Fatigue and Fracture behaviour of Al-Mg-Zn Alloy

Thesis submitted in partial fulfillment for the Award of degree

DOCTOR OF PHILOSOPHY

By

Nidhi Chaubey

SCHOOL OF MATERIALS SCIENCE AND TECHNOLOGY
INDIAN INSTITUTE OF TECHNOLOGY
(BANARAS HINDU UNIVERSITY)
VARANASI – 221005
INDIA

Roll No. 19111502

2025

Dedicated to...

My Beloved Parents

(Smt. Asha Chaubey & Shri Jay Prakash Chaubey)

&

Grand Parents

(Lt. Subhadra & Lt. Radhe Shyam Chaubey)

"सर्वं ज्ञानं तस्य लभ्यते य: पुन: विद्या"

सारा ज्ञान उन्हीं को प्राप्त होता है जो ज्ञान का मार्ग खोजते हैं।

" All knowledge is gained by those who seek the path of wisdom."

(Source - Shrimad Bhagwat Geeta, Chapter 4, Sloka 39)

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INDIAN INSTITUTE OF TECHNOLOGY
(BANARAS HINDU UNIVERSITY)
VARANASI - 221005

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It is certified that the work contained in the thesis titled **"Fatigue and Fracture behaviour of Al-Mg-Zn alloy"** by **Ms. Nidhi Chaubey** has been carried out under my supervision and that this work has not been submitted elsewhere for a degree.

It is further certified that the student has fulfilled all the requirements of Comprehensive Examination, Candidacy, and SOTA for the award of Ph.D. Degree.

16/07/2025

Dr. Nikhil Kumar
(Supervisor)
School of Materials Science &Technology
Indian Institute of Technology (BHU),
Varanasi-221005, (U.P.), India

Assistant Professor/सहायक-आचार्य
School of Materials Sc. & Tech./पदार्थ विज्ञान एवं प्रौद्योगिकी स्कूल
Indian Institute of Technology/भारतीय प्रौद्योगिकी संस्थान
(Banaras Hindu University), Varanasi/(का.हि.वि.वि.),वाराणसी

Prof. Chandan Upadhyay
(Coordinator)
School of Materials Science &Technology
Indian Institute of Technology (BHU),
Varanasi-221005, (U.P.), India

Coordinator/समन्वयक
School of Materials Science & Technology/पदार्थ विज्ञान एवं प्रौद्योगिकी स्कूल
Indian Institute of Technology/भारतीय प्रौद्योगिकी संस्थान
(Banaras Hindu University),Varanasi/काशी हिन्दू विश्वविद्यालय, वाराणसी

DECLARATION BY THE CANDIDATE

I, "**Nidhi Chaubey**", certify that the work embodied in this thesis is my own bonafide work and carried out by me under the supervision of "**Dr. Nikhil Kumar**" from December 2019 to July 2025, at the School of Materials Science & Technology, Indian Institute of Technology (Banaras Hindu University), Varanasi. The matter embodied in this thesis has not been submitted for the award of any other degree/diploma. I declare that I have faithfully acknowledged and given credits to the research workers wherever their works have been cited in my work in this thesis. I further declare that I have not willfully copied any others work, paragraphs, text, data, results, etc., reported in journals, books, magazines, reports dissertations, thesis, etc., or available at websites and have not included them in this thesis and have not cited as my own work.

Date: 16/07/2025

Place: Varanasi

Nidhi Chaubey

(Ms. Nidhi Chaubey)

CERTIFICATE BY THE SUPERVISOR

It is certified that the above statement made by the student is correct to the best of my knowledge.

16/07/2025

Dr. Nikhil Kumar
(Supervisor)

Assistant Professor / सहायक-आचार्य
School of Materials Sc. & Tech. /पदार्थ विज्ञान एवं प्रौद्योगिकी स्कूल
Indian Institute of Technology / भारतीय प्रौद्योगिकी संस्थान
(Banaras Hindu University), Varanasi /(का.हि.वि.वि.),वाराणसी

Prof. Chandan Upadhyay
(Coordinator)

Coordinator/समन्वयक
School of Materials Science & Technology/पदार्थ विज्ञान एवं प्रौद्योगिकी स्कूल
Indian Institute of Technology/भारतीय प्रौद्योगिकी संस्थान
(Banaras Hindu University),Varanasi/(बनारस हिन्दू विश्वविद्यालय, वाराणसी

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Name of the Student: Nidhi Chaubey
Ms. Nidhi Chaubey

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Date: 16/07/2025
Place: Varanasi

Nidhi Chaubey
(Ms. Nidhi Chaubey)

ACKNOWLEDGEMENTS

It is simply impossible to imagine this odyssey of getting a doctoral degree without the assistance, guidance and support of my teachers, colleagues, family and friends. Although my adequate expression of gratitude for their contributions is also equally impossible to summarise in a few pages, I would still try to do that by mentioning their names. At this moment of accomplishment, first of all, I would like to pay homage to the founder of Banaras Hindu University, Pandit Madan Mohan Malviya ji, who made this glorious temple to appreciate spiritual, technical and scientific knowledge about this vast existing universe.

