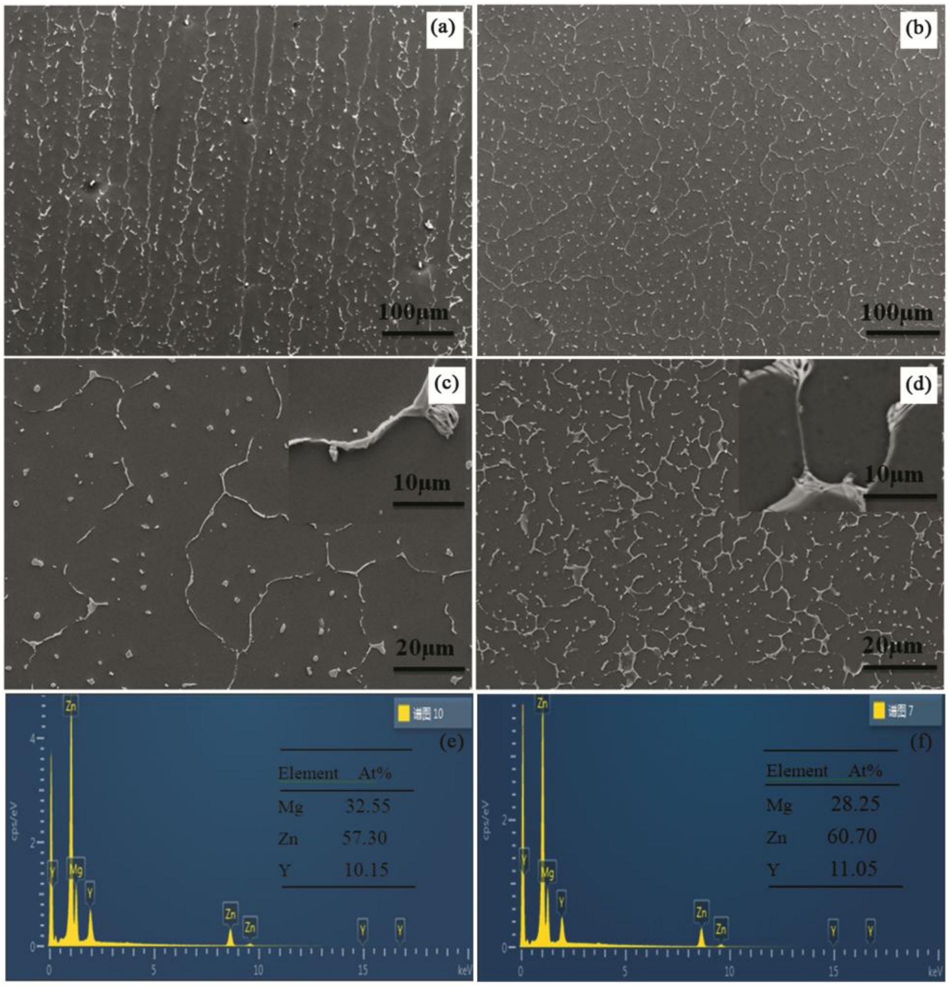

Fig. 1 — The solidified structure and morphology of Mg-Zn-Y alloy through scanning electron microscope (a)-(b) Longitudinal section and lateral section of directionally solidified alloys, (c) Lateral section of directionally solidified alloys, (d) Conventional casting alloy and (e)-(f) The energy spectrum of grain boundary phases and granular phases in the structure of directionally solidified alloys.

orientation. The vertical phase boundaries were straight, and the lateral boundaries were not present. In addition, the length was 35 mm. Fig. 1 b provided an intersecting surface counterpart of the microstructure of the lateral surface shown in Fig.1a. The grains of the lateral surface of the columnar crystals were cystiform, and the grainy phases were dispersed inside the grains granularity. Moreover, the directionally solidified alloys were compared to the conventional casting alloys (Fig 1d) using the same magnification. The directionally solidified alloys contained an improved average grain size, a smaller phase area, and a smaller grain (Fig 1c). The laminar eutectic structure in conventional casting alloys (Fig 1d) was different from the divorced eutectic structure of the secondary phases. There were more I phases in conventional cast alloys; however, many were continuously distributed between the dendrites in the form of lamellar eutectic. The atomistic percentage ratio between the phases near the grain boundaries and inside the grains provided by the EDS measurement were 32.55% for Mg, 57.30% for Zn, and 10.15% for Y and 28.25% for Mg, 60.70% for Zn, and 11.05% for Y, respectively. This indicated that they were both icosahedral quasi-crystals whose stoichiometric proportion was Mg:Zn:Y≈3:6:1.

