Microstructure and mechanical properties of NZ30K alloy by semi-continuous direct chill and sand mould casting processes

Zheng Xingwei [1], *Dong Jie [1], Liu Wencai [1] and Ding Wenjiang [1, 2]

(1. National Engineering Research Centre of Light Alloy Net Forming, Shanghai Jiao Tong University; 2. Key State Laboratory of Metal Matrix Composite, Shanghai Jiao Tong University, Shanghai 200240, China)

Abstract: The Mg-3.0Nd-0.2Zn-0.4Zr (NZ30K) alloys were prepared by direct-chill casting (DCC) and sand mould casting (SMC) processes, respectively and their microstructures and mechanical properties were investigated. The results indicate that casting method plays a remarkable influence on the microstructure and mechanical properties of as-cast NZ30K alloy. The grain size increases from 35–40 μm in the billets made by the DCC to about 100–120 μm in the billets by the SMC. The aggregation of $Mg_{12}Nd$ usually found at the triple joints of grain boundaries in the billets prepared by SMC while is not observable from the billets by DCC. The tensile strengths and elongations of the billets are 195.2 MPa and 15.5% by DCC, and 162.5 MPa and 3.2% by SMC, respectively. The tensile strength of the alloy by DCC is remarkably enhanced by T6 heat treatment, which reached 308.5 MPa. Fracture surfaces of NZ30K alloy have been characterized as intergranular fracture by SMC and quasi-cleavage fracture by DCC, respectively.

Key words: NZ30K alloy; direct-chill casting; microstructure; mechanical properties
CLC number: TG146.2+2 Document code: A Article ID: 1672-6421(2011)01-041-06

Magnesium alloys are the lightest structural alloys and have attracted increasing attentions in recent years. The applications of the magnesium alloys, especially for the cast and wrought products, are rapidly increasing due to their excellent properties such as high specific strength and low density [1]. Some magnesium alloys, such as AZ91 and AM50, have already been used for automotive components, however the application of these alloys is limited in some areas (such as instrument panel beams, valve covers etc.) due to the restriction of strength and creep resistance at elevated temperature.

It was reported that the addition of rare earth (RE) elements can improve the mechanical properties of the magnesium at both room and elevated temperatures [2–3]. The solubility of most RE in the magnesium matrix is relatively high and it decreases with the temperature drop, therefore the additions of RE to magnesium alloys influence both solid solution and precipitations hardening, leading to strengthening of the alloys at both ambient and elevated temperature [4–5]. Most recently, a lot of works have been focused on magnesium alloys containing heavy rare earth elements, such as Mg-Gd-Y-Zr [6–7], Mg-Dy-Gd-Nd [8] and Mg-Gd-Nd-Zr [9]. However high content of heavy rare earth elements increases the alloy cost, which could limit their engineering application.

Therefore, a new magnesium alloy containing only a small amount of light rare earth was developed in our institution, i.e., NZ30K (Mg-3.0Nd-0.2Zn-0.4Zr). Elongations of the NZ30K alloy prepared by metallic mould under the conditions of as-cast, T4 and T6 are 14%, 22% and 11%, respectively [10]. It seems that NZ30K alloy is not only capable of a potential casting alloy but also a potential wrought alloy. Microstructure, mechanical properties and corrosion behavior of the NZ30K alloy prepared by metallic mould casting had been investigated [11–12] in previous study. In order to promote the application of the new developed forgeable and heat resistant alloy, the microstructure and mechanical properties of the NZ30K alloy prepared by sand mould casting (SMC) and direct-chill casting (DCC) were investigated in the present study. The current results are also compared with previous ones of the same alloy prepared by metallic mould [10].

