Magnesium Technology 2012
Edited by: Suveen N. Mathaudhu, Wim H. Sillekens, Neale R. Neelameggham, and Norbert Hort
TMS (The Minerals, Metals & Materials Society), 2012

MICROSTRUCTURAL CHARACTERIZATION OF HOMOGENISED AND AGED Mg-Gd-Nd-Zn-Y-Zr ALLOYS

S. Khawaled, M. Bamberger, A. Katsman
Technion – Israel Institute of Technology, Haifa 3200, Israel

Keywords: Mg-Gd_Nd alloys, Precipitation, Microstructure, Mechanical properties

Abstract

The microstructure of the Mg—6%Gd-3.7%Nd-0.3%Zn-0.18%Y-0.15%Zr (%wt) alloy has been investigated after solution treatment at 540°C for 24hr followed by isothermal aging at 175°C up to 32 days by using of Vickers hardness, optical microscopy, scanning electron microscopy equipped with EDS, X-ray diffraction and transmission electron microscopy. It was observed that the homogenized alloy contained primary α-Mg solid solution, eutectic structures, cuboid shaped phases and Zr-rich clusters. The eutectic structures were the products of a 'quasibinary eutectic reaction' L→α-Mg+β-Mg_5RE. The eutectic phase was characterized to be of Mg_5Gd prototype with the composition $Mg_5(Gd_xNd_{1-x}$, x≈0.2). The cuboid shaped phases, with the composition $Gd_4(Nd_xY_{1-x}$, x≈0.5), grew during aging and reached ~3μm average size. Precipitation of β″ and β′ phases during aging was observed. The maximum microhardness was achieved after 16 days of aging.

Introduction

Magnesium alloys containing heavy rare earth metals are very attractive candidates for the automotive industry including racing cars and aerospace applications [1-8], due to their high strength properties combined with low density. Due to the large solubility of RE element in Mg matrix at high temperature and its rapid decrease with lowering temperature, the Mg–RE alloys show remarkable age-hardening response during isothermal aging at 175°C, and thus high strength is to be expected.

Microstructural characterization of the Mg–RE alloys performed by many researchers [1-11] has confirmed a four staged precipitation sequence: [α-Mg supersaturated solid solution (S.S.S.S.) → β″(DO_{19}) → β′(bco) → $β_1$(fcc) → β(fcc)] [2,3,9,10], where the later precipitate can coexist with the former. Among the four precipitated phases, the coherent β″and β′ phases are considered to be the primary strengthening phases [2,4,8,10]. The yield strength or hardness usually reaches a maximum, as the materials form a microstructure with fine β′ precipitates during aging [1-4].

In the present work, the microstructural evolution of a Mg-Gd-Nd-based alloy during isothermal aging at 175°C. This specific composition was selected in order to achieve maximum age hardening response, improved castability, grain refinement and strength. It was investigated by a combination of optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), energy dispersive X-ray spectrometry (EDS) and X-ray diffraction (XRD). Microhardness measurements were performed to reveal the effect of precipitation on the mechanical properties of the alloy.

Experimental

The chemical composition of the investigated alloy was Mg-6Gd-3.7Nd-0.3Zn-0.18Y-0.15Zr (wt.%).
Specimens cut from the cast ingot were solution treated (ST) at 540°C for 24hr, quenched into hot water of about 70°C, and then aged at 175°C in an oil bath for various periods of time up to 32days followed by water quenching.

The microhardness of aged specimens was measured by a Vickers hardness tester under a load of 50gr and holding time of 15sec. 20-40 indentations were conducted on each specimen. The specimens were polished with diamond paste and etched for 20sec in Ethanol 0.95%wt and HNO_3 0.05%wt (Nital) solution.

A combination of OM, SEM, TEM, EDS and XRD was carried out to characterize as- cast and homogenized micro-structure of the alloy. Metallographic specimens were polished and etched in an Acetic Glycohol enchant (100ml ethanol, 20ml water, 6gr picric acid and 5ml acetic acid). Thin foils for TEM characterization were prepared by ion beam thinning, and were examined at the acceleration voltage of 200 kV. The compositions of the matrix and of secondary phases in the TEM specimens were analyzed by EDS instrument attached to the SEM, operating at 10kV. The constituent phases of the alloy in different conditions were identified by XRD with Cu Kα radiation.

Results

As-cast microstructure

Fig.1 shows the as-cast alloy consists mainly of the α-Mg phase as a matrix, eutectic-like structures, cuboid-like phases and Zr-rich clusters.

