Abstract

Magnesium (Mg) is the lightest structural metallic material with high specific strength, good damping capacity, biodegradability, and biocompatibility. Therefore, it is a potential candidate for structural automobile, aerospace, and space-restricted bio-implant applications. However, their applications are limited due to poor ambient temperature strength, ductility, formability, fracture, and corrosion resistance. The low strength, ductility, and formability are attributed to the hcp crystal structure with near-ideal c/a ratio. The deformation is carried out by only two independent basal slip systems having low critical resolve shear stress (CRSS) at ambient temperature. A minimum of five independent slip systems are required for homogeneous plastic deformation to avoid failure in a polycrystal. The non-basal slips have comparatively very high CRSS and are difficult to activate under ambient conditions. The $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$-extension twin (ET) has a slightly higher CRSS than basal slip and can accommodate ambient temperature deformation; however, early fracture occurs upon saturation of ET. Other twinning modes are $\{10\bar{1}1\}\langle 10\bar{1}2\rangle$-contraction twins (CT), and $\{10\bar{1}2\}$-$\{10\bar{1}1\}$ double twin (DT) may also form during deformation.

Mg could be formable at $> 200℃$ through plastic deformation by basal, prismatic, pyramidal $\langle a\rangle$, and pyramidal $\langle c+a\rangle$ I and II slip systems. However, due to the significant difference in CRSS ratio between basal and non-basal slips, wrought forming produces a sharp basal texture, leading to high plastic anisotropy. Researchers are ambitious to improve the mechanical properties by various alloying additions that activate non-basal slips. Such alloying addition alters the microstructural factors, such as grain size and texture, and also forms precipitates. This enhances the slip and suppresses the twin activity, improving plasticity. Alloying with rare earth (RE) elements has been proven effective in improving strength and ductility by grain refinement, precipitation hardening, increasing non-basal slip activity, and texture weakening. Cerium (Ce) is considered a cost-effective rare earth (RE) alloy addition in Mg to enhance strength and ductility by solid solution, precipitation strengthening, and texture weakening. However, adding $\geq 0.5wt\%$ Ce degrades the surface finish and ductility drastically. The addition of $\sim 3wt\%$Al to Mg-0.5$wt\%$Ce forms $Al_{11}Ce_3$ intermetallic instead of $Mg_{12}Ce$, and restores the ductility without compromising strength. The Mg-3$wt\%$Al-0.5$wt\%$Ce alloy has been considered one of the potential structural load-bearing materials. However, it is essential to investigate the deformation and fracture behavior, including their anisotropy, to implement the alloy for applications. This PhD thesis examines the uniaxial

tensile, compressive, fracture toughness (FT), and fatigue crack growth rate (FCGR) behavior and their anisotropy in correlation with microstructure and texture of the hot-rolled Mg-3Al-0.5Ce alloy. The first three chapters discuss the 'Introduction to the fundamentals aspect of this research work, 'The literature review with the existing lacunae and motivation of the work', 'Objectives of the work', and 'Material and methods'. After that, the 'Results and Discussion' chapters are presented.