***Dong Jie**

Male, born in 1974, Ph.D. Associate professor. He got his doctor's degree from the Northeastern University in 2004. His research interests mainly focuse on the direct chill casting of magnesium alloys and their plastic forming. His more than 40 papers have seen published by now and he also possesses 5 patents of China.
E-mail: jiedong@sjtu.edu.cn
Received: 2010-04-26; Accepted: 2010-07-01

1 Experimental procedure

The experimental apparatus for the DCC is shown in Fig.1. The crystallizer used in the present study is made of Al alloy with a graphite ring insert in it. The inner diameter, outer diameter and the height of the graphite ring are 106 mm,

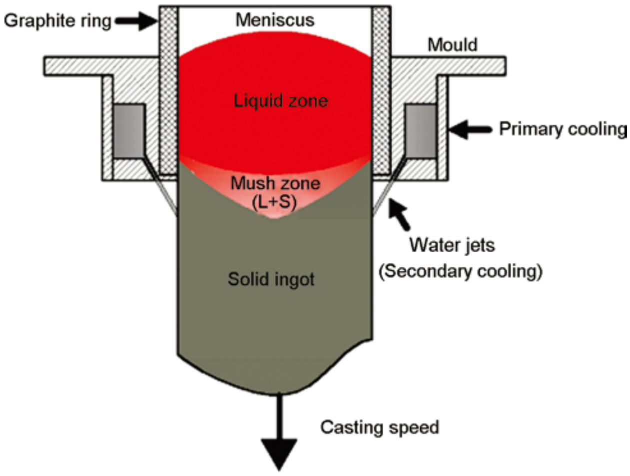

Fig. 1: Schematic of the DCC device

114 mm and 120 mm, respectively. The alloy of nominal composition Mg-3.0Nd-0.4Zn-0.2Zr (wt.%) was prepared by pure Zn, Mg and Mg-30%Nd, Mg-30%Zr master alloys (wt.%), and melted in an electrical resistance furnace under the protection of mixture gas of SF_6, CO_2 and air in this research. The melt was degassed, slag removed and refined at 740 ℃, then two types of casting processes were performed. First, for the billet cast by DCC, the casting speed, melt temperature and the water flow rate applied are 100 mm/min, 700 ℃ and 25 L/min, respectively. Second, for the billet with the diameter of 100 mm by SMC, the sand mould was preheated to 200 ℃ at first, then the billet was poured, and cooled in the mould.

The microstructures of the specimens cross-sectioned from the billets prepared by the different casting methods were examined under an optical microscope (OM). All the specimens for the microstructure observation were etched with a phosphoric-picral solution (0.7 mL phosphoric acid, 4.2 g picric acid and 100 mL ethanol). The average grain size was measured by the linear intercept method.

The specimens from the cast billets were firstly solution treated at 540 ℃ for 4 h and quenched into hot water at ~70 ℃, then aged at 200 ℃ in an oil-bath. Vickers hardness (HV) testing was measured with 5 kg load and holding time of 30 s. The samples for tensile testing were cut into rectangular tensile specimens with dimension of 10 mm width, 2 mm thickness and 30 mm gauge length by an electric-sparking wire cutting machine and all the test samples were taken from the middle section of the billet along the billet axis. The tensile tests were carried out on a Zwick/Roell-20KN material machine at a cross-head speed of 1 mm/min at room temperature.

2 Experiment and discussion

2.1 Microstructure

The microstructures from the NZ30K billets prepared by DCC and SMC are shown in Fig.2. Microstructures in the specimens from all of the billets, also incl., those either from the edges or from the centre of the billet, are identified by the presence of equiaxed grains. Furthermore, some compounds are observed along the grain boundary. The X-ray diffraction (XRD) analysis results of the alloys are shown in Fig.3. The results demonstrate that the billets, prepared by DCC and SMC, are mainly composed of α-Mg and $Mg_{12}Nd$. However the amount of the $Mg_{12}Nd$ in the billet prepared by SMC is more than that from the billet made by DCC. The same second phase was reported in other Mg-Nd based alloys [10, 13].