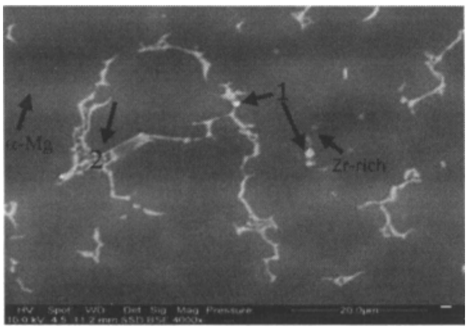

Fig. 1. Microstructure of the as-cast sample: SEM image shows the cuboid-like phase (1) and eutectic-like structure (2).

The cuboid-like particles in micrometer scale are frequently observed in the association with Zr-rich clusters and eutectic- like structures as shown in Fig.1. The cuboid-like particles are Y-rich phases containing Gd and Nd. Composition of these particles $Y_2(Gd_xNd_{1-x}$, $x \approx 0.9$) was determined by EDS SEM using TEM samples.

ST microstructure

The purpose of homogenization for precipitate hardened Mg-RE alloys is to dissolve soluble as cast phases into α-Mg matrix and obtain supersaturated solid solution by quenching. The microstructure of the alloy was investigated in the ST and aged (175°C) conditions. It was observed that the homogenized alloy contained primary α-Mg solid solution, eutectic structures, cuboid shaped phases and Zr-rich clusters (Fig. 2).

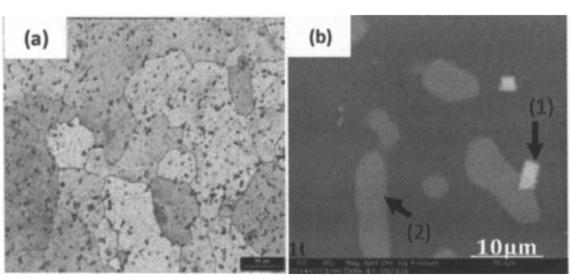

Fig. 2. Microstructure of the ST specimens: (a) Optical image showing the grain size and (b) SEM image showing the cuboid shaped phase (1) and eutectic structure (2).

The compositions of the phases were analyzed by EDS. The composition of the eutectic structures was characterized to be of α-Mg+β-$Mg_5(Gd_xNd_{1-x}$, $x \approx 0.2$). The cuboid shaped phases have the composition $Gd_4(Nd_xY_{1-x}$, $x \approx 0.5$). TEM micrographs (Fig.3) revealed the 1-2 μm cuboid one-phase particles (Fig.3a) and the two-phase skeleton-like eutectic structures (Fig.3b).

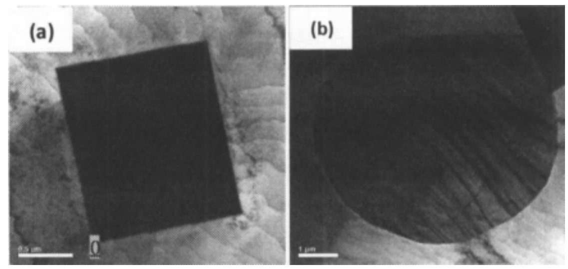

Fig. 3. TEM observations of **(a)** cuboid shaped phase and **(b)** eutectic-like structure.

Microstructure of aged samples

Microstructure of the aged samples consists of the eutectic structure, cuboid shaped particles and small precipitates uniformly distributed in the matrix. The cuboid shaped particles grow during aging up to ~3 μm in size; SEM+EDS analysis revealed that their composition did not change.

Crystal structure and composition of precipitates within the α-Mg matrix were investigated by TEM (Figs. 4-7); there were diffuse spots between the α-Mg matrix spots in the selected area electron diffraction (SAED) patterns, which were brighter than the matrix spots (Fig. 4). TEM images and corresponding SAED patterns of precipitates in a specimen aged for 16 days (peak-aged specimen) are shown in Fig.5. It was observed that the microstructure contains a high number density of fine plate shape β'' precipitates and a little number of globular particles β' uniformly distributed within the α-Mg matrix (Fig. 6).

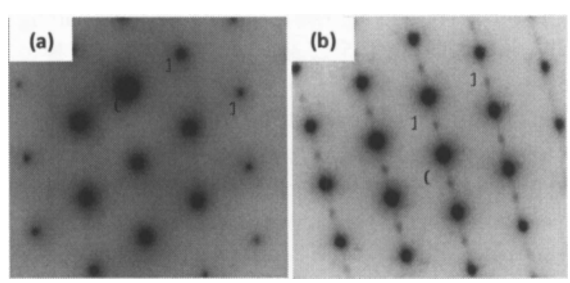

Fig. 4. SAED patterns of the specimen aged for (a) ST and (b) 32days with Z.A $[11\bar{2}3]_\alpha$.

