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Quantitative Atom Probe Tomography of Magnesium Alloys

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Research interest in age hardenable magnesium alloys has been recently revived because of increasing demand of high strength wrought magnesium alloys for weight reduction of transportation vehicles. The age-hardening response of Mg alloys can be promoted by the trace additions of ternary elements and the two-step aging (e.g. [1]), therefore the demand of the quantitative analysis of alloying elements and their clusters is increasing. In the Mg-Zn and Mg-Zn-Al systems, the formation of Guinier-Preston (G.P.) zones was confirmed, but their detailed chemistry has not been known. Atom probe tomography is an ideal experimental technique to detect solute clusters in alloys, but surprisingly little work has been done on magnesium alloys. This is because the field ion microscopy of magnesium alloys has been thought to be difficult, because of the low evaporation field and the difficulty of oxide-free specimen preparation. We succeeded 3DAP analysis of Mg-RE-Zn alloy [2,3] using voltage-pulse atom probe in 2002, and since then the technique has been applied to Mg-Ca-Zn[4], Mg-Gd-Zn[5,6], Mg-Zn-Al[7], Mg-Zn-Ag-Ca[8] with voltage pulse or laser assisted 3DAP. In this work, we discuss the quantitative analysis of various magnesium alloys using voltage pulse and laser pulse 3D atom probes.

Figure 1 shows the laser assisted atom probe tomography of the GP zones observed in pre-aged Mg-Zn-Al alloy. Although the GP zones in Mg-Zn systems was documented as platelet on the basal plane, this atom probe tomography clearly shows that the GP zones in the Mg-Zn(-Al) systems are spherical clusters. The composition directly estimated from the atom probe data was Mg-16at%Zn-3at%Al, but the composition of the zones are believed to be underestimated due to the convolution effect by the evaporation aberration.

Figure 2 shows the laser assisted atom probe tomography of Mg-1Gd-0.4Zn-0.17Zr alloys containing plate-like precipitates on the basal plane of the Mg matrix. Although (0002) atomic planes are resolved in the magnesium matrix, it is lost near the precipitates. This is because of the preferential retention of Gd atoms because of its higher evaporation field. The concentration of Gd, Zn in the platelets was estimated to be Mg-15at%Gd-15at%Zn. Since the loss of the atomic resolution is inevitable the near the precipitates, complementary HAADF observation is rather useful to determine the thickness of the precipitates.

Figure 3 shows the laser assisted atom probe of Mg-2.4Zn-2Li-0.16Zr alloy having rodlike precipitates along the [0001] axis of the Mg matrix. Systematic TEM analyses revealed that Li additions to Mg-Zn alloy lead to refinement of precipitates. But the role of Li in the microstructure changes is unclear because Li is difficult to quantify with TEM-EDS analysis. The atom probe tomography shows rodlike precipitates have a high concentration of Zn and Li. The concentration of Li enriched in the precipitates was about 8~12 at%.

