

**MICRO AND NANO-SCALE TRIBOLOGY OF
Mg-9Al-1Zn MAGNESIUM ALLOY UNDER DRY AND
LUBRICATED CONDITIONS**

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**DEPARTEMENT OF MATERIALS SCIENCE AND
ENGINEERING**

INDIAN INSTITUTE OF TECHNOLOGY DELHI

JULY 2021

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by

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**DEPARTMENT OF MATERIALS SCIENCE AND
ENGINEERING**

Submitted

**In fulfillment of requirements of the degree of Doctor of Philosophy
to the**



INDIAN INSTITUTE OF TECHNOLOGY DELHI

JULY 2021

Certificate

This is to certify that the thesis entitled “**Micro and Nano-Scale Tribology of Mg-9Al-1Zn Magnesium Alloy under Dry and Lubricated Conditions**” being submitted by **Mr. Deepak Kumar** to the Indian Institute of Technology Delhi for the award of the degree of **DOCTOR OF PHILOSOPHY** is a record of bonafide research work carried out by him under our supervision and guidance. The matter presented in this thesis has not been submitted, in part or in full to any other University or Institute for the award of any degree or diploma.

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Acknowledgments

First and foremost, I would like to express my profound and sincere gratitude towards my supervisors Prof. Nitya Nand Gosvami and Prof. Jayant Jain for the encouragement and thoughtful guidance during my tenure here as a Research Scholar. They have been extremely supportive and have given me the freedom to carry out my work in the manner in which I wished to execute it. The confidence and experience I gained from various projects will go a long way in my life. I hope, I will find opportunities to work with them in the future and learn more from them. They have been helpful in developing the research attitude by highlighting my shortcomings and suggesting ways to improve them.

I would also like to thank committee members Prof. Rajesh Prasad, Prof. S. K. Sinha and Prof. Suresh Neelakantan for providing me with various inputs and serving on my committee during the tenure. I thank my family members Ram Veer Singh, my father and Chandra Wati Devi, my mother. They did their best in terms of support and guidance. Special thanks to my colleagues in the lab including Dr. Gaurav, Dr. Anuz Zindal, Dr. Himanshu Vashishtha, Aravi Muzzafar, Dr. Aditya Gokhale, Dr. Vickey Nandal, Prashant Mittal, Chetan Singh, Shanta Mohapatra, Himanshu Rai, Abhishek Rastogi, Amit Kumar Prasad, Kasimuthu Maniyan, Nooruddin Ansari, Ranjeet Kumar and Shrikanth Shyam for making the lab a better and lively place to work and learn. Also, Department of Applied Mechanics and Materials Science and Engineering, IIT Delhi are highly acknowledged for providing the experimental and infrastructure. The central research facility of IIT Delhi is also acknowledged for various experimental facilities used in this work.

Deepak Kumar

Abstract

Magnesium and its alloys are receiving great attention in the automotive and aerospace industries due to their high specific strength. However, their use is limited due to poor tribological properties. In past, several macro-scale studies have been conducted to investigate and enhance the tribological properties of magnesium alloys under dry and lubricated conditions. Considering the highly anisotropic and multiphase nature of magnesium-based alloys, the localized tribological properties can be very different which cannot be easily discerned from the macro-scale studies owing to the complexity of macroscopic sliding interface. Therefore, the need for small-scale tribological studies arises to design the magnesium alloys for optimum tribological performance. Nanoindentation and atomic force microscopy (AFM) are the two most viable tools which can measure the tribological properties of localized regions of a multiphase alloy. In the present work, fundamental tribological properties of localized regions (different phases, grain orientations and precipitate orientations) of an AZ91 magnesium alloy has been investigated.

The anisotropy in wear and friction with respect to crystallographic orientation was investigated under dry conditions for solutionized AZ91 alloy using AFM (low-stress regimes) and nanoindentation (high-stress regimes). The basal oriented grains were found to be most wear-resistant and offered less friction force followed by prismatic and pyramidal oriented grains at room temperature. At higher temperatures and stress, dynamic recrystallization was observed within the scratched region for all orientations. Additionally, the anisotropy in wear volume of grains with different crystallographic orientations was found to be absent due to the homogeneity of slip systems at elevated temperatures.

