

Available online at www.sciencedirect.com



Trans. Nonferrous Met. Soc. China 22(2012) 1891-1895

Transactions of Nonferrous Metals Society of China

www.tnmsc.cn

## Microstructure and mechanical properties of extruded Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr alloy

ZHOU Hao<sup>1</sup>, WANG Qu-dong<sup>1, 2</sup>, CHEN Jie<sup>1</sup>, YE Bing<sup>1, 2</sup>, GUO Wei<sup>1</sup>

1. National Engineering Research Center of Light Alloy Net Forming,

Shanghai Jiao Tong University, Shanghai 200240, China;

2. State Key Laboratory of Metal Matrix Composite, Shanghai Jiao Tong University, Shanghai 200240, China

Received 10 November 2011; accepted 31 May 2012

Abstract: The microstructure, age hardening behavior and mechanical properties of an Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr alloy prepared by casting and hot extrusion techniques were investigated. The solution-treated (T4 temper) alloys were extruded at 400, 450 and 500 °C with an extrusion ratio of 10:1, respectively. Optimized mechanical properties were obtained by extrusion at 400 °C followed by T5 treatment under the combined effects of grain refinement and precipitation strengthening. The alloy exhibits a grain size of about 5.0  $\mu$ m, initial and peak microhardness of HV 109 and HV 129, respectively. The tensile yield strength, ultimate tensile strength and elongation at room temperature are 391 MPa, 430 MPa and 5.2%, respectively.

Key words: Mg-Gd-Y-Ag-Zr alloy; extrusion; age hardening; microstructure; mechanical property

## **1** Introduction

High performance magnesium alloys containing yttrium or heavy rare earth elements are attractive for the application of light structural materials in the electronics, automobile and aerospace industries [1-3]. It has been reported that recently developed Mg-10Gd-5Y-0.5Mn and Mg-10Gd-3Y-0.4Zr alloys exhibit higher specific strength at both room and elevated temperatures and better creep resistance than conventional Al and Mg alloys including WE54 with highest strength at elevated temperature among existing commercial magnesium alloys [4, 5]. Multi-alloying has become an effective way to improve the mechanical properties of magnesium alloys [6,7]. It has been reported that the addition of Ag could considerably enhance the precipitation-hardening of Mg-Gd alloys [8]. WANG et al [9] reported a novel super high strength cast magnesium alloy of Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr allov can be significantly strengthened after cast-T6 comparing to Mg-10Gd-2Y-0.5Zr alloy [7].

In order to extend the production of magnesium

with fine grains and superior mechanical properties by thermomechanical processing such as extrusion and rolling other than conventional pressure die casting technique, it is important to optimize the processing parameters [10,11]. The processing of magnesium alloy with extrusion has both technical and economic advantages in the production of structure components [12]. The effect of thermal processing on Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr alloy is not clear yet. In the present study, this alloy is prepared by hot extrusion technique to eliminate the casting porosities. The microstructure, age hardening behavior and mechanical properties at different extrusion temperature are investigated.

## 2 Experimental

The alloy with composition of Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr (mass fraction, %) (as listed in Table 1) was determined by an inductively coupled plasma atomic emission spectroscopy (ICP-AES) analyzer (Perkin– Elmer, Plasma 400). Ingots were prepared from high purity Mg and Ag ( $\geq$ 99.95%) elements, Mg-30Zr and Mg-25RE (mass fraction, %) (RE=Y and Gd) master

Foundation item: Projects (50674067, 51074106) supported by the National Natural Science Foundation of China; Project (2009AA033501) supported by the High-Tech Research and Development Program of China; Project (09JC1408200) supported by the Science and Technology Commission of Shanghai Municipality, China

**Corresponding author:** WANG Qu-dong; Tel: +86-21-54742715; Fax: +86-21-34202794; E-mail: wangqudong@sjtu.edu.cn DOI: 10.1016/S1003-6326(11)61403-9

