Scripta Materialia 128 (2017) 14–17

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat

Regular Article

Early precipitate morphologies in Mg-Nd-(Zr) alloys

Ellen L.S. Solomon, Vicente Araullo-Peters, John E. Allison, Emmanuelle A. Marquis *

Department of Material Science and Engineering, University of Michigan, 2300 Hayward St., Ann Arbor, MI 48109, USA

ARTICLE INFO

Article history:
Received 25 July 2016
Received in revised form 12 September 2016
Accepted 12 September 2016
Available online xxxx

Keywords:
Magnesium alloy
Precipitation
Atom probe tomography
Scanning/transmission electron microscopy (STEM)

ABSTRACT

Aging of Mg—Nd alloys involves the formation of Guinier-Preston zones followed by β‴ precipitation that is associated with peak hardness. Using atom probe tomography and high-angle annular dark-field scanning transmission electron microscopy, the three-dimensional morphologies of the coherent Guinier-Preston zones and β‴ precipitates were quantified. The Guinier-Preston zones form equiaxed plates while the β‴ precipitates are elongated along the $[0001]_{Mg}$ direction thereby minimizing elastic strains. The β‴ precipitates exhibit additional faceting along the $[0001]_{Mg}$ direction and along the $\langle 1\bar{1}01\rangle_{Mg}$ and $\langle 1\bar{1}02\rangle_{Mg}$ directions when viewed along the $\langle 11\bar{2}0\rangle_{Mg}$ zone axes, which can be affected by strain field interactions from nearby precipitates.
© 2016 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Magnesium alloys containing rare earth elements such as Nd are well known for their significant aging response and formation of precipitates with prismatic habit planes [1–5]. WE43 and WE54 are two commonly used Mg-rare earth (RE) element alloys containing Nd and Y as the major strengthening elements, and a significant amount of research has focused on characterizing precipitate phases in these alloys [3–8]. However, the different alloying elements make the microstructure complex and the role of each element on the precipitate processes difficult to identify. A more meaningful approach involves the analysis of model alloys, as illustrated through previous experimental [9–14] and computational [15–18] studies.

In the Mg—Nd system, the precipitation sequence involves the formation of metastable phases from the supersaturated solid solution and is understood to be [3,9–11,14,19,20]:

$$\text{SSSS}\rightarrow\text{GP Zones (N, V, hexagons)}\rightarrow\beta'''\rightarrow\beta_1(\text{Mg}_3\text{Nd})\rightarrow\beta(\text{Mg}_{12}\text{Nd})\rightarrow\beta_e(\text{Mg}_{41}\text{Nd}_5)$$

Guinier-Preston (GP) zones include the so-called "N", "V", and hexagon arrangements either as individual units or single layer sheets [9,11,20]. Precipitates often referred to as β′ are more accurately labeled here as β‴ according to [20]. The GP zones and β‴ precipitates form in the early stages of aging in Mg—Nd alloys and are responsible for the increased alloy strength when aged between 170 °C [9] and 250 °C [21]. Therefore, characterization of these phases is essential in order to understand the microstructure-property relationships in Mg-RE alloys.

While transmission electron microscopy (TEM) studies of Mg—Nd alloys have successfully characterized the atomic structure of the precipitates forming in the early stages of aging [9,11,19–21], their three-dimensional shape remains to be established. With the exception of Ref. [9,20,21], all previous work using transmission electron microscopy (TEM) have imaged these precipitates along the $[0001]_{Mg}$ direction. As a consequence, a more detailed description including the three-dimensional morphology and size distributions for each dimension is needed to understand the mechanisms controlling precipitate shape and size evolution and to relate the microstructure to the strengthening behavior. Phase field calculations of the 3D morphology suggested β‴ to be plate-shaped with the longest dimension along the $\langle 1\bar{1}00\rangle_{Mg}$ directions [17], yet these predictions have remained inconclusive due to the lack of experimental evidence.

To answer this question, this paper provides a quantitative description of the shapes of GP zones and β‴ precipitates in a Mg—Nd alloy using atom probe tomography (APT) and high-angle annular dark-field (HAADF) scanning transmission electron microscopy (STEM).