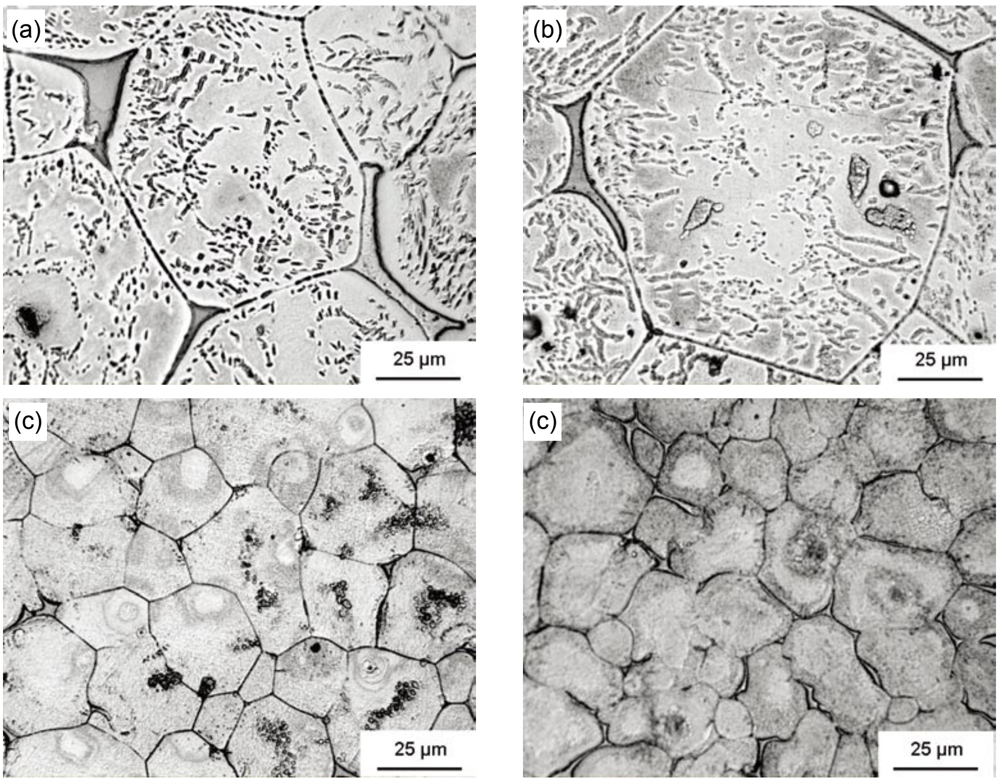

Fig. 2: Microstructures of the billets cast by SMC and DCC, respectively: SMC edge (a), SMC centre (b), DCC edge (c), DCC centre (d)

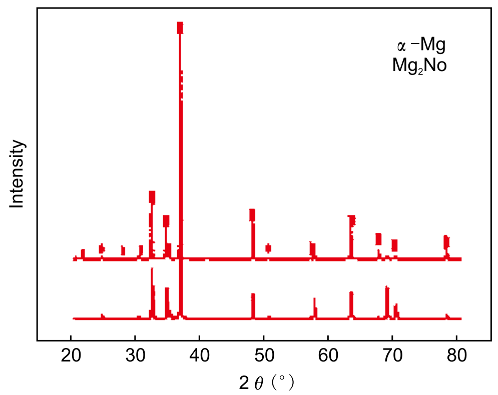

Fig. 3: The XRD analysis results of the billets prepared by DCC and SMC

The average grain sizes along the diameter of the billets prepared by DCC and SMC are shown in Fig.4. It is found that the grain sizes of the billets, both from the DCC and SMC, tend to increase from the edge region to the central region of the billet. However the grain sizes in the SMC billets, both at the centre and at the edge, are larger than the ones in the DCC billets. The reason can be attributed to the higher cooling rate of the DCC than that of SMC.