Table 1Composition of Mg-Gd-Y-Ag-Zr alloys (massfraction, %)

Gd	Y	Ag	Zr	Mg
8.5	2.3	2.0	0.4	Bal.

alloys in an electric-resistant furnace under a mixed atmosphere of CO<sub>2</sub> and SF<sub>6</sub> with volume ratio of 100:1. The ingots with a diameter of 50 mm were homogenized at 500 °C for 10 h followed by quenching into water at about 20 °C (T4 treatment). The ingots were further hot extruded into sheets with dimensions of 3 mm× 12 mm on the cross section with an extrusion ratio of 10:1 at extrusion temperatures of 400, 450 and 500 °C, respectively. Following extrusion, aging treatment was carried out at 200 °C in an oil-bath furnace (T5 treatment) for 0.5-300 h. The microstructures were characterized using an optical microscopy (OM) and scanning electron microscopy (SEM). Vickers microhardness testing was performed under 49 N loading for 15 s. Tensile testing was carried out on a Zwick-20 kN material test machine at room temperature at an initial strain rate of  $8.3 \times 10^{-4} \text{ s}^{-1}$ .

## **3** Results and discussion

#### **3.1 Microstructure**

The microstructure of the T4 solution treated ingot is shown in Fig. 1(a) with an averaged grain size of about 139  $\mu$ m. The precipitates completely disappear in the grain boundary although some cubic-shaped compounds are observed near the grain boundary.

The microstructures of Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr alloy after extrusion at 400, 450 and 500 °C are shown in Figs. 1(b)-(d). All the micrographs are taken along the longitudinal direction (the plane normal to the extrusion direction). In general, the grain size of as-extruded alloy is significantly refined compared to that of the as-cast alloy. The microstructure of as-extruded alloy is constituted by two different types of grains. One is the fine equiaxed grains with average grain size of a few microns. The grain boundaries between these grains can be clearly observed under the optical microscopy. The other is the ultrafine grains with grain size less than 1 µm. The grain boundaries between such grains could hardly be observed by the optical microscopy. Due to light scattering, these grains including grain boundaries appear as the black areas in the micrograph. The formation of fine equiaxed grains indicates that dynamic recrystallization has occurred during the hot extrusion process.

The mean grain size of the as-extruded alloy processed at 400, 450 and 500 °C estimated by linear section was 5.0, 5.3 and 5.9  $\mu$ m, respectively. A fine and homogeneous equiaxial recrystallization structure is formed by extrusion at 400 °C, as shown in Fig. 1(b). As the extrusion temperature increases, there is abnormal grain growth. The number of the larger grains increases significantly, and the microstructure becomes less

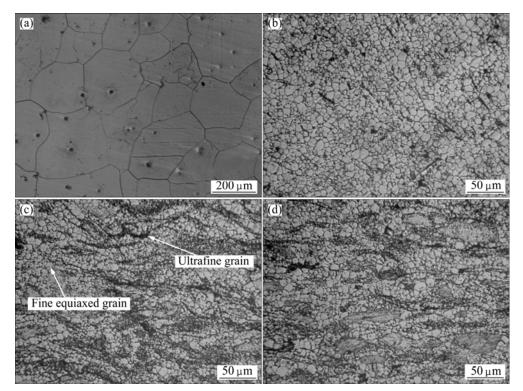
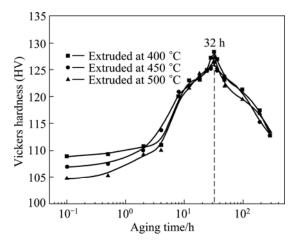


Fig. 1 Optical microstructures of longitudinal cross-section of samples after cast-T4 (a) and further hot extrusion at 400 °C (b), 450 °C (c) and 500 °C (d)

homogeneous after processing at 450 °C (Fig. 1(c)), and more prominently at 500 °C (Fig. 1(d)). Meanwhile, Mg–RE system alloy exhibits good plastic behaviors during extrusion at high temperature. The initial grains can be strongly elongated along the extrusion direction, resulting in the formation of streamline along the extrusion direction [13]. Therefore, the coarsened grains in this work elongated along the direction of extrusion further increase the inhomogeneity of the microstructure. The grains in the sample extruded at 400 °C can be broken more easily, which exhibits the best grain refinement and homogeneity of microstructure.