The XRD patterns (Fig. 2) of both alloys showed two points. First, the conventional casting and directionally solidified alloys were composed of Mg and quasi-crystal I-Mg_3Zn_6Y phases. Second, the diffraction peak of the conical surface (102) of the crystal was stronger than the cylindrical plane (100), while the diffraction peak of the close-packed plane (002) of the crystal was weaker.

There were multiple reasons to support this growth mechanism. During conventional solidification, atoms were more likely to accrete along the close-packed planes where the surface density was higher. Therefore, more latent heat from crystallization was released. To curtail the process of directional solidification, manipulation of the temperature gradients was necessary to ensure that the crystals could grow along the direction of the rising temperature. There were two practical methods to achieve such goals. First, the liquid region temperature always stays Ts (Solidus temperature) around; therefore, the thermal conductivity of the close-packed planes disappears near the melting point. Second, the cold tip temperature of the solid region was maintained at 20 °C, forcing the solid-liquid section of the solid-liquid phase region to develop to the liquid-solid section when the draw bar drew graphite out of the homothermal region. Consequently, the nucleation rate increased rapidly, and a large sum of liquid solidified and released latent heat from crystallization, preventing atoms from growing from the close-packed planes. This caused the latent heat of crystallization to lower and provided the possibility for growth of the close-packed planes.

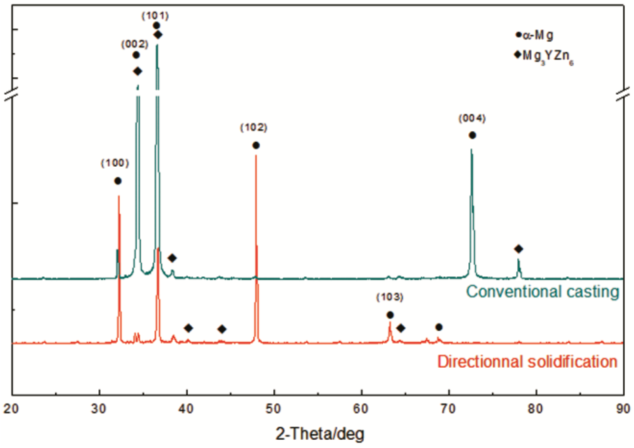

Fig. 2 — XRD of the two alloys.

3.2 Mechanical Properties of the Directionally Solidified Alloys

3.2.1 Mechanical properties at room temperature

The compression stress-strain curves of the directionally solidified samples (the compression direction was vertical to the columnar grains) and the conventional casting alloys (at 20 °C) are shown in Fig. 3. The maximum fracture resistance of the alloy specimen prepared by directional solidification was 357 MPa, which was approximately 54% higher than that of the conventional cast alloy (232 MPa). The

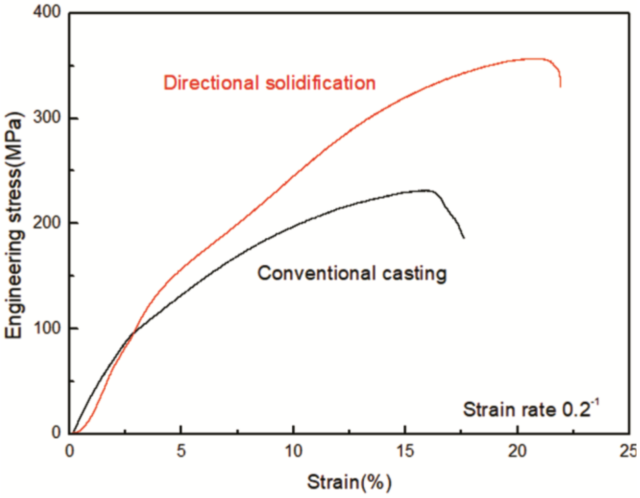

Fig. 3 — Engineering stress-strain curves of tested alloys under compression at 20 °C.

cross-sectional expansion rate was 21%. The cross-sectional expansion rate was 31% higher than that of the conventional casting alloy (16%). The improved performance was from the intricate microstructures and the following observations. First, the columnar crystals of the directionally solidified alloys are orientated in line with the direction of the stress. Most phase boundaries are parallel to the direction of the stress, and few are lateral. These microstructures could resist plastic deformation and compressive strength. Second, the mechanical properties of reticular quasi-crystals are inadequate in the conventional casting alloys.