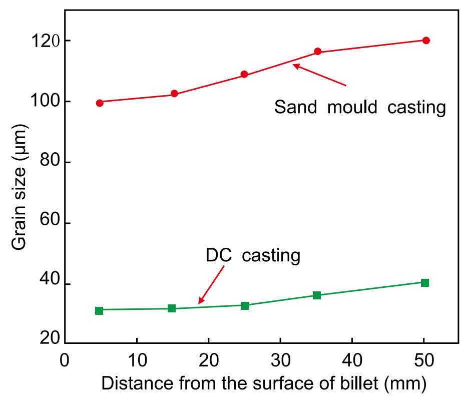

Fig. 4: The grain size along the radius of the billet

2.2 Aging characteristics

Figure 5 shows the aging hardening curves of the alloy at 200 ℃. It can be seen that the alloys prepared by both DCC and SMC exhibit strong aging hardening response. Consequently, the HV increases with aging time after an incubation period until it reaches the peak hardness (PHV) and then decreases with the aging time as an overaged result. The peak aging time and PHV value are about 15 h and 73 for the NZ30K alloys made by DCC, and 12 h and 68 for the alloys by SMC, respectively. According to the previous report [10] on the NZ30K alloy made by metallic mould casting method, the current results from the DCC billet have the similar peak aging hardness at about 73, which is higher than that from SMC method. The reason may be attributed to the similar grain sizes of NZ30K alloys achieved by both metallic mould and DCC methods, which are much finer than those made by SMC.

The solution treated and T6 treated microstructures (at peak aging state) of the NZ30K alloys prepared by SMC and DCC are shown in Fig. 6. It can be seen that most of $Mg_{12}Nd$ in the alloy prepared by DCC has been dissolved after 4 h solution treatment at the 540 ℃, however it takes 8 h to dissolve the $Mg_{12}Nd$ in the alloy prepared by SMC. And some Zr-containing particles and the precipitates appear in the T6 microstructure according to reference [10]. The precipitates are β'' phase with DO19 structure and which may have two kinds of habit planes {1100} and {1120} according to reference [10]. The average grain sizes of the solution treated alloy prepared by SMC and DCC are about 100–120 μm and 30–40 μm, respectively. Unlike Mg-Gd-Y-Zr [6] and Mg-Y-Sm-Zr alloys [14], the grain of NZ30K alloy nearly does not coarsen during solution treatment. The same phenomenon is observed in the NZ30K alloy prepared by metallic mould casting. The reason may be related to the addition of Zr and Nd which improves the heat resistance of the alloy.

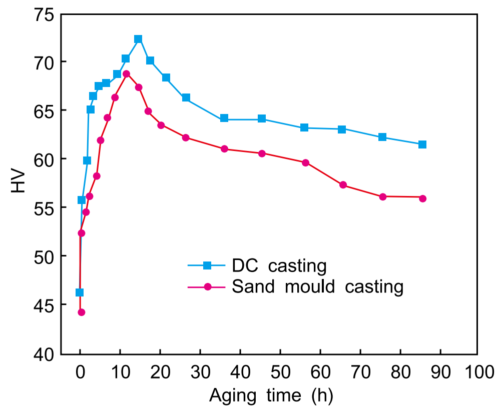

Fig. 5: The aging hardness curve of the NZ30K alloy prepared by DCC and SMC

2.3 Mechanical properties

The typical stress and strain curves, both the as-cast and T6 states, of the NZ30K prepared by DCC and SMC, respectively are shown in Fig.7. The curves indicate that mechanical properties, especially the elongation, of the NZ30K alloy are sensitive to the preparing method. It can be seen that the mechanical properties are remarkably enhanced by T6 treatment.The mechanical properties of the as-cast and T6 treated alloys prepared by DCC and SMC are listed in Table 1, and for comparison, the properties of the alloy prepared by the metallic mould are also attached. It can be seen that the as-cast NZ30K alloy prepared by DCC exhibits the highest ultimate tensile strength (UTS) and the yield strength (YS), and the corresponding values are 195.2 and 110.3 MPa, respectively. The results show that the elongation of NZ30K is very sensitive to preparing method, which drops from 15.5% of the DCC to only 3.2% of the SMC. However the NZ30K alloys prepared by DCC and metallic mould have similar elongation which is about 14%. The UTS and YS of the NZ30K alloy prepared by the DCC are enhanced by T6 treatment, but as to the elongation, there is only insignificant difference between the two casting methods.

