Applications of HAADF-STEM and EDS-STEM techniques in Mg-Sn and Mg-Sn-Zn alloy

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Mg-Sn based alloys have received considerable attention in recent years due to their potential to develop high strength wrought magnesium alloys. These alloys are precipitation hardenable as the solid solubility of Sn decreases dramatically with the decrease in temperature. The precipitation process has been traditionally accepted as involving the exclusive formation of the equilibrium β-Mg_2Sn phase; any metastable phases have not been reported so far in those alloys. Considering that the formation of metastable phases is very important in the whole precipitation process, it is necessary to systematically investigate whether there is any metastable phase forming during ageing, especially at the early stage of ageing. In addition, β precipitates, as the main strengthening phase, have a coarse distribution in binary alloys, which results in a poor age-hardening response of the binary alloys. Additions of Zn can remarkably improve the age-hardening response of binary alloys by refining the distribution of β precipitates, but its role remains unclear.

Therefore, in our studies, we used atomic resolution high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) imaging and energy dispersive X-ray spectrometry (EDS) STEM spectroscopy techniques to systematically characterize the precipitates formed in aged samples of Mg-9.8Sn (wt.%) [1] and Mg-9.8Sn-1.2Zn alloys [2]. HAADF-STEM imaging is ideal to study precipitates in the binary alloy as it can clearly show the distribution of Sn-rich particles in the magnesium matrix because the brightness of HAADF-STEM images is proportional to the square of atomic number (Mg: 12, Sn: 50). With this technique, we found the formation of GP zone and a metastable Mg_3Sn phase designated β' prior to the equilibrium β phase. Fig. 1 shows their HAADF-STEM images. However, in the ternary alloy, it is difficult for HAADF-STEM imaging to distinguish the distributions of Sn and Zn since both of them have larger atomic numbers than Mg (Zn: 30). The EDS-STEM technique can solve this problem in this situation. Figs. 2a-c show the HAADF-STEM images of three different β precipitates in the aged ternary alloy. Based on these images, it is difficult to distinguish Sn and Zn distributions in and/or surrounding these precipitates. With the aid of EDS-STEM mapping (Figs. 2d-i), we found that Zn atoms invariably segregate to the interphase boundaries between β precipitates and Mg matrix, irrespective of the interfacial structures and orientation relationships of the β precipitates. Fig. 3 shows an atomic resolution HAADF-STEM image and EDS-STEM maps of a β precipitate. At this scale, the EDS-STEM technique exhibits its unique advantage, clearly showing both composition and structure information of the precipitate simultaneously. By applying these two techniques, we found two metastable phases in the binary Mg-Sn alloy and Zn segregation in the ternary Mg-Sn-Zn alloy, which sheds light on the precipitation sequence in Mg-Sn based alloys and provides insight to the understanding of the enhanced nucleation and thermal stability of β precipitates in the Zn-containing Mg-Sn alloys.

[1] C.Q. Liu, H.W. Chen, H. Liu, X.J. Zhao, J.F. Nie, Acta Mater., 144 (2018) 590-600.
[2] C.Q. Liu, H.W. Chen, J.F. Nie, Scripta Mater., 123 (2016) 5-8.

