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Compositional evolution of long-period stacking ordered structures in magnesium studied by atom probe tomography

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ABSTRACT

Mg alloys containing long-period stacking ordered (LPSO) structures are strong and ductile compared to conventional Mg alloys. We study here the compositional evolution of LPSO structures in a Mg97Y2Zn1 (at.%) alloy upon annealing at 500 °C using atom probe tomography. In the material annealed for 2.5 h, the Zn/Y ratio of the building blocks in the interdendritic LPSO phase (0.73) is close to the stoichiometric composition of Y_8Zn_6 $L1_2$ clusters while that in plate-type LPSO structures (0.66) slightly deviates from the ideal value. The Y/Zn enrichment in LPSO structures in the α-Mg matrix slightly decreases with increasing annealing time.

Long-period-stacking-ordered (LPSO) structures in Mg-Y-Zn alloys have unique crystal structures [1–4] and lead to considerable improvement in mechanical properties [5–9]. Mg-Y-Zn alloys containing LPSO structures typically show yield strength values ranging from 260 to 380 MPa and a total elongation ranging from 6 to 20% in uniaxial tensile tests [7–9]. LPSO structures show a modified stacking periodicity along the c-axis as compared to Mg and are chemically ordered by solute enrichment of particular layers [1]. LPSO structures include several polytypes, referred to as 10H, 14H, 18R and 24R, where 14H and 18R structures have frequently been observed [1–3,10–12]. They consist of Y/Zn-enriched building blocks that have a local FCC stacking sequence on {0001} close packed planes [3,11]. In the building blocks of Mg-Y-Zn alloys, $L1_2$ type Zn_6Y_8 clusters have often been observed [2,13–15].

Previous works reported on the evolution of LPSO structures in Mg-Y-Zn alloys upon annealing [4,16–18]. After casting, dilute Mg-Y-Zn alloys consist of an interdendritic LPSO phase and an α-Mg matrix with LPSO structures [3,4,19]. The 18R phase is formed during solidification of Mg-Y-Zn alloys and is transformed to the 14H phase upon heat treatment in the temperature range of 350–500 °C [10,18]. CALPHAD-based thermodynamic calculations of Mg-Y-Zn alloys showed that the 14H is an equilibrium phase below 537 °C [12]. Kim et al. [4] reported on the structural evolution of LPSO phases in a $Mg_{97}Y_2Zn_1$ (at.%) alloy upon annealing at 500 °C for times between 2.5 and 10 h. In the α-Mg matrix, single building blocks and various metastable LPSO building blocks transform to the 14H structure upon annealing. In the interdendritic LPSO phase, diverse metastable LPSO building blocks grow from the 18R structure upon annealing. Liu et al. [20] reported that the volume fraction of LPSO structures in an α-Mg matrix increased for a $Mg_{97}Y_2Zn_1$ (at.%) alloy upon annealing at 500 °C for less than 10 h, and decreased when annealing was extended for longer than 10 h.

Chemical compositions of LPSO structures estimated in the previous works are not consistent. An atom probe tomography (APT) analysis on a nanocrystalline $Mg_{97}Y_2Zn_1$ (at.%) alloy showed that the chemical composition of Y/Zn enriched layers was approximately 10 at.% Y and 3 at.% Zn [21]. A TEM energy dispersive X-ray spectroscopy (EDX) analysis on a $Mg_{97}Y_2Zn_1$ (at.%) alloy yielded an average chemical composition of 18R and 14H interdendritic LPSO phases for Mg-8 at.% Y-8 at.% Zn and Mg-7 at.% Y-6 at.% Zn, respectively [3]. Abe et al. [1] reported that compositions of Y/Zn-enriched layers were most likely identical for all the LPSO polytypes. Egusa et al. [2] suggested that $L1_2$ type Zn_6Y_8 clusters in LPSO building blocks could tolerate a considerable degree of disorder at their Y and Zn sites and led to the non-stoichiometric composition of LPSO structures. Thus, estimation of chemical composition of LPSO building blocks could provide information on a degree of chemical ordering in LPSO structures. Therefore, systematic estimation of chemical composition of LPSO structures is required to understand the chemical ordering behavior depending upon location, i.e. the interdendritic LPSO phase or the α-Mg matrix, and depending upon annealing time.

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Here we report on atom probe tomography (APT) analyses on (1) the chemical composition of LPSO building blocks in the interdendritic LPSO phase and in the α-Mg matrix in the material annealed at 500 °C for 2.5 h and (2) the evolution of chemical composition of LPSO structures in the α-Mg matrix upon annealing at 500 °C between 2.5 and 10 h.

A $Mg_{97}Y_2Zn_1$ (at.%) alloy was molten and cast in an induction furnace under 20 bar pressure Ar atmosphere. The cast material was annealed at 500 °C for 2.5 h and 10 h, followed by water quenching. A series of needle-shaped APT specimens were site-specifically prepared from the bulk materials using a dual-beam focused-ion-beam (FIB, FEI Helios NanoLab™ 600i) system. A local electrode atom probe (LEAP, 3000× HR™, Cameca Instruments) was employed to collect APT data. The measurements were performed in voltage mode at 60 K under ultra-high vacuum of about 8×10^{-9} Pa. The pulse fraction and repetition rates were 15% and 200 kHz, respectively. The data sets were reconstructed by tip profile reconstruction and evaluated using the IVAS 3.6.6 software provided by Cameca Instruments. Samples for TEM analysis were cut into discs with a diameter of 3 mm and a height of 1 mm. The discs were ground to a thickness of 200 μm, then twin-jet electro-polished in a solution of 5.3 g lithium chloride, 11.2 g magnesium perchlorate, 500 ml methanol and 100 ml 2-butoxy-ethanol at −30 °C. A more detailed experimental description of the applied high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) protocols is available elsewhere [4].