It reveals that the preparing method of DCC could improve the mechanical properties of the as-cast NZ30K alloy. The following factors are mainly related to the improvement of mechanical properties. Firstly, the grain size of the as-cast

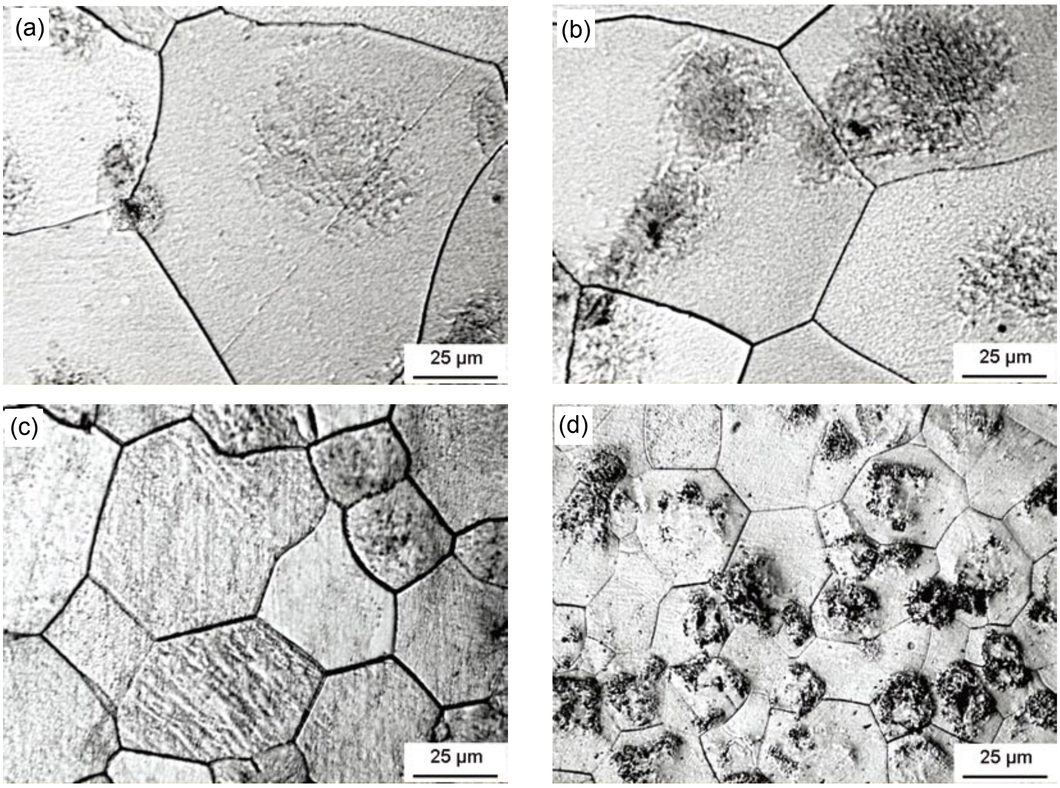

Fig. 6: The solution treated and T6 treated microstructures: SMC + 8 hours solution (a), SMC +T6 treatment (b), DCC + 4 hours solution (c), DCC +T6 treatment (d)

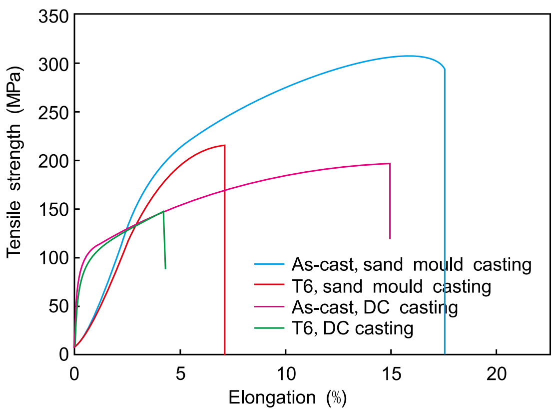

Fig. 7: The typical stress and strain curves of the NZ30K alloys prepared by various methods