The alloys examined in this paper were provided by Magnesium Elektron North America Inc. The compositions of the two alloys were measured by inductively coupled plasma mass spectrometry and found to be Mg-2.35 wt.% Nd (Mg-0.40 at.% Nd) and Mg-2.18 wt.% Nd-0.32 wt.% Zr (Mg-0.38 at.% Nd-0.09 at.% Zr at.%). Results from APT were collected from the Mg-Nd-Zr alloy while results from HAADF-STEM were obtained from the binary Mg—Nd alloys. Precipitation is not affected by the presence of Zr thereby allowing direct comparisons to be made between the two alloys. The alloys were encapsulated in quartz tubes in an argon atmosphere and solution treated at 560 °C for 24 h followed by water quenching. The solution treatment was chosen to ensure the sample was sufficiently homogenized. Although the

* Corresponding author.
E-mail address: emarq@umich.edu (E.A. Marquis).

http://dx.doi.org/10.1016/j.scriptamat.2016.09.015
1359-6462/© 2016 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

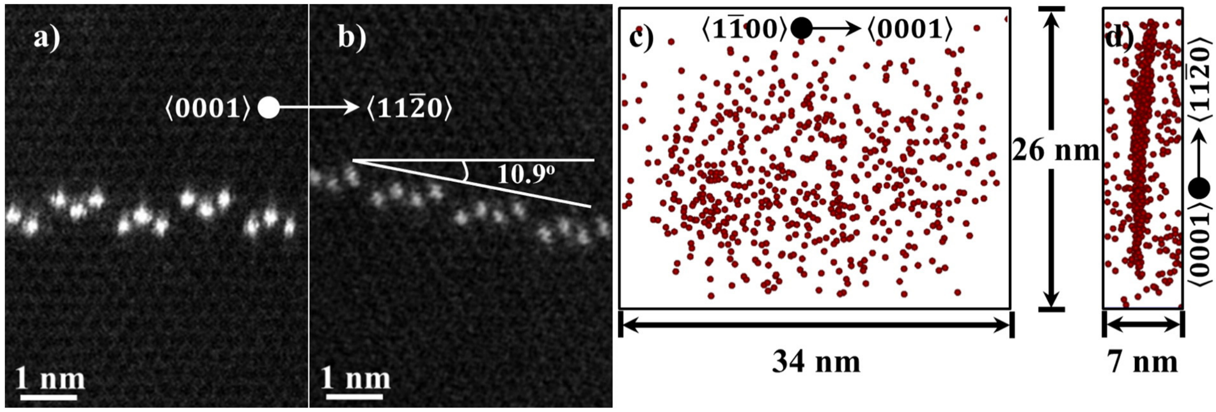

Fig. 1. a) and b) HAADF-STEM images of the two GP variants after aging at 200 °C for 16 h with the incident beam parallel to the $[0001]_{Mg}$ zone axis. c and d) APT data of a GP variant after aging at 250 °C for 15 min. Precipitates are viewed along the c) $\langle 1\bar{1}00\rangle_{Mg}$ and d) $[0001]_{Mg}$ directions. Nd atoms are shown in red. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

temperature is above that of the eutectic, the sample did not experience any melting. Samples were then aged in an oil bath at either 200 °C for 1 h and 16 h or 250 °C for 3 min, 15 min, and 1 h followed by water quenching. The APT specimens were electropolished with a solution of 25% perchloric acid in acetic acid using 14–20 V and then fine polished with a solution of 2% perchloric acid in butoxyethanol with 5–10 V. The APT data was collected on a Cameca LEAP XHR4000 instrument operated in voltage mode with a specimen temperature of 50 K. The data was collected at detection rates varying between 0.005 and 0.01 atoms per pulse on average using a voltage pulse fraction of 20% and a repetition rate of 200 kHz. The data was then reconstructed using the Integrated Visualization and Analysis Software (IVAS) package. The reconstruction parameters, geometric factor, and image compression factor were chosen to ensure that the planar spacings observed at identified poles and the angles between such planes match that of the hexagonal magnesium matrix. Crystallographic analysis methods developed for APT [22] were used to determine the orientation of the APT data. TEM specimens were prepared using the method described in Ref. [23]. Micrographs were taken using a JEOL 2100 with a collection angle of 52 mrad and a double-corrected JEOL 3100R05 microscope operated at 300 kV with a collection angle of 74 mrad.