#### 3.2 Aging characteristic

Figure 2 shows the evolution of microhardness with isothermal aging time at 200 °C for the samples hot extruded at 400, 450 and 500 °C, respectively. The initial microhardness, peak microhardness and peak time are summarized in Table 2. The microhardness is increased by HV 22-26 after extrusion compared to the cast T4 alloy with a microhardness of HV 83 due to remarkable grain refinement and work hardening. Samples extruded at different temperatures exhibit similar microhardness evolution during isothermal aging. After the initial incubation stage of about 1.5 h, the microhardness increases rapidly until it reaches the peak at about 32 h. After that, the microhardness keeps stable in a relatively long range and then drops gradually as a result of over-aging. Extrusion temperature has some effects on the age hardening response in terms of as-extruded hardness, peak microhardness and hardening effect. At higher extrusion temperature, the grains coarsen and the precipitation tendency reduces, and thereby the as-extruded microhardness decreases with increasing extrusion temperature. Besides, the hardening effect, namelv microhardness increment. increases with increasing extrusion temperature due to more precipitation during isothermal aging. The sample



**Fig. 2** Age hardening curves of Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr alloy isothermally aged at 200 °C

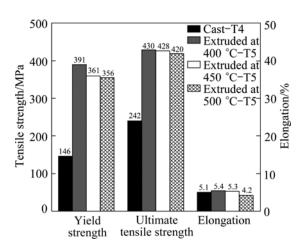
**Table 2** Key features of age hardening curves of Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr alloys isothermally aged at 200 °C

Processing status	Hardness of cast T4 alloy (HV)	Hardness of as- extruded (HV)	Peak	Hardness incremen (HV)	Peak
Extruded at 400 °C	· · ·	109	128.3	19.3	32
Extruded at 450 °C	83	107	126.5	19.5	32
Extruded at 500 °C		104.8	127.3	22.5	32

extruded at 400 °C exhibits the minimum hardening response, with the peak microhardness and microhardness increment of HV 128.3 and HV 19.3, respectively. With the extrusion temperature increasing, the age hardening response of extruded samples is improved. The peak microhardness and microhardness increment reach HV 126.5 and HV 19.5 for the sample extruded at 450 °C, and HV 127.3 and HV 22.5 at 500 °C, respectively. The improvement in the hardening response can also be attributed to the reduced extrusion precipitation amount with increased extrusion temperature.

#### 3.3 Mechanical properties

The tensile properties of the extruded samples at room temperature in extruded-T5 temper are shown in Fig. 3. The extrusion ratio is 10:1 and the T5 treatment is carried out at 200 °C for 32 h. The initial cast-T4 alloy without extrusion exhibits yield strength, ultimate tensile strength and elongation of 146 MPa, 242 MPa and 5.1%, respectively. With the extrusion temperature increases from 400 to 500 °C, the yield strength and ultimate tensile strength of the extruded Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr alloys decrease from 391 and

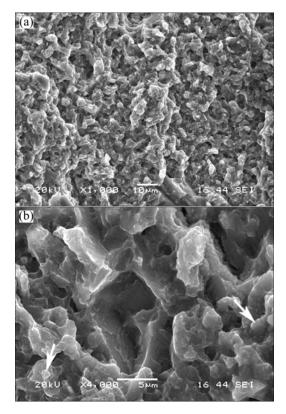


**Fig. 3** Mechanical properties of Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr alloy extruded at 400, 450 and 500 °C after isothermal aging at 200 °C for 32 h (extruded-T5 temper)