Under dry conditions, the sliding direction effects on wear and friction of discontinuous (DP) and continuous precipitate (CP) regions of an aged AZ91 alloy was investigated under low stress (using AFM) and high stress (using nanoindentation) regimes. In DP regions, a strong dependence of friction and wear with respect to the sliding direction was observed in lower stress regimes, while no sliding direction dependency was observed under high stress for any precipitate containing region. The obtained anisotropic behavior of DP regions was explained by taking the contact geometry between the precipitate regions and the AFM probe into account. Additionally, a mathematical model of friction correlation with interprecipitates spacing was used to validate the experimental results.

In-situ AFM experiments were carried out to understand the fundamental tribological properties of the AZ91 alloy under lubricated conditions in presence of lubricant additives which form sliding induced boundary films due to tribochemical reactions. The tribofilm growth and corresponding friction and wear over localized regions of as-cast and aged AZ91 alloy were investigated in presence of zinc dialkyl dithiophosphate (ZDDP) and acrylamide additive mixed lubricants. The role of stress and temperature on tribofilm growth and friction force was also systematically explored. The tribofilm growth and friction force in the presence of ZDDP and acrylamide additives were found to be highly sensitive to mechanical properties of different phases, temperature and microstructure (e.g., precipitate size) at a given applied stress. Overall, the acrylamide additive was found to be superior to ZDDP as a potential replacement as it offers lower friction force along with tribofilm formation on the matrix as well as precipitate regions at similar stress values whereas ZDDP failed to produce tribofilms on matrix regions.

In summary, the present small-scale tribological study of AZ91 alloy will be very useful for enhancement in tribological properties for various engineering applications under dry as well as lubricated conditions.

सार

मैग्नीशियम और इसके मिश्र धातु अपनी उच्च विशिष्ट शक्ति के कारण ऑटोमोटिव और एयरोस्पेस उद्योगों में बहुत ध्यान आकर्षित कर रहे हैं। हालांकि, खराब ट्राइबोलोजीकल गुणों के कारण उनका उपयोग सीमित है। अतीत में, शुष्क और चिकनाई वाली परिस्थितियों में मैग्नीशियम मिश्र धातुओं के ट्राइबोलोजीकल गुणों की जांच और उन्हें बढ़ाने के लिए कई मैक्रो-स्केल अध्ययन किए गए हैं। मैग्नीशियम-आधारित मिश्र धातुओं की अत्यधिक अन-आइसोट्रोपिक और मल्टीफेज प्रकृति को ध्यान में रखते हुए, लोकलाइज्ड ट्राइबोलोजीकल गुण बहुत भिन्न हो सकते हैं जिन्हें मैक्रोस्कोपिक स्लाइडिंग इंटरफ़ेस की जटिलता के कारण मैक्रो-स्केल अध्ययनों से आसानी से नहीं पहचाना जा सकता है। इसलिए, इष्टतम लोकलाइज्ड प्रदर्शन के लिए मैग्नीशियम मिश्र धातुओं को डिजाइन करने के लिए छोटे पैमाने पर लोकलाइज्ड अध्ययन की आवश्यकता उत्पन्न होती है। नैनोइंडेंटेशन और परमाण्विक बल सूक्ष्मदर्शी यंत्र (एफएम) दो सबसे व्यवहार्य उपकरण हैं जो एक मल्टीफेज मिश्र धातु के लोकलाइज्ड क्षेत्रों के ट्राइबोलोजीकल गुणों को माप सकते हैं। वर्तमान कार्य में, AZ91 मैग्नीशियम मिश्र धातु के लोकलाइज्ड क्षेत्रों (विभिन्न फेजिज, ग्रेन ऑरीएंटेशनस और प्रैसिपिटेड ऑरीएंटेशनस) के मौलिक ट्राइबोलोजीकल गुणों की जांच की गई है।

क्रिस्टलोग्राफिक ऑरीएंटेशन के संबंध में घिसाव और घर्षण में अन-आइसोट्रॉपी की जांच एफएम (कम-तनाव वाला क्षेत्र) और नैनोइंडेंटेशन (उच्च-तनाव वाला क्षेत्र) का उपयोग करके AZ91 मिश्र धातु के लिए शुष्क परिस्थितियों में की गई थी। कमरे के तापमान पर, प्रिज़्मैटिक और पिरामिडल ऑरीएंटेड ग्रेन्स की तुलना में, बेसल ऑरीएंटेड ग्रेन्स सबसे अधिक घिसाव के लिए प्रतिरोधी पाए गए और कम घर्षण बल की पेशकश की। उच्च तापमान और तनाव पर, सभी ऑरीएंटेशनस के लिए घिसाव वाले क्षेत्र के भीतर गतिशील पुनः क्रिस्टलीकरण देखा गया। इसके अतिरिक्त, उंचे तापमान पर, विभिन्न क्रिस्टलोग्राफिक ऑरीएंटेशनस वाले ग्रेन्स के घिसाव की मात्रा में अन-आइसोट्रॉपी स्लिप सिस्टम की एकरूपता के कारण अनुपस्थित पाया गया।