I am extremely grateful to my mentor and supervisor, **Dr. Nikhil Kumar**, Asst. Prof. School of Materials Science and Technology [SMST], Indian Institute of Technology (Banaras Hindu University) [IIT(BHU)] for his guidance throughout the journey. I am very thankful for his scholarly suggestions, which will remain with me as an inexhaustible source of scientific learning throughout my life. His approach of encouragement and the stress-free environment he created in the laboratory have made me a far more confident and independent scientist than I was a few years ago.

I want to thank my RPEC committee members, Dr. Joy Prakash Mishra, Asst. Prof., Department of Mechanical and Dr. Akhilesh Kumar Singh, Prof., SMST for their advice and instructions along the way. I would also like to thank the faculty members of the school (SMST) Prof. Rajiv Prakash, Prof. Pralay Maiti, Prof. Chandan Upadhyay, Prof. Chandana Rath, Prof. Bhola Nath Pal, Dr. Ashish Kumar Mishra, Dr. Shrawan Kumar Mishra, Dr. Ravi Panwar and Dr. Uday Shankar for the valuable discussions and suggestions given by them during seminars and otherwise.

I gratefully acknowledge my Institute IIT(BHU), Varanasi for providing me with the necessary funding and fellowship to pursue the research work. I acknowledge Central Instrument Facility, IIT(BHU) for providing various instrumental facilities for research work. I am also thankful to OIM & Texture Lab, Metallurgical Engineering and Material Science Department, Indian Institute of Technology Bombay, Mumbai, for facilitating the EBSD facility and the Science and Engineering Research Board (SERB), India for financial support in doing so. I would like to acknowledge Mechanical and Metallurgy department of IIT(BHU) for extending their laboratory facilities to complete my research work.

I am indebted to my friends and colleagues, for always being there and bearing the good and bad

times with me during my PhD.

I am thankful to all non-teaching staff of the school for their cooperation at all levels.

I am very grateful for being loved and brought up by my grandparents Late Smt. Subhadra Devi and Late Shri Radheshyam Chaubey. I express my indebtedness to my parents, Smt. Asha Chaubey (who has loved me unconditionally, supported me and stand by me throughout this journey) and Shri Jay Prakash Chaubey (who has been the source of my inspiration), and my maternal grandfather Late Shri Prem Chandra Mishra (who encouraged me to focus on my study from my childhood) and my maternal grandmother Sri Lalmani Mishra (whose affection and care have been incredible).

I am also thankful to all whom I could not mention here and who helped me directly or indirectly throughout the PhD duration.

Finally, I am grateful to Almighty God Shiva and his Kashi Vishwanath Dham for giving me the strength to face all the challenges along the journey.

(Nidhi Chaubey)

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ABBREVIATIONS

SHT	Solution heat treated
PA	Peak aging
CR	Cold rolling
WR	Warm rolling
SEM	Scanning electron microscopy
TEM	Transmission electron microscopy
OM	Optical microscope
XRD	X-ray diffraction
FCGR	Fatigue crack growth rate
PSBs	Persisting slip bands
EBSD	Electron backscattered diffraction

SAED	Selected area electron diffraction
EDX	Energy dispersive X-ray
H	Hour
Ecorr	Corrosion voltage
Icorr	Corrosion current
OCP	Open circuit potential
GOS	Grain orientation spread
IPF	Inverse pole figure
IQ	Image quality
ODF	Orientation distribution function
mmpy	mm per year
LPD	Load point displacement
COD	Crack opening displacement

NOTATION

η''	Eta double prime
η'	Eta prime
η	Eta
J	Elastic plastic fracture toughness
J_{1c}	Critical value of J integral in mode I
a	Crack length
b	Uncrack ligament
W	Width of the sample
K	Stress intensity factor
B	Sample thickness
J_{pl}	Plastic component of J integral
A_{pl}	Region under the COD vs Load curve from the origin to the maximum load
TN	Sample thickness
v	Poisson ratio
E	Young modulus of elasticity
n_{pl}	Plastic correction factor
ρ	Dislocation density
σ	Stress
ε	Micro strain
D	Coherent domain size
Ω	Electrical resistance
χ	Chi
b	Burger vector
Hz	Hertz

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 utilized 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. A Maximum of 50 to 60% deformation has been achieved after cold rolling in 7075 Al alloy till date. 70% deformation is achieved after cryo-rolling of 7075 Al alloy. But in this study 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 that 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 (J_{1c}) fracture toughness was studied as well on the SHT, peak aged and rolled sample. Additionally, Vickers hardness and tensile test were performed. Aging and Cold rolling treatment effectively enhanced tensile strength (526 MPa) and hardness (226 HV) ascribed to formation of fine rod shape precipitates of η"(Mg_2Zn_3) 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 (J_{1c}) 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 ×10^(14) 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}<112>) texture, whereas SHT+60% WR sample is showing strong rotated cube({001}<110>) texture, while 90% CR sample is showing strong brass({110}<112>), strong Cu({112}<111>) and strong S({123}<634>) 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 J1c 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 J1c 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 $J_{1}c$ 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 J1c value and fatigue crack growth rate of A