In this work, as-cast Mg-3Al-0.5Ce alloy having heterogeneous coarse grains with dendritic structure, uneven distribution of $\mathrm{Al_{11}Ce_3}$ precipitates and random texture were homogenized and hot-rolled at $350°C$. After the hot-rolling, coarse and fine equiaxed and elongated grains along the rolling direction (RD) with an average grain size of ~ 24μm were observed. The precipitates were distributed within the elongated grain boundaries, i.e., along RD. The basal texture was observed in the rolling plane, i.e., c-axis ǁ to normal direction (ND). The $\{10\bar{1}0\}$ poles were oriented along RD and $\{11\bar{2}0\}$ along the transverse direction (TD) with higher multiples of random distribution (MRD) due to dynamic recrystallization (DRX) during hot-rolling. The hot-rolled microstructure and texture influenced the uniaxial tensile, compressive, FT, and FCGR behavior. In Chapter 4, the anisotropic uniaxial tensile behavior of the hot-rolled Mg-3Al-0.5Ce alloy was studied. The biaxial deformation behavior has been theoretically analyzed, considering the texture and uniaxial tensile properties of the hot-rolled alloy. Chapter 5 investigates the anisotropy in uniaxial compressive deformation. Analytical and crystal plasticity-based visco-plastic self-consistent predominant twin reorientation (VPSC-PTR) approaches were employed to simulate the flow curve and texture evolution for deeper insight into the deformation behavior. Chapter 6 examines the anisotropic FT behavior of hot-rolled Mg-3Al-0.5Ce alloy. It was explored that the presence of $\mathrm{Al_{11}Ce_3}$ precipitates alter the crack tip stress state and thereby $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ET lamellae formation w.r.t. the notch, leading to FT anisotropy. Therefore, The FT test of hot-rolled pure Mg was also carried out to examine the FT anisotropy in the absence of precipitates. Chapter 7 presents the anisotropic FCGR behavior of hot-rolled Mg-3Al-0.5Ce alloy. Finally, chapter 8 gives the conclusions and summarizes the thesis.

It was revealed that anisotropy in uniaxial tension and compression behavior was mainly related to texture. The effect of $\mathrm{Al_{11}Ce_3}$ precipitates were to delay the nucleation events of $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ET during compression along RD and TD. However, FT and FCGR anisotropy was affected by the long alignment of $\mathrm{Al_{11}Ce_3}$ precipitates along RD, altering the crack-tip stress state. For pure Mg, FT anisotropy was related to initial texture.

In Chapter 4, tensile anisotropy was observed with higher strength along TD ($\sigma_{YS} = 167MPa$, $\sigma_{TS} = 210MPa$) compared to RD ($\sigma_{YS} = 138MPa, \sigma_{TS} = 180MPa$). Ductility along RD and TD were ~0.18 and ~0.13. This could be due to a higher average Taylor factor (M) for tensile along TD ($M = 6.36$) than RD ($M = 5.45$). A high normal anisotropy ($\bar{r} = 7.17$) and low planar anisotropy ($\Delta r = 0.13$) was obtained by crystal plasticity and validated by tensile tests' results. Due to high $\bar{r}$, remarkable biaxial strength and ductility could be obtained in hydraulic bulging ($\sigma_1,\ \sigma_2 = 475MPa,\ 475MPa$ and $\varepsilon_1,\ \varepsilon_2 = 0.66, 0.66$) and plane strain ($\sigma_1,\ \sigma_1 = 338MPa, 169MPa$ and $\varepsilon_1,\ \varepsilon_2 = 0.93, 0$) conditions on the onset of diffused necking, provided sufficient slip systems are available.

Compressive deformation anisotropy along RD, TD, and ND was observed to be mainly due to texture in Chapter 5. The anisotropy was less along RD and TD as the texture favored c-axis extensions. A significantly different deformation behavior was observed along ND due to c-axis contraction. The flow behaviors for compression directions (CD) ∥ to RD and TD were sigmoidal, exhibiting increasing stage-II strain hardening rate (SHR) due to the texture with c-axis ~ ⊥ to CD, favoring ET formation and geometrical hardening. For CD ∥ to ND, parabolic flow behavior with decreasing SHR was due to slip-based deformation, majorly by basal $\langle a \rangle$, and pyr $\langle c + a \rangle$-I and II slips. For CD ∥ to ND, due to geometrically hard orientation, yield strength (σ_{ys}) was higher ($\sim 205MPa$) than for CD ∥ to RD ($\sim 74MPa$) and TD ($\sim 76MPa$). The ultimate compressive strength (σ_c) was higher for CD ∥ to RD ($\sim 275MPa$) and TD ($290MPa$) than for CD ∥ to ND ($\sim 248MPa$). The insignificant anisotropy along RD and TD could be due to a major $\{10\bar{1}0\}$ poles oriented along RD and $\{11\bar{2}0\}$ along the TD.