430 MPa to 356 and 420 MPa, respectively. Besides, the elongation decreases from 5.4% to 4.2%. These results indicate that raising extrusion temperature is detrimental to yield strength, ultimate tensile strength and elongation of the alloys. The data obtained from the tensile test are identical with the results from microstructure analysis. Consequently, 400 °C is the optimized extrusion temperature for this alloy. When the alloy extruded at lower temperature (400 °C), stronger strain hardening effect and banded structure lead to higher mechanical properties at room temperature as compared to those of the alloy extruded at 450 and 500 °C [14].

#### **3.4 Fractography**

The fractography of peak-aged Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr alloy (extruded at 400 °C) is shown in Fig. 4. At room temperature, the specimens exhibit predominant brittle fracture features due to their restricted dislocation slip systems, which usually tend to fail by cleavage fracture or quasi-cleavage fracture [15]. The close-up of the fracture surface shows that the fracture surface is composed of some spherical features which may be related with small particles embedded in the matrix, as pointed by the arrows in Fig. 4(b). This indicates that the small particles on the grain boundary may provide additional sites for crack nucleation [16].



**Fig. 4** Typical tensile fracture surfaces of Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr alloy extruded at 400 °C after isothermal aging at 200 °C for 32 h (extruded-T5 temper)

### 4 Conclusions

1) Due to severe dynamic recrystallization, the hot-extruded samples have much finer microstructure comparing with the as-cast ones. The grain size of the samples extruded at 400, 450 and 500 °C are 5.0, 5.3 and 5.9  $\mu$ m, respectively. The samples extruded at 400 °C exhibit the finest grain size.

2) After extrusion, the initial microhardness increases to HV 105–109 from HV 83. Both the time of peak aging for as-cast and as extruded alloy are 32 h. The peak microhardness of samples extruded at 400, 450 and 500  $^{\circ}$ C are HV 128.3, HV 126.5 and HV 127.3, respectively. The samples extruded at 400  $^{\circ}$ C exhibit the highest microhardness.

3) The tensile properties at room temperature of the samples extruded at 400, 450 and 500 °C followed by T5 temper are 391 MPa, 430 MP and 5.4%, 361 MPa, 428 MPa and 5.3%, 356 MPa, 420 MPa and 4.2%, respectively. The samples extruded at 400 °C exhibits optimized tensile properties.

## References

- LIANG S Q, GUAN D K, CHEN L, GAO Z H, TANG H X, TONG X T, XIAO R. Precipitation and its effect on age-hardening behavior of as-cast Mg–Gd–Y alloy [J]. Materials and & Design, 2011, 32(1): 361–364.
- [2] SOUBEYROUX J L, PUECH S. Phases formation during heating of Mg-Cu-Ag-Y bulk metallic glasses [J]. Journal of Alloys and Compounds, 2010, 495(2): 330–333.
- [3] ZHU Y M, MORTON A J, NIE J F. Improvement in the age-hardening response of Mg-Y-Zn alloys by ag additions [J]. Scripta Materialia, 2008, 58(7): 525-528.
- [4] KAMADO S, KOJIMA Y, NINOMIYA R, KUBOTA K. Ageing characteristics and high temperature tensile properties of magnesium alloys containing heavy rare earth elements [C]//LORIUER G W. International Magnesium Conference, 3rd, UMIST. Manchester, UK: 1997: 327–342.
- [5] ROKHIN L L, NIKITINA N I. Magnesium–gadolinium and magnesium–yttrium alloys [J]. Metallkd Z, 1994, 85(12): 819–823.
- [6] HOU X L, CAO Z L, ZHANG L L, FANG D Q, XU S W, KAMADO S, WANG L M. Microstructure and mechanical properties of extruded Mg-6.5Gd-1.3Nd-0.7Y-0.3Zn alloy [J]. Transactions of Nonferrous Metals Society of China, 2010, 20(s2): s508-s512.
- [7] HE S M, ZENG X Q, PENG L M, GAO X, NIE J F, DING W J. Microstructure and strengthening mechanism of high strength Mg-10Gd-2Y-0.5Zr alloy [J]. Journal of Alloys and Compounds, 2007, 427(1-2): 316-323.
- [8] GAO X, NIE J F. Enhanced precipitation-hardening in Mg–Gd alloys containing Ag and Zn [J]. Scripta Materialia, 2008, 58: 629–622.
- [9] WANG Q D, CHEN J, ZHAO Z, HE S M. Microstructure and super high strength of cast Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr alloy [J]. Materials Science and Engineering A, 2010, 528(1): 323-328.
- [10] CHEN Y J, WANG Q D, PENG J G, ZHAI C Q, DING W J. Effects of extrusion ratio on the microstructure and mechanical properties of

