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Structural Change of MgNiY Long Period Stacking Order Investigated with Atom Probe

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Magnesium is emerging as a lightweight material for mass reduction and structural efficiency in many automotive and aerospace applications. Poor corrosion resistance and brittleness are the main concerns that hinder the application of Mg and Mg-based alloys. Alloying with a few atomic percent of rare earth (RE) elements and transition metals (TM) have been proven effective in countering these issues. $Mg_{97}Y_2Ni$ synthesized via rapid solidification exhibits a tensile yield strength as high as 610 MPa along with improved resistance to corrosion [1]. The improved mechanical properties can be attributed to the long period stacking ordered (LPSO) structure developed during solidification [2]. Its structure is described as a periodic stacking fault consisting of $L1_2$ type RE_8TM_6 clusters interrupting the ordered hexagonal Mg matrix. Examples of LPSO structural variations include 10H, 18R and 14H ("R" stands for Rhombohedral and "H" for hexagonal) where the numbers represent structural block size (i.e. 10H has 10 Mg (0001) atomic planes forming the basic unit).

Magnesium is highly sensitive to galvanic corrosion, due to its low corrosion potential. Several studies have reported an initial barrier effect for LPSO to protect the Mg matrix from corrosive media [3]. In alloys with an LPSO structure, Mg and RE/TM act as galvanic couples over a small distance that only extends a few lattice planes. To understand the corrosion behavior of LPSO structures, further investigation at the relevant scale is an essential building block for alloy development. Although transmission electron microscopy (TEM) provides excellent lateral resolution, information in the depth direction is lost because images are formed by a beam of electrons transmitted through a specimen typically over tens of nanometers in thickness. Atom probe tomography (APT) is a unique complementary technique that achieves sub-nanometer resolution in three-dimensional space. Combining APT and TEM provides extremely powerful insights into structural analysis. The growth mechanisms of LPSO in MgAlGa revealed by Inoue et.al. is an excellent example of obtaining microstructure and atom distribution at the atomic scale [4].

This work focuses on comparing $Mg_{91}Ni_4Y_5$ and $Mg_{92}Ni_3Y_5$ samples (both annealed at 500℃ for 10 days) using APT and TEM. Microscopically, both samples consist of α-Mg phase and an LPSO phase that is clearly different with respect to the size and volume fraction. The different hydrogen generation yields exhibited by the materials indicates that they do not present the same amount of corrodible Mg atoms. This means that the bonding between Mg and Ni and/or Y atoms is not the same in the two materials. $Mg_{92}Ni_3Y_5$ alloy has a lower yield due to Mg-Ni-Y nano complexes that do not react with water to form H_2. To successfully reveal the LPSO structure in APT, the C-axis must be aligned with the evaporation direction, which takes advantage of the highest spatial resolution. LPSO grains exposed to the mechanically polished surface are randomly distributed. Electron backscatter diffraction (EBSD) mapping was carried out to locate C-axis grains that are normal to the surface (Figure 1). APT specimens were extracted using the standard lift-out method and sharpened to a needle-shaped geometry using a focused ion beam [5]. APT analysis was carried out in voltage pulsing mode at 15% pulse

fraction and a base temperature of 30 K. Atom maps along the pole show lattice planes consisting of Mg atoms revealing atomic resolution along the (0001) plane. Ni/Y enrichment periodicity in Mg can be further confirmed using spatial distribution map (SDM) analysis derived from the APT data (Figure 2) [6]. In contrast, Ni and Y peaks in the SDM are broader and appear to occupy the same lattice plane that repeats at the frequency of every 4th Mg layer. Synchronization of Ni/Y enriched layers with Mg (0001) plane is expected for the LPSO structure but the number of Ni/Y layers observed by APT is different from the theoretical $L1_2$-type RE_8TM_6 clusters. Further investigation using high-resolution TEM observation and simulations will be presented.

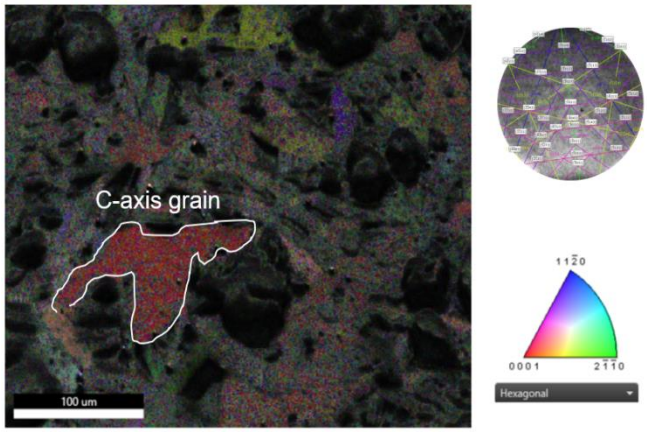

Figure 1. EBSD inverse pole figure map overlayed with SEM image in grayscale. The inset Kikuchi-diffraction pattern confirms the c-axis of the highlighted grain normal to the surface direction.

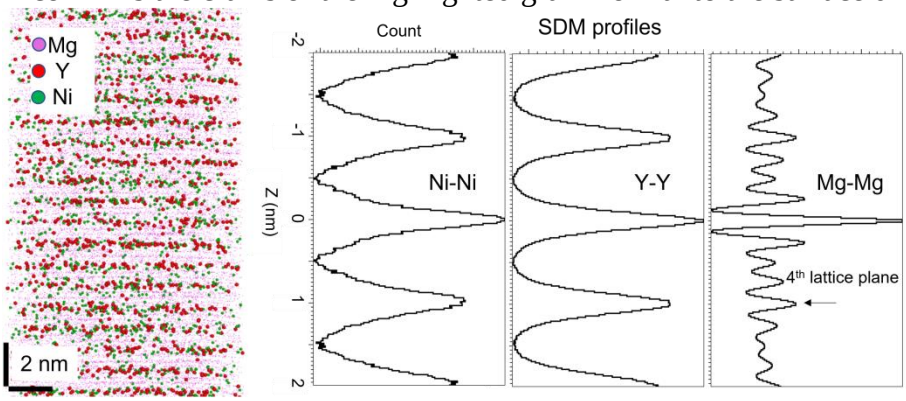

Figure 2. Atom distribution maps and SDMs creat ed along the (0001) pole of LPSO-MgNiY showing synchronized co-appearance of Y and Ni that occurs at every 4th layer of the Mg lattice.

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