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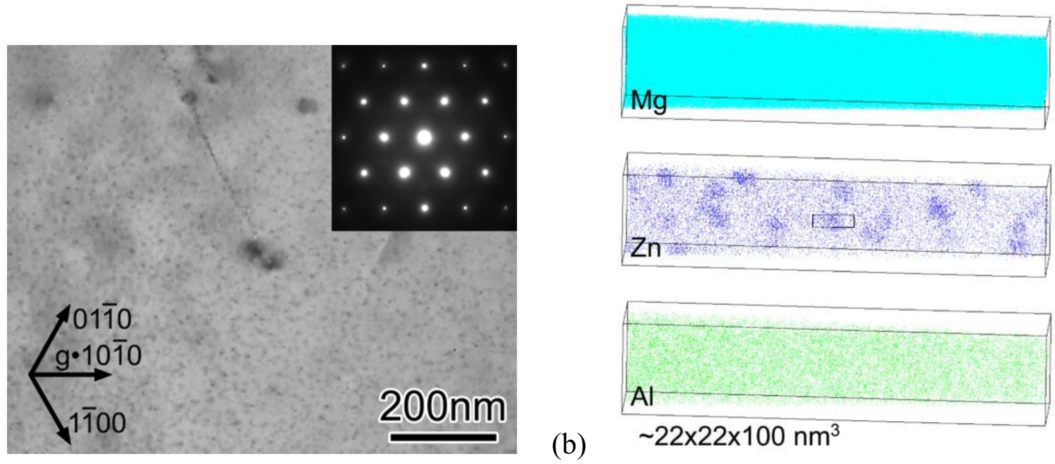
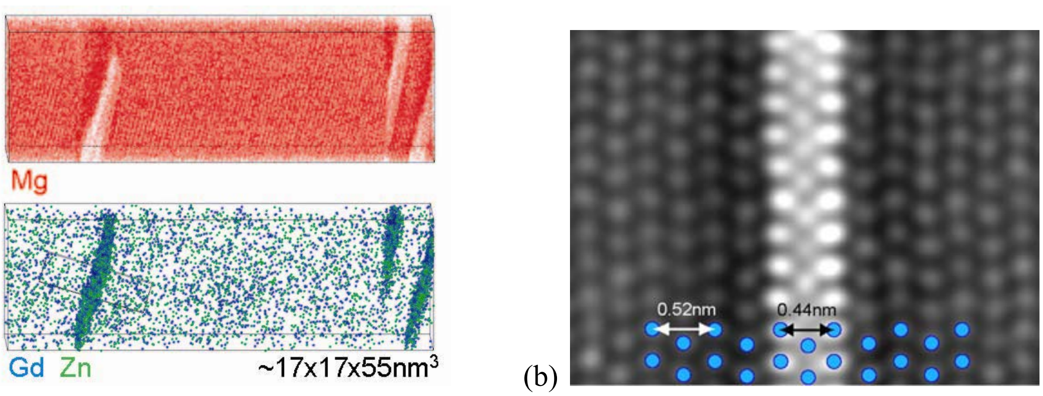


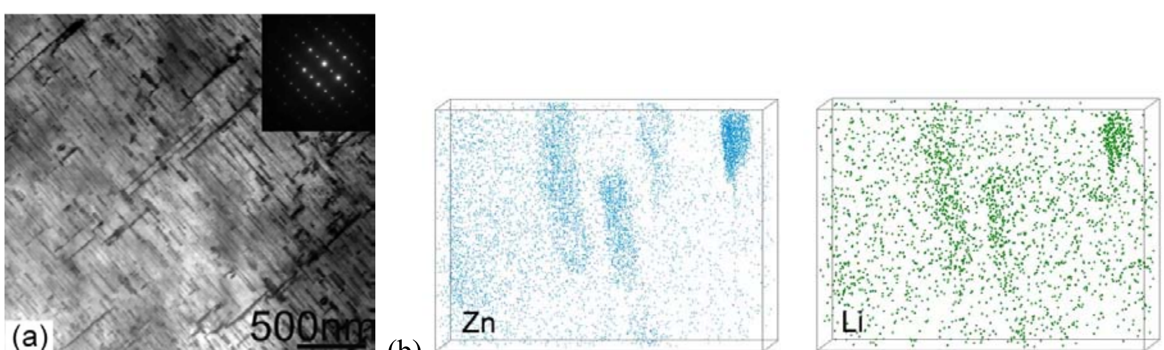
(a) (b)

Fig. 1 (a) TEM image for Mg-Zn-Al alloy aged at 70 ºC for 48 h taken from the [0001] zone axis. (b) 3DAP atom maps of Mg, Zn and Al for the Mg-Zn-Al alloy pre-aged at 70 ºC for 48 h.



(a) (b)

Fig.2 (a) Atom maps of Mg, Gd and Zn in Mg-Gd-Zn alloy aged at 200 ºC for 8 h. (b) High resolution HAADF image of Mg-Gd alloy aged at 200 ºC for 2 h taken from the [11-20].



(b)

Fig. 3 (a) TEM image of Mg-Zn-Li alloy peak-aged at 160 ºC. (b) Atom maps of Zn and Li of the Mg-Zn-Li alloy (analyzed volume: ~16 x 16 x 22 nm^3).

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