Table 1: The as-cast and peak aging mechanical properties of the NZ30K prepared by DCC, SMC and metallic mould casting [10]

NZK30		R_m (MPa)	$R_{p0.2}$ (MPa)	A (%)
DCC	As-cast	195.2	110.3	15.5
	T6	308.5	160.7	14.2
SMC	As-cast	162.5	82.3	3.2
	T6	220.2	165.5	4.6
Metallic mould	As-cast	175.0	86.3	13.6
	T6	312.5	138.8	11.7

NZ30K alloy has reduced from about 100–120 μm of SMC to about 35–40 μm of DCC (as shown in Fig.2) and as a reference, the grain size of NZ30K alloy prepared by metallic mould is about 50 μm [10]. Secondly, the amount of $Mg_{12}Nd$ in the alloy prepared by SMC is much more than that in the alloys prepared by DCC or metallic mould casting. Furthermore, most of the phases are segregated in the triple joints of grain boundaries. This will lead to the dislocation pile up and stress concentration during the tensile deformation. On the other hand, the crystal phase of $Mg_{12}Nd$ has tetragonal structure (a=b=10.31, c=5.93), while the crystal structure of α-Mg is close-packed hexagonal structure (a=b=3.209, c=5.211) [15]. Therefore incoherent phase boundary will be formed between $Mg_{12}Nd$ and α-Mg according to the definition of lattice misfit, which will lead to the brittleness of grain boundary. This explains why the elongation of the NZ30K alloy is reduced from about 14% of DCC and metallic mould casting to 3.2% of SMC. Consequently it is reasonable to confirm that the DCC plays a positive role in the improved mechanical properties of the NZ30K alloy comparing with the metallic mould casting and SMC.

Heat treatment enhances the mechanical properties of NZ30K alloy prepared by DCC, especially the UTS of the peak aged alloy, increased by more than 100 MPa.

In present work, the $Mg_{12}Nd$ is the exclusive strengthening precipitate. The $Mg_{12}Nd$ precipitates in the grain after T6 heat treatment are as shown in Fig.6. The existence of the $Mg_{12}Nd$ can effectively restrain the gliding of the dislocation at room temperature. Furthermore there is no precipitation and segregation along the grain boundary after T6 heat treatment, which will release the stress concentration during the tensile deformation. On the other hand, the grains are nearly not coarsened during T6 treatment. Consequently the UTS and YS of the NZ30K alloy increase from 195.2 and 110.3 MPa at the as-cast state to 308.5 and 160.7 MPa at T6 state, respectively. And there is no obvious reduction of the elongation by T6 heat treatment.

2.4 Fracture mechanism

Figure 8 shows the as-cast microstructures near tensile fracture of the specimens tested at room temperature. It can be seen that cracks are all initialized from the triple joints boundary. However the cracks of the NZ30K alloy prepared by DCC run through the grain while the cracks of NZ30K alloy prepared by SMC mainly propagate along the grain boundary. The fracture surfaces of the billets prepared by DCC and SMC are shown in Fig. 9, both lacerated ridges and cleavage steps are observed on the fracture surface of the alloy prepared by DCC. Consequently, the fracture surface of the NZ30K alloy by DCC is observed to be typical quasi-cleavage fracture in nature. On the contrary, only a small amount of cleavage steps are observed in the alloy prepared by SMC, and most of the grain still keep the original morphology after fractured. Therefore the fracture surface of the NZ30K alloy prepared by SMC belongs to intergranular fracture.

