

**INFLUENCE OF TEXTURE AND  
TEMPERATURE ON THE SMALL-SCALE  
MECHANICAL BEHAVIOR IN ZINC**

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**DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING  
INDIAN INSTITUTE OF TECHNOLOGY DELHI  
FEBRUARY 2021**

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MECHANICAL BEHAVIOR IN ZINC**

*by*

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**DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING**

*Submitted*

**in fulfilment of the requirements of the degree of Doctor of Philosophy**

*to the*



**INDIAN INSTITUTE OF TECHNOLOGY DELHI**

**FEBRUARY 2021**

# CERTIFICATE

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This is to certify that the thesis entitled “**INFLUENCE OF TEXTURE AND TEMPERATURE ON THE SMALL-SCALE MECHANICAL BEHAVIOR IN ZINC**” being submitted by **Mr. ADITYA GOKHALE** 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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## Acknowledgement

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The culmination of this exhaustive research would not have been possible without the valuable guidance and support of my two eminent supervisors, faculty and administrative staff of IIT Delhi, my colleagues, family and friends. It would be pertinent to acknowledge their contributions and efforts.

First and foremost, I would like to express my profound and sincere gratitude towards my supervisors **Prof. Rajesh Prasad** and **Dr. Jayant Jain**, for the encouragement and professional guidance throughout my tenure here as a Research Scholar. They have been not only been incredibly supportive but gave me the freedom and latitude to carry out my work in the manner I wished to execute. The confidence and experience I gained from various projects I were assigned, will be of immense help to me for a long time to come. I hope I will find opportunities to work with them in the future also and learn from them. They enabled me to develop scientific temper and develop the aptitude for research by questioning my studies, highlighting my shortcomings, and suggesting ways to improve them, and for which I shall remain indebted to them. I would also like to thank the committee members Prof. Puneet Mahajan, Dr. Ajeet Kumar, and Dr. Deepak Kumar, for providing me with various inputs and serving on my committee during the tenure. I would like to thank B. Prasad for allowing me to access and learn about various equipment at Lecture Hall Complex.

A very special thanks to my colleague and a thorough professional R. Sarvesha for helping me and spending his valuable time and energy with all the EBSD characterization of zinc. My interactions with him served as a turning point in my Ph.D. I shall forever be grateful for his invaluable help and thoughtful suggestions for improvement of my research work. I would also miss my heated debates and discussions and the tireless nights in the SEM room for long EBSD scans done with him. I would like to thank Dr. Sudhanshu Shekhar Singh for helping me learn

the nitty-gritties of nanoindentation, which helped me in better understanding the machine and the technique. I am truly grateful to him for extending all the help and support in using the ACMS facility at IIT Kanpur.

A big thanks to my seniors Anurag, Mohit, Karnika, Arzoo and Adnan for being my mentors and providing valuable guidance and support. Special thanks to beloved lab mates, Vicky, Prashant, Chetan, Shantha, Aravi, Abhishek, Kasi, Noor, Ranjeet, Shrikanth, Himanshu, Amit and Deepak for making the lab a better and lively place to work and learn; I will miss you guys. Special thanks to Sakshi and Pranjal for familiarizing and training me on the nanoindenter and AFM. I also thank our lab staff Prasad and Pramod for making the lab equipment accessible to me. A very special thanks to the staff of the Department of Applied Mechanics and Department of Materials Science and Engineering who took care of my official needs. A big thanks to the management of Aravali hostel for making my stay comfortable. Needless to say, hostel will always be missed.

My heartfelt thanks to my family members Col. Ravindra Gokhale, my father, Veena, my mother, and Sarangi, my sister, for their unstinting support and constant encouragement during this entire academic marathon. I would like to express my sincere thanks to my wife, Bhakti, for her love and encouragement. I am grateful to her for patiently enduring this strenuous period in our married life and supporting me tenaciously throughout this journey.

Friends are the family we choose. Thanks to my friends Anil, Sanjeet and Ayan for their unconditional support and love; and for lending an ear during my ups and downs.

I am grateful to the almighty for bestowing me with a good health and fulfilling my wishes, holding and carrying me in every crisis I faced.