Decreasing the grain boundaries of the quasi-crystals increased the granular quasi-crystal phase. Third, the lateral boundaries and the boundary area of the columnar crystals were less than those of the conventional casting alloys, and the resistance to the movement of the dislocations along the columnar crystal direction was less than those of the conventional casting alloys. Therefore, makes it more difficult for the accumulation of the strain in high stress concentration formed by the piling up of the dislocations to reach the nucleation of crack and to fracture, thus the plasticity is higher. Fourth, the XRD results showed that the external stress was approximately 45 ° in the normal direction of the base surface (0002) when the compression direction was parallel to the growth direction of the columnar crystals during the compression experiment. For the directionally solidified alloy cone (1012) under an external force, the orientation factor of the base slip system was higher. The base slip was the easiest to start, and the plasticity was improved. Finally, the directionally solidified alloys were more compact and contained less microdefects.

3.2.2 Properties under heat compression

Directionally solidified alloys are stronger at high temperature than conventional casting alloys, as shown in Fig. 4. In Fig 4a, the peak stress of the directionally solidified sample reached 238 MP at 200 °C with a strain rate of 0.1 s^{-1}. This was 57% higher than that of the conventional casting sample (152

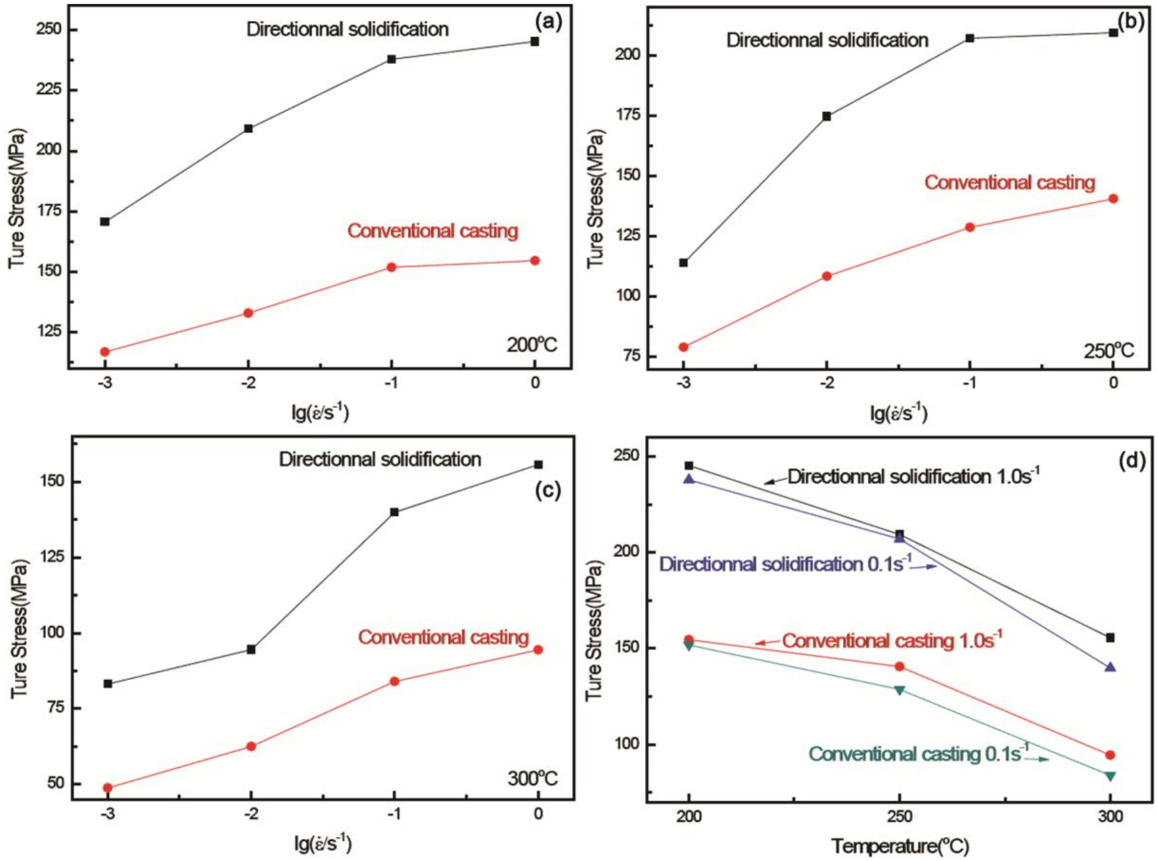

Fig. 4 — Curves of peck strain changes of tested alloys (a-c) Figures of peck stress with strain rate variation and (d) The figure of peck stress with temperature variation.