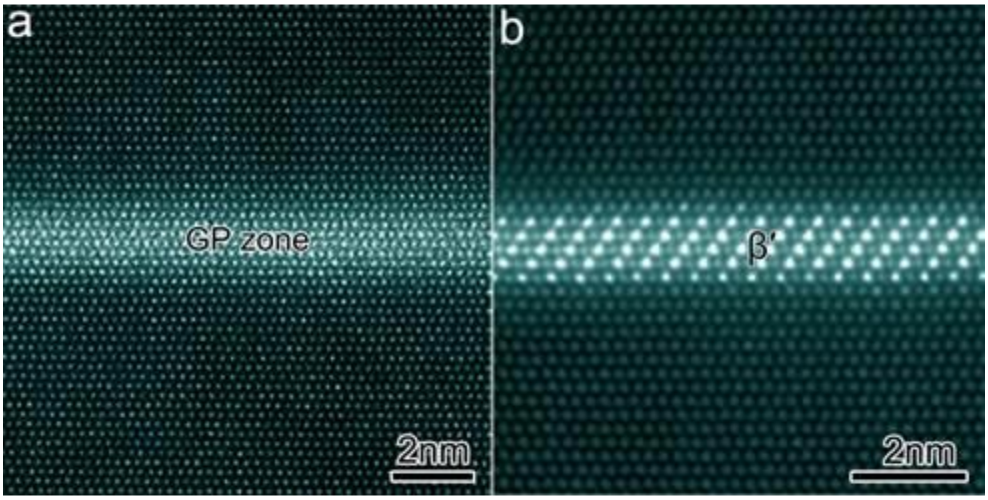

Fig. 1. HAADF-STEM images showing (a) a GP zone and (b) a β' precipitate in Mg-9.8Sn alloy aged at 100 °C for 2056 h. Electron beam is parallel to $[2\bar{1}\bar{1}0]_{Mg}$.