In Chapter 6, the FT anisotropy in hot-rolled Mg-3Al-0.5Ce alloy was observed with elastic-plastic FT (J_{IC}) of $\sim 20.7N/mm$ and $\sim 15.8N/mm$ for a_n ∥ to RD and TD, respectively. The plastic zone ahead of the crack tip encountered triaxial in-plane and out-of-plane tensile stresses. The presence of long-aligned $Al_{11}Ce_3$ precipitates within the elongated grain boundaries along RD generated back-stresses in this zone. For a_n ∥ to RD and TD, the in-plane stress ~ ∥ and ~ ⊥ to a_n reduced, respectively. Thereby activating profuse lenticular $\{10\bar{1}2\}\langle 10\bar{1}1 \rangle$ ET ~ ⊥ and ~ ∥ and c-axis ~ ∥ and ~ ⊥ to crack path, leading to trans and inter lamellar crack and anisotropic J_{IC} for a_n ∥ to RD and TD, respectively. Translamellar crack in a_n ∥ RD could blunt the crack tip by facing considerable deflections, leading to plastic energy dissipation. ET-matrix interface is a region of strain incompatibility and potent crack nucleation sites; therefore, interlamellar crack in case of a_n ∥ TD led to less plastic energy dissipation.

For hot-rolled pure Mg, the basal texture with $\{10\bar{1}0\}$ poles mostly along RD and $\{11\bar{2}0\}$ along the TD was observed. The out-of-plane tensile stresses were found to be responsible for the activation of $\{10\bar{1}2\}\langle10\bar{1}1\rangle$ET, i.e., Σ15b coincidence site lattice boundary (CSLB). While the tensile stress $\perp$ to crack-tip activated $\{10\bar{1}1\}\langle10\bar{1}2\rangle$CT that transforms into double twins $\{10\bar{1}1\}$-$\{10\bar{1}2\}$- DT_a, and DT_b with Σ23b and Σ15a CSLB, respectively. The anisotropic J_{IC} could be due to variation in the ET and DT lamellae formation w.r.t to notch due to initial texture. The J_{IC} and the crack-tip plastic zone decreases, while the elastic component of J-integral (J_{el}) and the ET formation increases from a_n ∥, $\perp$, to $\sim 45°$ to RD. The strain incompatibility between matrix-DT Σ23b interfaces is lesser than the matrix-ET Σ15b interfaces. In case of a_n ∥ RD, where the ET activation was meager, the crack propagation via the matrix-DT Σ23b interfaces led to considerable plasticity and higher J_{IC}. While crack propagation through Σ15b and Σ23b interfaces in a_n $\perp$ RD led to lower J_{IC}. The crack growth only through Σ15b led to the lowest plasticity and, thereby, the least J_{IC}.

Chapter 7 demonstrates that during the FCGR cyclic loading of hot-rolled Mg-3Al-0.5Ce alloy also, the presence of long-aligned $Al_{11}Ce_3$ precipitates along RD generated backstress in the crack tip region. Therefore, the crack-tip stress state was modified in a manner similar to during the FT test, leading to ET formation ~ $\perp$ and ~ ∥ to crack path for a_n ∥ RD and TD, respectively. This led to trans and inter lamellar crack with lower and higher FCGR for a_n ∥ RD and TD, respectively. The fracture surface for all the testing conditions was cleavage-type with elongated grooves. For a_n ∥ RD elongated grooves deviated from the crack direction, indicating several crack arrests. For a_n ∥ TD, large elongated grooves were observed, indicating easy crack propagation. For all the testing conditions, the linear load-displacement curve during each cyclic loading indicates that crack closure didn't occur. Thus, ΔK_{eff} were equal to the ΔK in all the conditions.