

PREFACE

Al-Mg-Zn (7075 Al alloy) alloy, known for its age-hardening properties, offers several advantages, such as its low weight, impressive specific strength, excellent resilience, ease of shaping, and cost-effectiveness. Aluminum 7075 alloy demonstrates an impressive strength-to-weight ratio, making it a popular choice for construction materials and aerospace applications, including wings and fuselage components. Its exceptional strength and lightweight characteristics also find applications in various other fields, such as rock-climbing gear and bicycle parts. As per the consensus of numerous experts, it finds extensive use in industries such as aerospace, aviation, transportation, and others that require lightweight, high-strength structural components with excellent resistance to corrosion. However, their application is impeded by their limited cold-forming capabilities and the intricate and costly hot-forming process. Currently, Al-Mg-Zn alloy sheets are utilizing in aircraft applications as thick plates without requiring complex forming procedures. Therefore, it is crucial to enhance their cold-rolling capacity and mechanical properties to fully unlock their potential for improving safety, reducing weight, increasing fuel efficiency, and expanding the utilization of Al-Mg-Zn based alloys. Cold rolling of 7075 Al alloy is very challenging due to its high hardness value among all Al alloys. The traditional 7075 aluminum alloy sheet underwent hot rolling from 30 mm to 1 mm at 400 °C to prevent fractures, as cold rolling resulted in structural damage, and achieved 90% cold rolling after adding ZnO nanoparticles. Maximum 50 to 60% deformation has achieved after cold rolling in 7075 Al alloy till date. 70% deformation is achieved after cryo-rolling of 7075 Al alloy. But in this paper 90% cold rolling has been achieved successfully with the help of noble heat treatment process without adding any foreign element or compound. Over the years, extensive research has been done on the precipitation mechanisms in 7xxx series Al alloy, as well as the order in which precipitation occurs, have been recognized as SSSS -> GP zones/atomic clusters -> η'' (eta double prime) -> η' (eta prime) -> η (eta), (equilibrium

phase). The η'' metastable phase has the maximum strengthening impact, making it the most sought phase. However, it should be emphasized the simplified order of precipitation ignores the effects of additional phases or changes in chemical composition that could change or accelerate the sequence. Many researchers have studied about η and η' precipitates thoroughly but very less study is found regarding η'' precipitate. These studies are compiled in seven chapters which are given below:

In **Chapter 1**, we offer a concise overview of 7075 Al alloy, elucidating its precipitation, compositions, properties, and application along with fatigue and fracture behavior with a brief literature survey on Al-Mg-Zn alloy and the effect of corrosion on this alloy is also illustrated. Based on the literature survey, the objective of the thesis is outlined and accordingly, the results with discussions and important findings followed by conclusions are presented in subsequent chapters.

In **Chapter 2**, we present the methodology utilized for thermomechanical processing (solution heat treatment +artificial aging + cold rolling). The chapter also addresses characterization techniques that are used for data collection and analysis. X-ray diffraction (XRD), optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and electron backscattered diffraction (EBSD) was employed to assess the microstructure and phase constituents. Elastic plastic (J1c) fracture toughness was studied well on SHT, SHT+PA, SHT+45% CR, SHT+60% WR, and SHT+PA+90% CR samples. Additionally, Vickers hardness and tensile test and corrosion testing in 3.5% NaCl solution were performed.

In **Chapter 3** we investigate the impact of thermomechanical processing route on the microstructural evolution, and mechanical properties (tensile strength, hardness and elastic plastic (J1c) fracture toughness) of the 7075-aluminum alloy. As received 7075 Al Alloy underwent for the solution heat treatment (SHT) followed by artificial aging and cold rolling (CR) process, respectively. Generally, it was observed that cold rolling of 7075 Al alloy is very challenging, but

in this work 90 % cold rolling successfully achieved by optimizing the thermo-mechanical process. The novel heat treatment process for achieving the 90% cold rolling reduction as follow: firstly, SHT was performed at 470°C for 8 hours(h), there after aging at 140°C for 21 h was performed. Characterization techniques like X-ray diffraction (XRD), optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) was employed to assess the microstructure and phase constituents. Elastic plastic (J1c) fracture toughness was studied well on SHT, peak aged and rolled sample. Additionally, Vickers hardness and tensile test were performed. Aging and Cold rolling treatment effectively enhanced tensile strength and hardness ascribed to formation of fine rod shape precipitates of η'' (Mg₂Zn₃) and formation of sub grains with localized strain accumulation, respectively. Split diffraction spots with satellite pattern in long range ordering has also observed in selected area electron diffraction (SAED) pattern of η'' attributable to stacking faults and periodic arrangement of precipitates, respectively, as a consequence of this 90% cold rolling of 7075 Al alloy was accomplished. The maximum Vickers hardness, Tensile strength and Elastic plastic (J1c) fracture toughness values were achieved after SHT (470°C for 8 h), peak aged (PA) (140°C for 21 h) and 90% cold rolling are 226 HV, 526 MPa and 344.54 kJ/m², respectively.