MPa). When the temperature was constant, the peak stress decreased as the strain rate decreased. When the strain rate was 0.001 s^{-1}, the peak stress of the directionally solidified alloy was 171 MPa, which was approximately 46% higher than that of the conventional casting sample (117 MPa). The peak stress decreased with an increasing temperature. When the temperature was 250 °C, and the strain rate was 0.1 s^{-1}, the peak stress of the directionally solidified alloy was 207 MPa. This was a 60% higher than that of the conventional casting sample (129 MPa). When the temperature was 250 °C and the strain rate was 0.001s^{-1}, the peak stress of the directional solidification alloy was 114 MPa. This was 44% higher than that of the conventional casting sample (79 MPa) (Fig.4b). The peak stress of the directional solidification alloy was 140 MPa, while that of the conventional casting was 84 MPa at 300 °C with a strain rate of 0.1 s^{-1} (Fig. 4c).

The reasons for the superior performance of the directionally solidified alloys are described below.

First, for the composition, the directionally solidified Mg-Zn-Y alloys used solid solution α-Mg as a matrix, forming the columnar crystal with a specific orientation. The grain boundary was quasi-crystal I-Mg_3Zn_6Y with granular quasi-crystals dispersed in the columnar crystal clearance. The inter crystalline second phases typically form networks of grain boundaries that are perpendicular with the principal axes of the stress in the polycrystalline conventional casting alloys, thus causing cracks under high-temperature stress. At a high temperature, the grain boundaries of the columnar crystals of directional solidification will rearrange their direction parallel to the principal axis of stress minimizing the stress on the weak grain boundaries, thus delaying the formation of cracks and improving the high-temperature strength.

Second, according to the basic theory of motion of dislocation, the thermal deformation of metals is caused by a dislocation motion. Dislocations are blocked during motion, and a high-density region is formed. Dynamic recrystallization starts to nucleate when the dislocation density reaches a critical point[15]. The Mg-Zn-Y alloys with columnar crystals contain less horizontal grain boundaries, less grain boundary area, and a smaller resistance parallel to the columnar crystals than the conventional casting Mg-Zn-Y alloys. Therefore, its dislocation mobility is improved, and accumulation is difficult[16]. Thus, the dislocation density has a lower possibility of reaching the critical point of nucleation of dynamic recrystallization.

Furthermore, because of the small misorientations in the Mg-Zn-Y alloys with a continuous columnar crystal, the parallel structures must become deformation bands when compressed to increase the misorientations of the local area; therefore, large angles can form and provide the appearance of dynamic recrystallization with the necessary conditions. The grain boundary areas of the original structures of conventional casting Mg-Zn-Y alloys are large, and the misorientations between the grains are large. Therefore, high-density dislocation regions and large-angle grain boundaries are formed. Consequently, Mg-Zn-Y alloys with continuous columnar crystals need more energy for dynamic recrystallization than conventional casting Mg-Zn-Y alloys; therefore, dynamic recrystallization is difficult, and softening of the dynamic recrystallization is reduced[17]. Figure 5 is the optical images of the alloys with different solidification modes were obtained at 250 °C and the strain rate was 0.1 s^{-1}. The micrograph