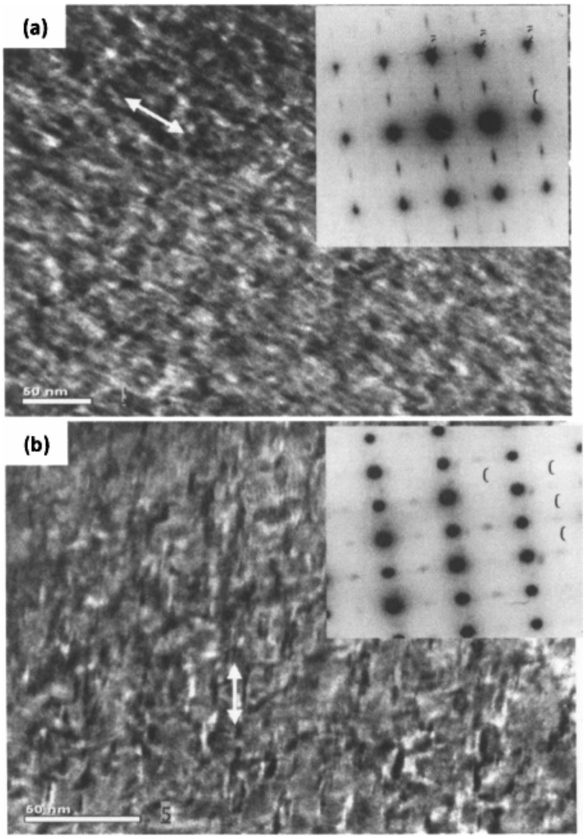

Fig. 5. TEM images and corresponding SAED patterns of the specimen aged for 16days at 175C: Z.A $[01\bar{1}0]_\alpha$ in (a) and Z.A $[2\bar{1}\bar{1}0]_\alpha$ in (b).

Combination of reflections from the two precipitates, β″and β′, appears in the corresponding SAED pattern (Fig. 5). The β″ precipitates with DO_{19} structure $(a{\sim}2a_{\alpha-Mg} = 0.64\text{nm}, c{\sim}c_{\alpha-Mg} = 0.52\text{nm})$ correspond to diffuse spots at 1/2 distance of $\{01\bar{1}0\}_\alpha$ or $\{2\bar{1}\bar{1}0\}_\alpha$ reflections, with the following orientation relationships (OR): $[0001]_{\beta''}//[0001]_\alpha$, $\{01\bar{1}0\}_{\beta''}//\{01\bar{1}0\}_\alpha$; and the β′ phase with Base Centered Orthorhombic (BCO) structure $(a{\sim}2a_{\alpha-Mg} = 0.64\text{nm}$, $b{\sim}2.2\text{nm}$, $c{\sim}c_{\alpha-Mg} = 0.52\text{nm})$ gives rise to the additional spots at 1/4 $\{01\bar{1}0\}_\alpha$, 1/2 $\{01\bar{1}0\}_\alpha$ and 3/4 $\{01\bar{1}0\}_\alpha$, with the following OR: $[001]_{\beta'}//[0001]_\alpha$, $\{100\}_{\beta'}//\{2\bar{1}\bar{1}0\}_\alpha$.

The microstructure of the sample aged for 32 days contains a little number of plate-shaped FCC β_1 precipitates ($a \approx 0.74\text{nm}$) (Fig. 7) along with β″and β′ small precipitates.

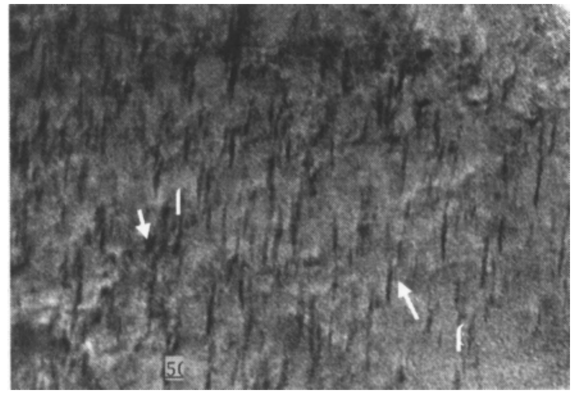

Fig. 6. TEM image of the specimen aged for 16days at 175C.

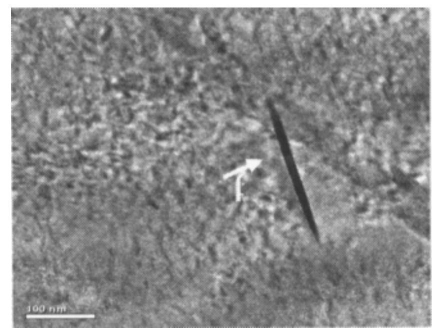

Fig. 7. TEM image of the specimen aged for 32days at 175C.

Microhardness

The microhardness evolution during isothermal aging at 175°C up to 32days is shown in Fig. 8. Microhardness increased and reached a maximum value of about 100 HV after 16 days of aging. Further aging led to decrease in the microhardness.