Fig. 1(a) shows an SEM overview image and a typical HAADF-STEM image of an interdendritic LPSO phase observed after annealing at 500 °C for 2.5 h. The interdendritic LPSO phase consists of LPSO structures with bright contrast and α-Mg bands with dark contrast [4]. Fig. 1 (b) shows the 3D elemental distribution of Y and Zn atoms, acquired from an APT analysis containing an interdendritic LPSO phase. Fig. 1 (c) presents the distribution of Mg, Y and Zn atoms within a sub-

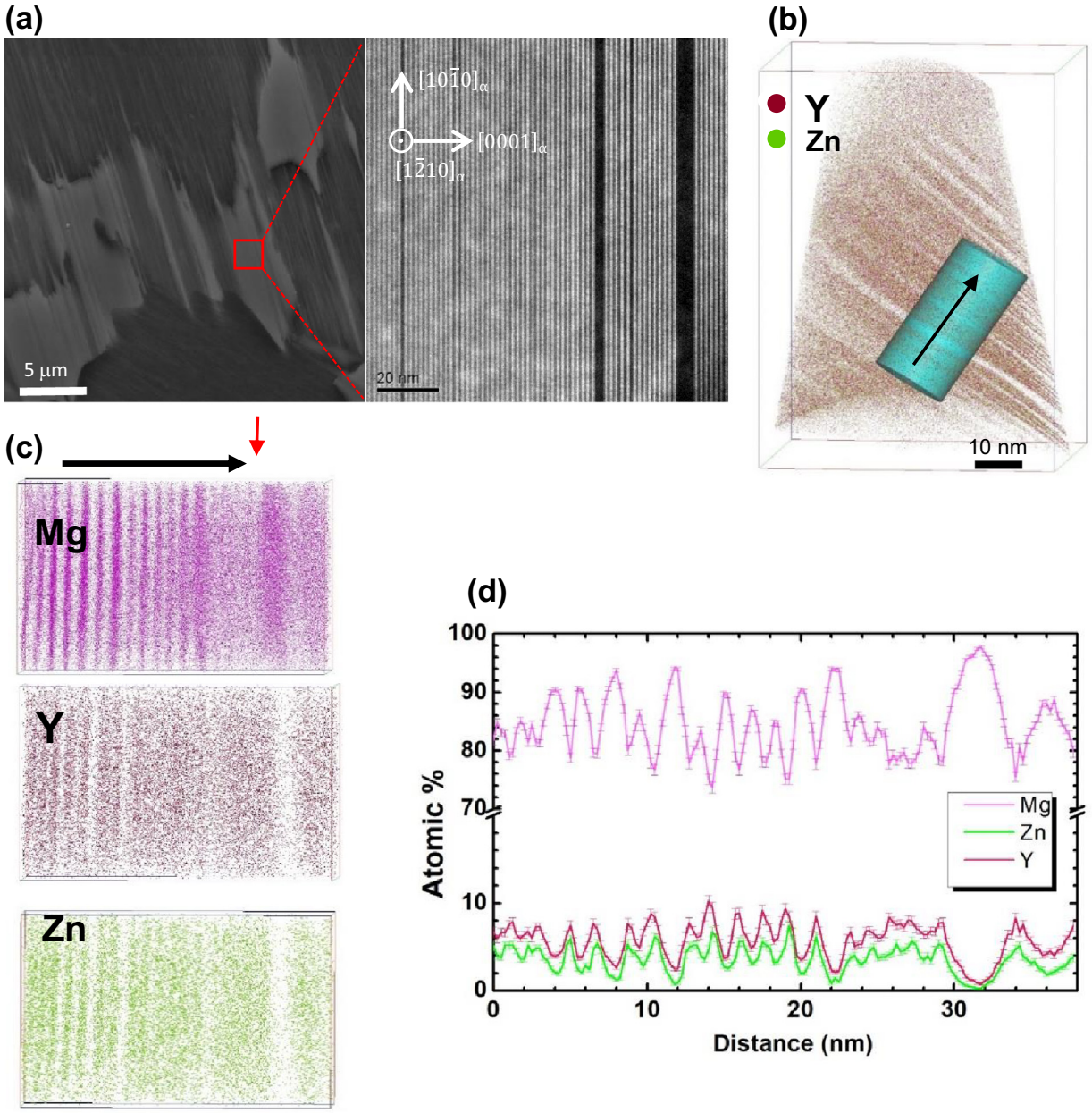

Fig. 1. (a) An overview SEM image and a typical low magnification HAADF-STEM image of an interdendritic LPSO phase of the material annealed at 500 °C for 2.5 h. (b) The 3D elemental distribution of Y and Zn in an APT tip obtained from the interdendritic LPSO phase. (c) The distribution of Mg, Y and Zn in the cylindrical region of interest (ROI) shown in (b). (d) The 1D-concentration profile of Mg, Y and Zn from the ROI shown in (b) and (c).