Examination of the morphology and size in 3D is experimentally challenging for TEM, especially for GP zones where the dimension along $\langle 1\bar{1}00\rangle_{Mg}$ directions is only a couple atomic layers thick. However, the 3D nature of APT data provides a means to measure shape and dimensions. Since APT cannot always resolve the atomic structure of the precipitates, the GP zones and β‴ were distinguished by their crystallographic orientations.

Selected images illustrating the 3D morphology of GP zones using APT and HAADF-STEM are shown in Fig. 1. As previously reported [9], the GP zones are thin platelets of Nd atoms, parallel to $\{1\bar{1}00\}_{Mg}$ planes. The N and V arrangements observed by STEM are not recognizable by APT, despite the slight difference in habit plane orientation (Fig. 1). This difference in habit plane is related to the staggering of the N and V units. The V units (Fig. 1a) alternate such that their apex is pointing up in one unit and down in the neighboring unit. Each matching unit lies on the same $\{1\bar{1}00\}_{Mg}$ plane. Conversely, the N units (Fig. 1b) have identical atomic arrangements and each subsequent N unit sits on different but parallel $\{1\bar{1}00\}_{Mg}$ planes creating an angle of 10.9° from the $\{1\bar{1}00\}_{Mg}$ plane. Size distributions and aspect ratios of the GP platelets were measured independently by APT and STEM and are shown in Fig. 2, revealing comparable dimensions from the two techniques. Most of the GP platelets are slightly longer in the $[0001]_{Mg}$ directions and occasionally in the $\langle 11\bar{2}0_{Mg}\rangle$ directions. The dimension along $\langle 1\bar{1}00_{Mg}\rangle$ directions is fixed by the atomic structure and therefore not plotted. The linear correlation between the dimensions along the $[0001]_{Mg}$ and the $\langle 11\bar{2}0\rangle_{Mg}$ directions suggests that GP zones are thin plates with a constant aspect ratio that is independent of size.

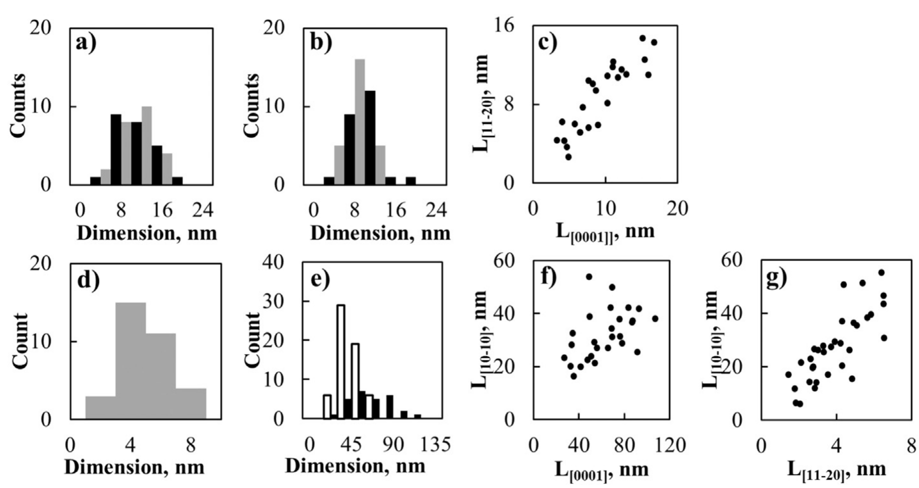

Fig. 2. Size distributions and aspect ratios of a-c) GP zones and d-g) β‴ precipitates after aging at 200 °C (for GP zones) or 250 °C (for β‴) for 1 h. Dimensions along $\langle 11\bar{2}0\rangle_{Mg}$ are in grey, $\langle 1\bar{1}00\rangle_{Mg}$ in white, and along $[0001]_{Mg}$ are in black. The data was obtained from a) APT b) HAADF-STEM c) APT d-g) HAADF-STEM.

Download English Version:

https://daneshyari.com/en/article/5443755

Download Persian Version:

https://daneshyari.com/article/5443755

Daneshyari.com