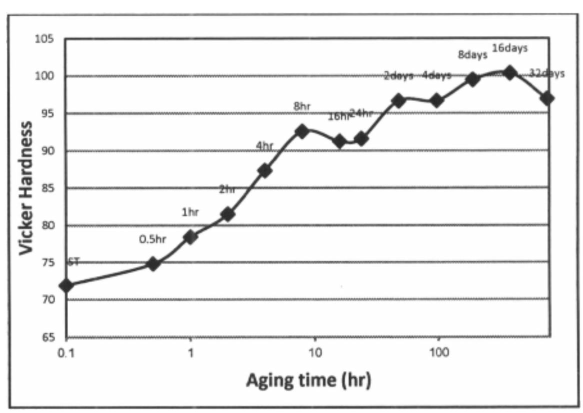

Fig. 8. Age hardening curve of the alloy aged at 175°C

Discussion

The as-cast microstructure of the Mg-Gd-Nd-Zn-Y-Zr alloys consists of primary α-Mg solid solution, skeleton-like eutectic structure [1], small Y-rich cuboid-like phase and Zr-rich clusters. The eutectic structures were the products of a 'quasibinary eutectic reaction' L→α-Mg+β-Mg_5RE. The eutectic phase was characterized to have Mg_5Gd type FCC crystal structure with a ≈ 2.2nm and a composition of $Mg_5(Gd_xNd_{1-x}$, x≈0.2).

During solution treatment at 540°C, Gd and Nd atoms are transferred from eutectic-like structure to cuboid-like phase to form the cuboid shaped phase with the stable composition $Gd_4(Nd_xY_{1-x}$, x≈0.5). The cuboid shaped particles grow during aging and reach ~3μm average size.

Aging of solution treated and quenched alloy results in precipitation of β'' and β' phases. Generally, the precipitation sequence in the investigated alloy during aging is identical to that reported for many Mg-RE alloy systems [2,3,9,10], i.e. the four staged precipitation sequence:[$\alpha - \text{Mg S.S.S.S.} \rightarrow \beta''(D0_{19}) \rightarrow \beta'(\text{bco}) \rightarrow \beta_1(\text{fcc}) \rightarrow \beta(\text{fcc})$]. The peak of microhardness after 16 days of aging corresponds to the combination of β''and β' precipitates (Fig.5). The plate shape coherent β'' precipitates in α − Mg matrix have OR: $[0001]_{\beta''}//[0001]_{\alpha}$, $\{01\bar{1}0\}_{\beta''}//\{01\bar{1}0\}_{\alpha}$. The globular precipitates β' have OR: $[001]_{\beta'}//[0001]_{\alpha}$, $\{100\}_{\beta'}//\{2\bar{1}\bar{1}0\}_{\alpha}$. These results are similar to those reported by K.Y. Zheng et al. [10] for Mg-11Gd-2Nd-0.5Zr. The further aging up to 32 days results in coarsening of β'' and β' precipitates and their transformation to β_1 phase (Fig.7). These processes are accompanied by decrease of microhardness (Fig.8).

Conclusion

The Mg-Gd-Nd based alloy containing Y, Zn and Zr was investigated in the as-cast, ST and aged conditions. Skeleton-like eutectic structure α-Mg+β-$Mg_5(Gd_xNd_{1-x}$, x≈0.2) and cuboid shaped particles were found in all conditions. Y-rich cuboid-like particles found in the as-cast alloy transformed to cuboid shaped $Gd_4(Nd_xY_{1-x}$, x≈0.5) during ST and then grow during aging. Precipitation of plate shape β'' and globular β' particles during aging resulted in maximum of microhardness increase after 16 days. The further aging results in coarsening of β'' and β' precipitates and their transformation of β_1 phase.

Reference

1. K.Y. Zheng et al. / Materials Science and Technology (2008) VOL24 NO3.
2. S.M. He et al. / Materials Science and Engineering A 431 (2006) 322-327.
3. K.Y. Zheng et al. / Materials Science and Engineering A 492 (2008) 185-190.
4. L. Gao et al. / J Materials Science (2009) 44:4443-4454.
5. Zheng Kai-yun, et al. / Trans. Nonferrous Met. Soc. China 17 (2007).
6. D. Li et al. /Journal of Alloys and Compounds 439 (2007) 254-257.
7. K.Y. Zheng et al. / Materials Science and Engineering A 491 (2008) 103-109.
8. K.Y. Zheng et al. / Materials Science and Engineering A 454-455 (2007) 314-321.
9. K.Y. Zheng et al. / Materials Characterization 59 (2008) 857-862.
10. K.Y. Zheng et al. / Materials Science and Engineering A 489 (2008) 44-54.
11. G. Sha et al. / Materials Science and Engineering A 527 (2010) 5092-5099.