शुष्क परिस्थितियों में, एक एज्ड AZ91 मिश्र धातु के असंतत (डीपी) और निरंतर प्रैसिपिटेड (सीपी) क्षेत्रों के घिसाव और घर्षण पर स्लाइडिंग दिशा प्रभावों की जांच कम तनाव (AFM का उपयोग करके) और उच्च तनाव (नैनोइंडेंटेशन का उपयोग करके) क्षेत्रों के तहत की गई थी। डीपी क्षेत्रों में, कम तनाव वाले क्षेत्रों में, स्लाइडिंग दिशा के संबंध में घर्षण और घिसाव की एक मजबूत निर्भरता देखी गई, जबकि किसी भी प्रैसिपिटेड युक्त क्षेत्र के लिए उच्च तनाव के तहत कोई स्लाइडिंग दिशा निर्भरता

नहीं देखी गई। डीपी क्षेत्रों के प्राप्त अन-आइसोट्रोपिक व्यवहार को प्रैसिपिटेट क्षेत्रों और एएफएम संपर्क ज्यामिति को ध्यान में रखकर समझाया गया था। इसके अतिरिक्त, प्रयोगात्मक परिणामों को मान्य करने के लिए इंटरप्रैसिपिटेट्स स्पेसिंग के साथ घर्षण सहसंबंध के गणितीय मॉडल का उपयोग किया गया था।

इन-सीटू एएफएम प्रयोग AZ91 मिश्र धातु के मौलिक ट्राइबोलोजिकल गुणों को चिकनाई परिस्थितियों में चिकनाई एडिटिव्स की उपस्थिति में समझने के लिए किए गए थे, जो ट्राइबोकेमिकल प्रतिक्रियाओं के कारण स्लाइडिंग प्रेरित सीमा ट्राइबोफिल्म्स का निर्माण करते हैं। ऐज-कास्ट और एज्ड AZ91 मिश्र धातु के स्थानीय क्षेत्रों में ट्राइबोफिल्म वृद्धि और संबंधित घर्षण की जिंक डायएल्काइल डाइथियोफॉस्फेट (ZDDP) और एक्रिलामाइड एडिटिव मिश्रित चिकनाई की उपस्थिति में जांच की गई। ट्राइबोफिल्म वृद्धि और घर्षण बल पर तनाव और तापमान की भूमिका का भी व्यवस्थित रूप से पता लगाया गया था। ZDDP और एक्रिलामाइड एडिटिव्स की उपस्थिति में ट्राइबोफिल्म की वृद्धि और घर्षण बल किसी दिए गए लागू तनाव पर विभिन्न फेजिज, तापमान और माइक्रोस्ट्रक्चर (जैसे, प्रैसिपिटेट आकार) के यांत्रिक गुणों के प्रति अत्यधिक संवेदनशील पाए गए। कुल मिलाकर, एक्रिलामाइड एडिटिव एक संभावित प्रतिस्थापन के रूप में ZDDP से बेहतर पाया गया क्योंकि यह मैट्रिक्स तथा प्रैसिपिटेट पर ट्राइबोफिल्म निर्माण के साथ-साथ समान तनाव पर मैट्रिक्स तथा प्रैसिपिटेट क्षेत्रों को कम घर्षण बल प्रदान करता है जबकि ZDDP मैट्रिक्स क्षेत्रों पर ट्राइबोफिल्म का उत्पादन करने में विफल रहा।

संक्षेप में, AZ91 मिश्र धातु का वर्तमान लघु-स्तरीय ट्राइबोलोजिकल अध्ययन शुष्क और साथ ही चिकनाई वाली परिस्थितियों में विभिन्न इंजीनियरिंग अनुप्रयोगों के लिए ट्राइबोलोजिकल गुणों में वृद्धि के लिए बहुत उपयोगी होगा।

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