

## ABSTRACT

The primary aim of the present study is to develop lean Mg alloys for elevated temperature automobile applications. Towards this, the microstructure evolution and its implications on the room temperature mechanical properties and elevated temperature creep properties were studied in lean Mg-Zn-Al and lean Mg-Al-Ca-Mn alloys, in as-cast, thermomechanically and thermally treated conditions. Since the as-cast Mg-3Zn-1Al alloy exhibits poor room temperature mechanical properties, it was subjected to hard plate hot forging (HPHF) at different temperatures to modify the microstructure through dynamic recrystallization (DRX). The DRX fraction increases from  $22\pm6\%$  to  $73\pm5\%$  as the temperature is raised from 523 K to 623 K, reducing the fraction of deformed grains and subsequently, the yield strength (YS  $\sim 232\pm4$  MPa to  $\sim 168\pm2$  MPa). However, the basal texture is weakened due to the random orientations of the DRX grains and consequently, the ductility is improved in the specimen forged at 623 K (uniform elongation, UEl  $\sim 10.7\pm1.2\%$ ). At 723 K, grain growth is initiated which increases the grain size and re-strengthens the basal texture, resulting in decrease of both YS ( $151\pm7$  MPa) and ductility (UEl  $\sim 7.1\pm1.1\%$ ) of the corresponding specimen. Solution annealing results in significant grain growth, second phase dissolution and texture weakening, which decreases the YS, but improves the ductility significantly, in all the HPHF specimens. In order to optimize the Mg-Al-Ca-Mn alloy composition, the Ca/Al ratio was varied within  $\sim 0.06$ -2.21, which is observed to not only dictate the second phase evolution, but also control the solid solution environment in the as-cast alloys. Consequently, despite having coarser grains ( $354\pm43$   $\mu\text{m}$ ), the as-cast Mg-0.9Al-0.6Mn-0.1Ca alloy exhibits higher strength (YS  $\sim 130\pm5$  MPa), owing to greater contribution from solid solution strengthening as well as precipitation strengthening from the  $\text{Mg}_{17}\text{Al}_{12}$  phase. The mechanical properties deteriorate in the alloys with higher Ca/Al ratio owing to the strong connected networks of hard and brittle  $\text{Mg}_2\text{Ca}$  phase, which promote initiation and propagation of cracks. Post HPHF, the grain size is significantly reduced due to DRX, while typical basal fiber textures are developed in the Mg-Al-Ca-Mn alloys. The Mg-1.3Ca-0.8Al-0.4Mn alloy exhibits maximum YS ( $\sim 167\pm3$  MPa) predominantly due to the larger strengthening contributions from its finer grains and precipitates. However, despite having weaker basal texture, this specimen displays poor ductility (UEl  $\sim 2.7\pm1.0\%$ ) owing to the networks of coarse  $\text{Mg}_2\text{Ca}$  particles. The Mg-1Al-0.28Ca-0.13Mn alloy, which shows optimum combinations of strength and ductility in both as-cast (YS  $\sim 90\pm7$  MPa and UEl  $\sim 4.8\pm1.6\%$ ) and HPHF conditions ( $\sim 132\pm12$  MPa and UEl  $\sim 5.0\pm1.0\%$ ), was selected for thermal treatments to attain further strengthening. The aging treatment of the cast Mg-1Al-0.28Ca-0.13Mn alloy led to the formation of fine  $\text{Al}_2\text{Ca}$  precipitates from pre-existing  $(\text{Mg},\text{Al})_2\text{Ca}$  phases, along with evolution of new  $(\text{Mg},\text{Al})_2\text{Ca}$  particles. Simultaneously, the size, area fraction and interfacial lattice misfit of the nano-sized  $\text{Al}_8\text{Mn}_5$  precipitates increase with aging duration. These precipitates, depending on their coherency with the matrix, are either sheared off or bypassed by the dislocations, which predominantly dictate the strength in these specimens. Thus, the relatively smaller size ( $\sim 16\pm4$  nm), higher area fraction ( $\sim 2.4\pm0.9\%$ ), and larger misfit ( $\sim 14\pm9\%$ )



of the semi-coherent  $\text{Al}_8\text{Mn}_5$  precipitates result in higher strengthening (YS  $\sim 133 \pm 5$  MPa and UTS  $\sim 190 \pm 9$  MPa) in the peak-aged specimen. Contrarily, the coarser  $(\text{Mg},\text{Al})_2\text{Ca}$  particles act as crack-initiating sites and deteriorate the ductility during the later stages of aging. The HPHF specimen of the Mg-1Al-0.28Ca-0.13Mn alloy was subjected to aging treatment directly as well as after an intermediate solution-annealing treatment. While evolution of very fine  $\text{Al}_8\text{Mn}_5$  precipitates takes place during direct peak-aging, a large proportion of the fine precipitates dissolves during the solution annealing treatment after HPHF. However, due to the formation of new precipitates, the area fraction of the fine  $\text{Al}_8\text{Mn}_5$  precipitates increases during peak-aging of the ‘HPHF+solution-annealed’ specimen. The fine DRX grains (originated during HPHF) and the finer  $\text{Al}_8\text{Mn}_5$  precipitates result in significant strengthening (YS  $\sim 162 \pm 14$  MPa) in the directly peak-aged HPHF specimen. In the ‘HPHF+solution-annealed’ specimen, the strength is considerably decreased ( $\sim 88 \pm 2$  MPa) owing to grain growth and dissolution of precipitates, but the ductility is significantly improved ( $11.8 \pm 1.7\%$ ) due to texture weakening. During peak aging of this specimen, strength is further improved ( $\sim 105 \pm 1$  MPa) owing to the relatively higher fraction of the nano-sized  $\text{Al}_8\text{Mn}_5$  precipitates. Few specimens from both the Mg-Zn-Al and Mg-Al-Ca-Mn systems, selected on the basis of the microstructure and room temperature tensile properties, were subjected to compressive creep tests at 448 K under 50 MPa stress. The grain size is observed to play a dominant role in dictating the creep performance in the Mg-3Zn-1Al alloy. Thus, the solution-annealed specimens having larger grain size exhibit lower steady state strain rate (SSSR) than the HPHF specimens. Similarly, the as-cast, ‘HPHF+solution-annealed’ and ‘HPHF+solution-annealed+peak-aged’ specimens of the Mg-1Al-0.28Ca-0.13Mn alloy exhibit better creep performance due to the presence of much coarser grains. Importantly, both the as-cast and the ‘HPHF+solution-annealed’ specimens exhibit relatively better creep performance than their respective peak-aged counterparts, due to dynamic evolution of fine  $\text{Al}_8\text{Mn}_5$  precipitates during creep.

**Keywords:** Aging; Casting; Creep; Dislocations; Dynamic recrystallization; Grain growth; Grain size; Hard plate hot forging; Lean Mg-Al-Ca-Mn alloys; Lean Mg-Zn-Al alloy; Microstructure; Precipitates; Solid solution; Solution annealing; Tensile properties; Texture; Twins; Work hardening.