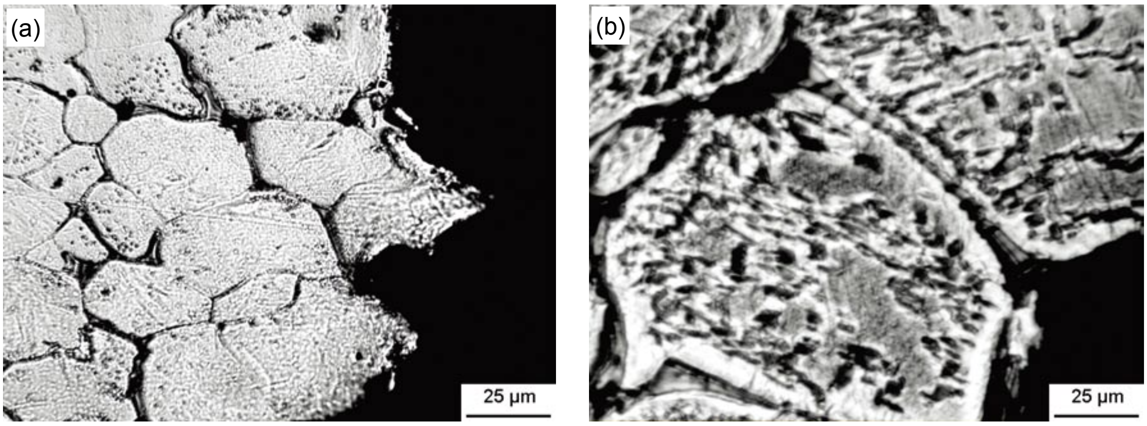

Fig. 8: Optical microstructure of near fractured tensile specimens at room temperature: DCC (a), SMC (b)

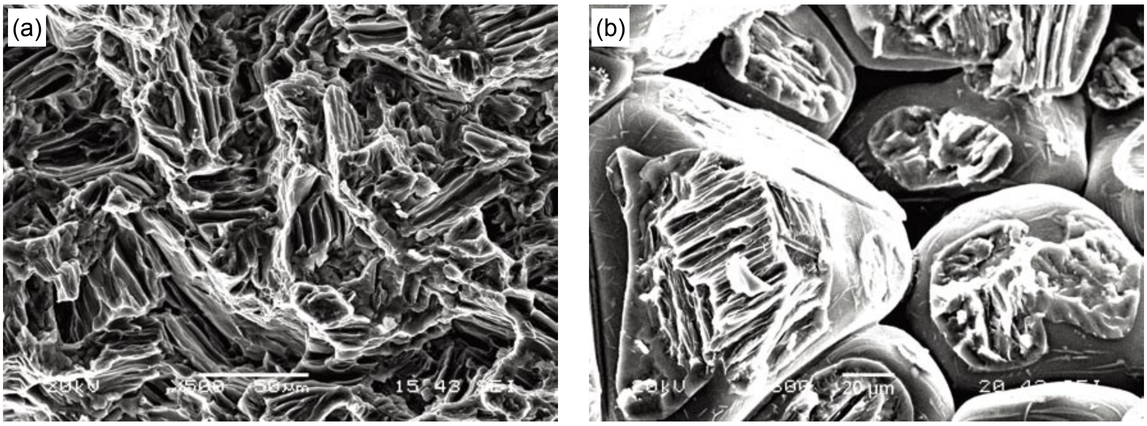

Fig. 9: SEM images of the fracture surfaces from tensile specimens cast by two different methods at room temperature: DCC (a), SMC (b)

4 Conclusions

(1) Both the microstructures of the NZ30K alloy cast by DCC and SMC, respectively consist of equiaxed grains and eutectic compounds $Mg_{12}Nd$, and the grain size of the NZ30K alloy is sensitive to casting method.

(2) The mechanical properties of the as-cast NZ30K are remarkably enhanced by the DCC comparing with those from SMC, especially the elongation increased from 3.2% to about 14%. The tensile strength and the Vickers hardness of the T6 treated NZ30K alloy cast by DCC are 308.5 MPa and 73, respectively.

(3) The fracture surface of the NZ30K alloy prepared by DCC has typical appearance of quasi-cleavage fracture while the alloy prepared by SMC has intergranular fracture in nature.

