

Microstructure Characterization of Microalloyed 5xxx Aluminum Alloys with Er and Zr using Analytical Transmission Electron Microscopy and Synchrotron X-ray Fluorescence Microscopy

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5xxx aluminum alloys with the major alloying element of Mg, have been widely used as structural materials in all kinds of fields, due to their excellent strength, corrosion resistance, formability and weldability. As non-heat treatable alloys, their strength is derived mainly from the work hardening and the solid solution strengthening of Mg which has a substantial solid solubility in Al matrix. However, high Mg concentration in 5xxx alloys will cause processing challenges and increase of the susceptibility to stress corrosion cracking. It is well known that microalloying, namely addition of elements in trace amount, is an effective way to improve the comprehensive properties of aluminum alloys [1]. Recently, it has been proved that joint adding of rare earth element Er and transition element Zr into Al alloys can enhance the mechanical behaviors, thermal stability and corrosion resistance comprehensive significantly due to the formation of thermal stable $L1_2$ -ordered-structured $Al_3(Er, Zr)$ precipitates [2-4].

The work reported here focuses on a newly registered 5xxx alloy in China, named 5E61, with a nominal composition of Al-6Mg-0.9Mn-0.07Zr-0.2Er (wt.%), which has a relatively high Mg concentration and microalloyed with Er and Zr. The microstructure and trace element distributions of this alloy have been characterized using an analytical electron microscope (AEM) equipped with x-ray energy dispersive spectroscopy (XEDS) and synchrotron-based X-ray fluorescence microscopy (XFM) at the Advanced Photon Source (APS) of Argonne National Laboratory.

The experimental alloy was prepared by traditional casting in an iron mold and homogenized at 280°C for 10h and then 460°C for 36h. After the homogenization, the ingot was hot-rolled to 8mm thick (~70% rolling reduction) at 460°C, followed by an intermediate annealing at 350°C for 2h. Subsequently, the sheet was cold-rolled down to 4mm thick (~50% rolling reduction) followed by annealing at 260 °C for 4 h. Thin foils for TEM analysis were prepared by electropolishing. AEM measurements were carried out on an FEI Talos 200kV S/TEM equipped with a SuperX quad spectrometer system. The X-ray induced X-ray fluorescence results were obtained at the Bionanoprobe and 2-ID-D microprobe at the APS.

Figure 1 presents the AEM XEDS color coded hyperspectral elemental imaging, as well as the annular dark field image from the corresponding area. Nanosized Er, Zr-enriched particles were observed *the first time* in these alloys (as arrowed), which are likely $L1_2$ -ordered-structured $Al_3(Er, Zr)$ precipitates as seen in Al-Er-Zr alloy [4]. Micron and submicron Er-enriched precipitates were also observed, and are presumably the phase produced during the casting due to the very limited solid solubility of Er in Al. Mg-enriched β phases were also present as well as the rod-shaped Mn-enriched phases, which are likely Al_6Mn primary phase, elongated along the rolling direction. Elemental distributions were also acquired using XFM from both the thin area measured on the AEM and a thicker region of the sample, with the later shown in Figure 2. Both Mn-enriched and Er-enriched phases, again are very likely the primary

phases from the casting, and were observed ranging from a couple hundred nanometers to a few microns in size. While AEM offers unprecedented resolution to study micron to sub-nanoscale precipitates, the synchrotron-based XRM, without bremsstrahlung background, can in some cases provide a higher elemental sensitivity as well as survey larger and thicker regions of the sample, albeit at lower resolution. By combining the complimentary information obtained from the two techniques, we aim to understand the sample across multi scales. Further quantitative analysis and correlation are undergoing and will be reported separately.

References

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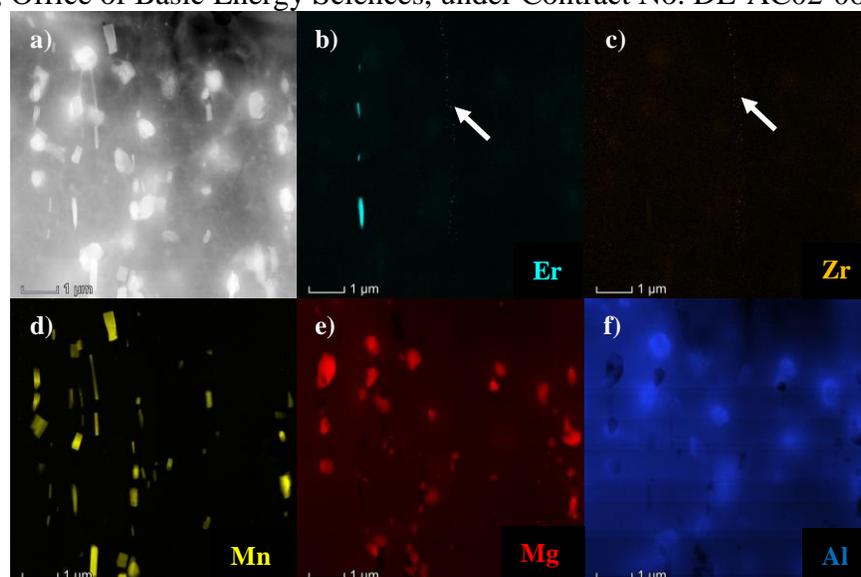


Figure 1. AEM results from the 5E61 alloy. a) HAADF image; hyperspectral images of b) Er; c) Zr; d) Mn; e)Mg and f) Al. Conditions: Eo: 200 keV , 4 nm pixel size, 3 ms dwell time per pixel

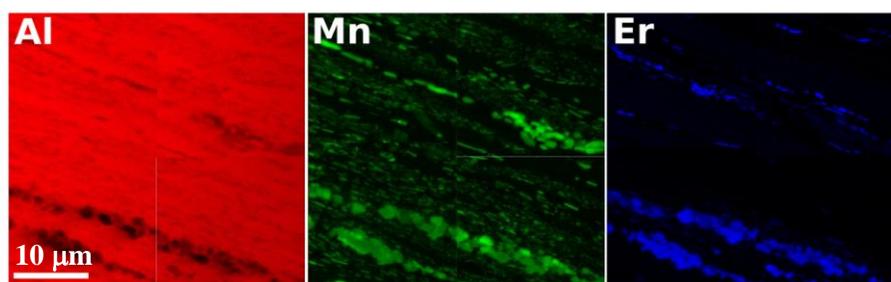


Figure 2. XRF results collected at the ANL Bionanoprobe with 10 keV incident photons, which is not enough energy to be tuned to measure Zr, 80 nm scanning pixel size, 100 ms dwell time per pixel.