AZ31 Mg alloy [J]. Journal of Materials Processing Technology, 2007, 182(1-3): 281-285.

- [11] LIU X B, CHEN R S, HAN E H. Effects of ageing treatment on microstructures and properties of Mg-Gd-Y-Zr alloys with and without Zn additions [J]. Journal of Alloys and Compounds, 2008, 465(1-2): 232-238.
- [12] HE S M, PENG L M, ZENG X Q, DING W J, ZHU Y P. Comparison of the microstructure and mechanical properties of a ZK60 alloy with and without 1.3 wt.% gadolinium addition [J]. Materials Science and Engineering A, 2006, 433(1–2): 175–181.
- [13] WU Y J, PENG L M, ZENG X Q, LIN D L, DING W J, PENG Y H. A high-strength extruded Mg-Gd-Zn-Zr alloy with superplasticity

[J]. Journal of Materials Research, 2009, 24: 3596-3602.

- [14] YUAN G Y, LIU Y, DING W J, LU C. Effects of extrusion on the microstructure and mechanical properties of Mg–Zn–Gd alloy reinforced with quasicrystalline particles [J]. Materials Science and Engineering A, 2008, 474(1–2): 348–354.
- [15] LV Y Z, WANG Q D, ZENG X Q, ZHU Y P, DING W J. Behavior of Mg–6Al–xSi alloys during solution heat treatment at 420 °C [J]. Materials Science and Engineering A, 2001, 301(2): 255–258.
- [16] LI Z, DONG J, ZENG X Q, LU C, DING W J. Influence of Mg<sub>17</sub>Al<sub>12</sub> intermetallic compounds on the hot extruded microstructures and mechanical properties of Mg-9Al-1Zn alloy [J]. Materials Science and Engineering A, 2007, 466(1–2): 134–139.

# 挤压 Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr 合金的 显微组织与力学性能

周浩<sup>1</sup>, 王渠东<sup>1,2</sup>, 陈杰<sup>1</sup>, 叶兵<sup>1,2</sup>, 郭炜<sup>1</sup>

上海交通大学 轻合金精密成型国家工程研究中心,上海 200240;
上海交通大学 金属基复合材料国家重点实验室,上海 200240

**摘 要:** 研究铸态和挤压态 Mg-8.5Gd-2.3Y-1.8Ag-0.4Zr 合金的显微组织、时效强化和力学性能。铸锭在 T4 处 理后分别于 400、450 和 500 ℃ 进行挤压,挤压比为 10:1。在细晶强化和析出强化的共同作用下,于 400 ℃ 挤压 的样品经 T5 处理后可以得到最优的力学性能,所得的晶粒尺寸约为 5.0 μm,其初始和峰值硬度分别为 HV109 和 HV129。室温下的拉伸屈服强度、抗拉强度和伸长率分别达到 391 MPa、430 MPa 和 5.2%。 关键词: Mg-Gd-Y-Ag-Zr 镁合金;挤压;时效强化;微观组织;力学性能

(Edited by FANG Jing-hua)