References

[1] Pekguleryuz M, Kaya A. Creep Resistant Magnesium Alloys for Powertrain Applications. Advance Engineering Material, 2003, 5(12), 866–878.

[2] Xie Jianchang, Li Quanan, Wang Xiaoqiang, Li Jianhong. Microstructure and mechanical properties of AZ81 magnesium alloy with Y and Nd elements. Transactions of Nonferrous

Metals Society of China, 2008, 18(2): 303–308.
[3] Liu Bin, Zhang Milin and Wu Ruizhi. Effects of Nd on microstructure and mechanical properties of as-cast LA141 alloys. Material Science and Engineering A, 2008, 487(35): 347–351.
[4] Suzuki M, Sato H, Maruyama K. Creep deformation behavior and dislocation substructures of Mg–Y binary alloys. Material Science and Engineering A, 2001, 319–321(58): 751–755.
[5] Suzuki M, Kimura T, Koike J. Effects of zinc on creep strength and deformation substructures in Mg–Y alloy. Material Science and Engineering A, 2004, 387–389(102): 706–709.
[6] He S M, Zeng X Q, Peng L M, Gao X, Nie J F, Ding W J. Precipitation in a Mg–10Gd–3Y–0.4Zr (wt.%) alloy during isothermal ageing at 250 ℃. Journal of Alloys Compounds, 2006, 421(37): 309–313.
[7] Peng Qiuming, Wang Jianli, Wu Yaoming and Wang Limin. Microstructures and tensile properties of Mg–8Gd–0.6Zr–*x*Nd–*y*Y ($x + y = 3$, mass%) alloys. Material Science and Engineering A, 2006, 433(32): 133–138.
[8] Li Dehui, Dong Jie, Zeng Xiaoqin, Lu Chen and Ding Wenjiang. Characterization of precipitate phases in a Mg–Dy–Gd–Nd alloy. Journal of Alloys and Compounds, 2006, 439(67): 254–257.
[9] Zheng K Y, Dong J, Zeng X Q and Ding W J. Effect of thermo-mechanical treatment on the microstructure and mechanical properties of a Mg–6Gd–2Nd–0.5Zr alloy. Material Science and Engineering A, 2007, 454–455: 314–321.
[10] Fu Penghuai, Peng Liming, Jiang Haiyan, Chang Jianwei and Zhai Chunquan. Effects of heat treatments on the microstructures and mechanical properties of Mg-3Nd-0.2Zn-0.4Zr (wt.%) alloy. Material Science and Engineering A, 2008, 486: 183–192.
[11] Chang J W, Peng L M, Guo X W, Atrens A, Fu P H, Ding W J and Wang X S. Comparision of the corrosion behavior in 5% NaCl solution of Mg alloys NZ30K and AZ91D. Journal of Apply Eletrochemcal, 2008, 38: 207–214.
[12] Chang Jianwei, Guo Xingwu, Fu Penghuai, Peng Liming, Ding WenJiang. Effect of heat treatment on corrosion and electrochemical behaviors of Mg-3Nd-0.2Zn-0.4Zr alloy. Electrochemic Acta, 2007, 52: 3160–3167.
[13] Yan Jingli, Sun Yangshan, Xue Feng, Xue Shan and Tao Weijian. Microstructure and mechanical properties in cast magnesium-neodymium binary alloys. Material Science and Engineering A, 2008, 476: 366–371.
[14] Li Daquan, Wang Qudong and Ding Wenjiang. Characterization of phases in Mg-4Y-4Sm-0.5Zr alloy processed by heat treatment. Material Science and Engineering A, 2006, 428: 295–300.
[15] Massalski T B (Ed.). Binary Alloy Phase Diagrams. ASM, Metals Park, OH, 1986.

The project was finacially supported by the National High-tech R&D Program of China (863 Program), grant No. 2009AA03Z521, and the foundation of Shanghai Rising-Star Program (A type), grant No. 09QA1403100.