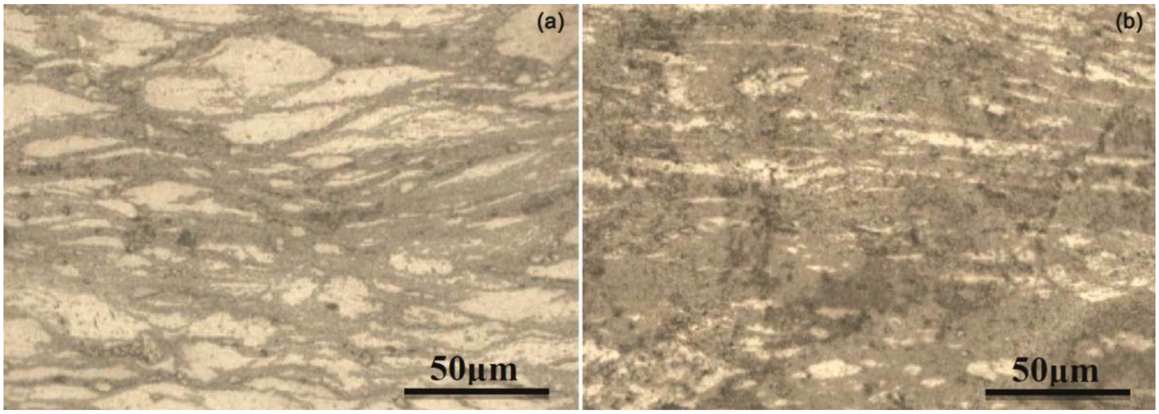

Fig. 5 — Optical images of the samples with different solidification modes at 250 °C with a strain rate of 0.1 s^{-1} (a) Directional solidification and (b) Conventional casting.

shows that the dynamic recrystallization in directionally solidified alloy is much less than that in conventional cast alloy.

Finally, the second phase in the grains is the granular quasi-crystal I phase dispersed in the matrix, which contain good stability to hinder the movement of dislocation and to guarantee the strength at a high temperature.

4 Conclusions

The microstructure and mechanical properties of directionally solidified alloys of Mg-9Zn-2Y were discussed. The following conclusions were drawn:

(i) A specific orientation of columnar crystals was observed in directionally solidified alloys. The grainy phases, icosahedral quasi-crystals of I-Mg_3Zn_6Y, were dispersed inside the grains.

(ii) After directional solidification, the Mg-9Zn-2Y alloy showed a higher maximum breaking resistance and section expansion ratio than conventional casting alloys at room temperature.

(iii) The directionally solidified alloys showed higher mechanical properties at a high temperature than conventional casting alloys. The peak stress of the directionally solidified alloys reached 238 MP at 200 °C with the strain rate of 0.1 s^{-1}.

Acknowledgements

This study was supported by National Natural Science Foundation of China (No. 51775099) and Natural Science Foundation of Hebei Province (E2018501032 and E2018501033).

References

1 Peng Z K & Zhang X M, *T Nonferr Metal Soc Chin*, 15 (2005) 917.
2 Fu D F, Zhang H, Xia W J & Huang C Q, *Light Alloy Fabrication Technol (Chin)*, 3 (2004) 41.
3 Luo Z P, Zhang S Q, Wei G, Lu L Q, Tang Y L & Zhao D S, *Acta Aeronautica et Astronautica Sinica*, 7 (1994) 860.
4 Singh A, Watanabe M, Kato A & Tsai A P, *Mater Sci Eng A*, 397 (2005) 22.
5 Wan D Q & Yang G C, *J Mater Sci Technol*, 24 (2008) 317.
6 Chen Q, Lin J, Shu D Y, Hu C K, Zhao Z D, Kang F, Huang S H & Yuan B G, *Mater Sci Eng A*, 554 (2012) 129.
7 Singh A & Tsai A, *Scripta Mater*, 53 (2005) 1083.
8 Ma R, Dong X P, Chen S, Pan Z, Ling H & Fan Z, *Mater Sci Eng A*, 587 (2013) 328.
9 Sun L, Fan Z B, Lin X P, Ye J, Guan C & Yang H G, *Chin Foundry*, 63 (2014) 1153.
10 Singh A, Somekawa H & Mukai T, *Mater Sci Eng A*, 528 (2011) 6647.
11 Ver Snyder F L & Shank M E, *Mater Sci Eng A*, 6 (1970) 2l3.
12 Xu C, Zhou L Z, Guo J T & Yang G X, *T Nonferr Metal Soc Chin*, 21 (2011) 754.
13 Zou M Q, Huang C Q, Xia W J, Chen Z H & Liao K, *Chin Foundry*, 9 (2006) 890.
14 Zhao Y M, Zhang J Q, Xiang W & Guo G L, *Hot Work Technol (Chin)*, 13 (2012) 75.
15 Xu S W, Zheng M Y, Kamado S, Wu K, Wang G J & Lv XY, *Mater Sci Eng A*, 528 (2011) 4055.
16 Chen X H, Liu L Z, Liu J & Xu D K, *Mater Sci Eng A*, 443 (2007) 248.
17 Yu J W, Liu X F & Xie J X, *T Nonferr Metal Soc Chin*, 4 (2011) 482.