In **Chapter 4**, we explored the fatigue crack growth rate (FCGR) characteristics correlating with microstructure of Solution heat treated (SHT), SHT+ peak aged (PA), SHT+45% cold rolled (CR), SHT+ 60% warm rolled (WR) and SHT+PA+90% CR Al-Mg-Zn alloy. The findings indicate that artificial aging and cold rolling process decelerate fatigue crack initiation because of precipitation hardening and work hardening as well as grain boundary strengthening, respectively. Utilizing the Paris model, fatigue crack growth rates during the linear extension stage were analyzed. Analysis of crack paths via scanning electron microscope (SEM) techniques revealed ductile fracture(dimples) in case of SHT and SHT+60% WR sample, while ductile and brittle mix fracture

(dimples and facets) in case of SHT+PA and SHT+ 45% CR sample, whereas brittle fracture (river pattern, facets) in case of SHT+PA+90% CR sample were observed. The broadening of precipitate peaks in the X-ray diffraction (XRD) graph of SHT+ PA+ 90% CR indicates a rise in dislocation density which is $11.1 \times 10^{14} \text{ m}^{-2}$. The microstructural evolution is characterized using optical -microscopy, EBSD and transmission electron microscopy (TEM) techniques. Rod like shape η " precipitates were observed in TEM images in the case of SHT+PA+90% CR sample. Through the partition of IPF image it was observed that higher volume fraction of recrystallized grains was formed in SHT+60% WR sample, whereas nano-meter to micrometer size sub grains were formed in the case of SHT+PA+90% CR sample. It was observed through orientation distribution function that SHT+45% CR is showing strong brass ($\{110\}\langle 112\rangle$) texture, whereas SHT+60% WR sample is showing strong rotated cube($\{001\}\langle 110\rangle$) texture, while 90% CR sample is showing strong brass($\{110\}\langle 112\rangle$), strong Cu($\{112\}\langle 111\rangle$) and strong S($\{123\}\langle 634\rangle$) texture. Mechanical properties are assessed through tensile, hardness, and fracture tests. The highest values for Vickers hardness (226 HV), tensile strength (526 MPa), and conditional elastic-plastic fracture toughness (JQ) (344.54 kJ/m²) were obtained after SHT (470°C for a duration of 8 hours), PA (140°C for a duration of 21 hours) and 90% CR.

In **Chapter 5** we investigated the elastic plastic J_{1c} fracture toughness of Solution heat treated (SHT), SHT+ peak aged (PA), SHT+45% cold rolled (CR), SHT+ 60% warm rolled (WR) and SHT+PA+90% CR Al-Mg-Zn alloy in 3.5% NaCl solution. Electrochemical testing was conducted using an electrochemical workstation (Gamry Interface 1010 E). Microstructural is investigated with the help of scanning electron microscope (SEM) and transmission electron microscopy (TEM). Mechanical property is investigated by tensile testing, Vickers hardness testing and elastic plastic J_{1c} fracture toughness test. The results indicate that the SHT condition exhibited the highest corrosion resistance due to the dissolution of precipitates; however, it showed poor mechanical

properties. In contrast, the SHT + PA + 90% CR condition was found to be optimal, offering a good balance of mechanical strength and improved corrosion resistance although slightly lower than the SHT and SHT + 45% CR conditions owing to the formation of redistributed, very fine precipitates and enhanced passivation via high dislocation density.

In **Chapter 6** we investigated the fatigue crack growth rate of Al-Mg-Zn alloy under five different conditions: solution heat treated (SHT), SHT + peak aged (PA), SHT + 45% cold rolled (CR), SHT + 60% warm rolled (WR), and SHT + PA + 90% CR, in a 3.5% NaCl solution and compared it to the normal condition. Electrochemical testing was performed using a Gamry Interface 1010E electrochemical workstation. Microstructural characterization was carried out using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Mechanical properties were evaluated through tensile testing, Vickers hardness testing, and elastic-plastic J_{1c} fracture toughness testing. The three regions of fatigue pre crack((i) crack initiation, (ii) crack propagation(iii) unstable crack growth) is investigated very well with the help of Paris law and fractography of the pre-cracked samples. Results shows that ductility of all five conditions samples has decreased due the corrosion effect and in stable crack growth region length of striation as well as gap between them also increased which showing poor fatigue life.

In **Chapter 7**, A comprehensive overview of the undertaken work is provided. Our focus has been on investigating the elastic plastic J_{1c} value and fatigue crack growth rate of Al-Zn-Mg alloy