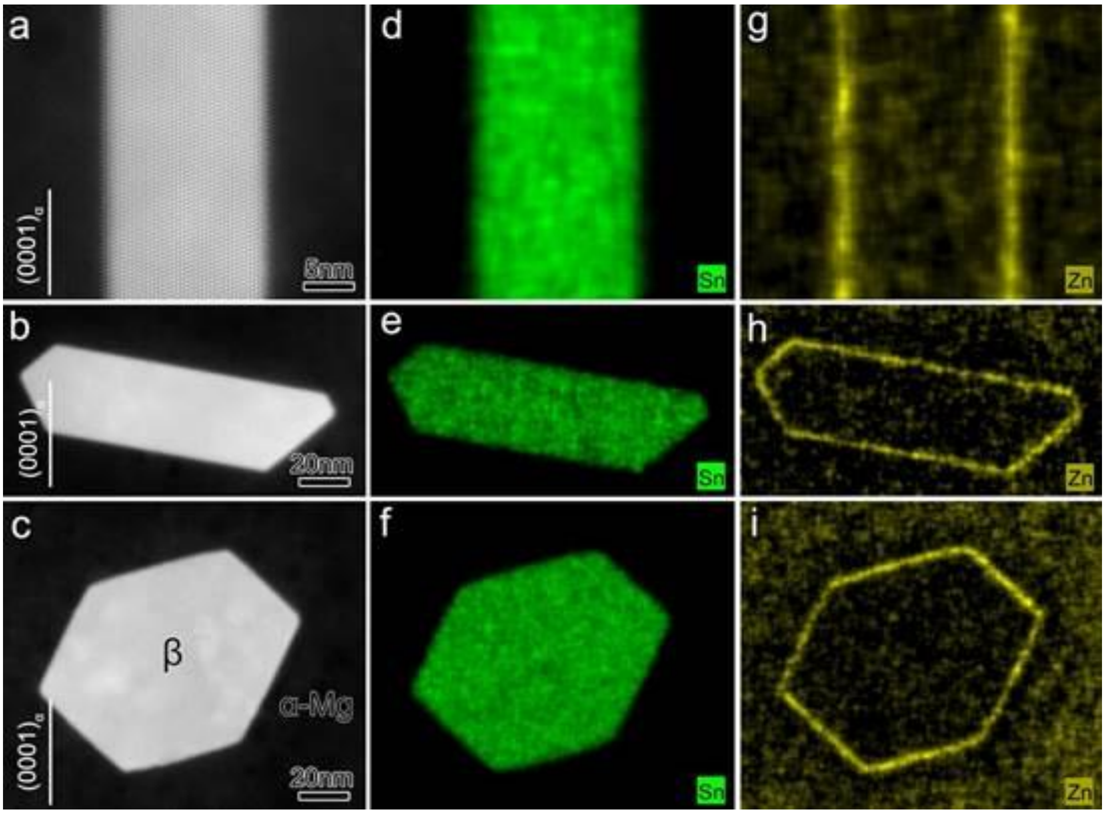

Fig. 2. (a-c) HAADF-STEM images showing three β precipitates with different morphologies and orientation relationships with the Mg matrix in Mg-9.8Sn-1.2Zn alloy aged at 200 °C for 88 h. (d-i) Corresponding EDS maps. (d-f) Sn maps and (g-i) Zn maps. Electron beam is parallel to $[2\bar{1}\bar{1}0]_{Mg}$.

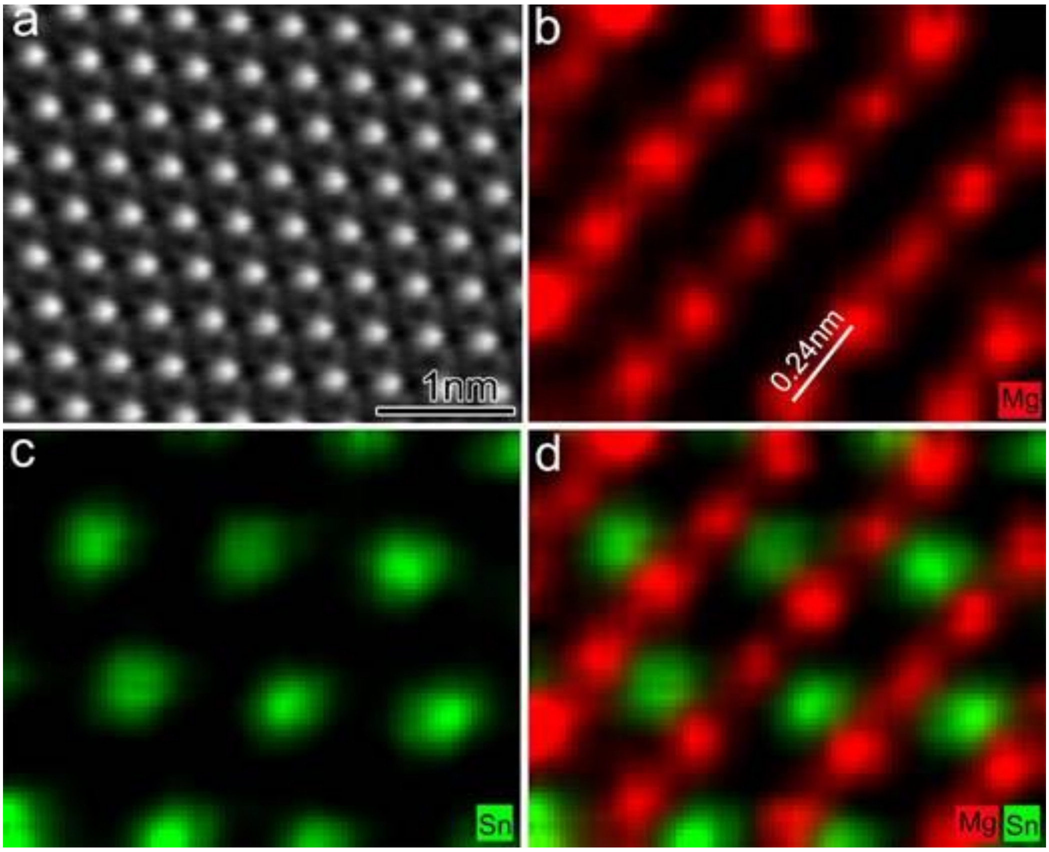

Fig. 3. (a) HAADF-STEM image showing atomic configuration of a β precipitate in Mg-9.8Sn-1.2Zn alloy aged at 200 °C for 88 h. (b-d) Atomic resolution EDS maps of the β precipitate. (b) Mg map, (c) Sn map and (d) combination of Mg and Sn maps. Electron beam is parallel to $[110]_{\beta}$.