**Aditya Gokhale**

## Abstract

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Single crystal studies are useful to extract the deformation behavior of different orientations. However, single crystals are difficult to fabricate and do not offer a complete spectrum of orientations. To circumvent this, different experimental approach like micropillar compression and in-situ testing methods have been developed and are in use. One such method which has established itself as a reliable and simple tool to help study the deformation behavior of individual grain in a polycrystalline material is nanoindentation. The focus of the present work was to study the grain-scale deformation behavior in pure zinc, using nanoindentation as the deformation tool. Hardness and Young's modulus were obtained for differently oriented grains in pure zinc using Berkovich indenter at room and elevated temperature of 150 °C. The basal grain had the lowest Young's modulus as compared to other orientations. Interestingly, Young's modulus at room and high temperature of 150°C were found to be almost similar. Deformation behavior was studied in differently oriented grains using a cono-spherical indenter. The study focused on the behavior viz., slip, twinning, and grain boundary sliding. Basal grains deformed by 2<sup>nd</sup> order pyramidal slip, whereas prismatic grains deformed by basal slip, 2<sup>nd</sup> order pyramidal slip, and contraction twinning. It was observed that the slip lines generated because of the indentation accumulated at the grain boundary. It was found that the accumulation of slip lines led to grain boundary sliding. Grain boundary sliding behavior at room temperature and 150 °C was also investigated using a cono-spherical indenter. As the temperature increases, the sliding of grains increases at the point where slip lines accumulate. Basal grains undergo a complete sliding of the grain. In prismatic grains, it is slip assisted and occurs at the grain boundary where the slip lines meet. Anisotropy in the deformation behavior stimulated further investigation in indentation induced creep response. Indentation induced creep was studied using Berkovich indenter and the creep results show that there is an anisotropy in the creep

response. Basal grain shows a higher creep displacement, than prismatic grains. However, the recovery post removal of the indenter is the largest for basal grain wherein 50% recovery was observed. Two different approaches were employed to estimate the creep stress exponent. Lastly, a more dynamic form of testing was carried out in the form of scratch testing. The texture of the as received material was kept in mind before scratching. The effect of texture on the scratch induced wear response was investigated. It was found that the texture affects the scratch wear response, even at high temperature, wherein scratching on a surface predominantly having basal grains resisted more to wear than the surface having prismatically oriented grains. An interesting outcome of the study was the evidence of recrystallization near the scratch lip and below the indenter. It was found that the recrystallization texture is always basal irrespective of the orientation of grains in the sample.

## सार

---

धातु की विकृति (डेफोर्मेशन) उसके उन्मुखीकरण से जुड़ी होती है और उसे समझने के लिए सिंगल क्रिस्टल का अध्ययन ज़रूरी होता है। सिंगल क्रिस्टल बनाना अपने आप में एक मुश्किल एवं महंगी प्रक्रिया ही नहीं, बल्कि, सिंगल क्रिस्टल सारे उन्मुखीकरण के नहीं बनाये जा सकते। अतः, इस समस्या का दूसरा उपाय यह है की इन-सितु टेस्टिंग के नमूनों अथवा माइक्रोपिल्लर के नमूनों पर प्रयोग किये जाये। इसके अलावा नैनोइन्डेन्टेशन एक सरल और विश्वशनीय प्रयोग साधन है जिससे कि एक पॉलीक्रिस्टलीन नमूने के विभिन्न ग्रेन्स में इंडेन्टेशन करके उनकी विकृति को समझा जा सकता है।

इस अनुसंधान का परम उद्देश्य जस्ता (जिंक) धातु के भिन्न ग्रेन्स में नैनोइन्डेन्टेशन की मदद से उनके विकृति एवं यांत्रिकी गुणों का अध्ययन करना है। बेरकोविच इन्डेण्टर के सहयोग से, रूम टेम्परेचर एवं १५० डिग्री सेल्सियस तापमान पर, जिंक के भिन्न ग्रेन्स की कठोरता (हार्डनेस) एवं यंग'स मॉड्युलस प्राप्त किये गए तथा उनका विश्लेषण किया गया। दूसरे ग्रेन्स के मुकाबले रूम टेम्परेचर पर, बेसल ग्रेन्स का यंग'स मॉड्युलस सबसे कम पाया गया था, किन्तु १५० डिग्री सेल्सियस तापमान पर और रूम टेम्परेचर पर मापे गए यंग'स मॉड्युलस में ज्यादा अंतर नहीं पाया गया। एक कोनो-स्फेरिकल इन्डेण्टर की मदद से भिन्न ग्रेन्स में स्लिप, ट्विनिंग एवं ग्रेन बाउंड्री स्लाइडिंग जैसे डेफोर्मेशन बिहेवियर का अध्ययन किया गया था। बेसल ग्रेन्स में डेफोर्मेशन सेकंड आर्डर पिरॅमिडल स्लिप के द्वारा पाया गया था, तथा प्रिस्मैटिक ग्रेन्स में डेफोर्मेशन बेसल स्लिप, सेकंड आर्डर पिरॅमिडल स्लिप एवं कॉन्ट्रेक्शन ट्विनिंग की वजह से हुआ था। ऐसा देखा गया कि, जो स्लिप लाइन्स उत्पन्न हुए थीं वह ग्रेन बाउंड्री पर जाकर जमा हो गयीं। इस कारण से ग्रेन बाउंडरीस एक दूसरे के साथ स्लाइड कर रही थीं। इसे ग्रेन बाउंड्री स्लाइडिंग कहते हैं और इसका जिंक में रूम टेम्परेचर अथवा १५० डिग्री सेल्सियस तापमान पर अध्ययन किया गया। अध्ययन से यह पता चला कि जिंक में जैसे तापमान बढ़ता है, वैसे ग्रेन बाउंड्री स्लाइडिंग भी बढ़ती है। बेसल ग्रेन पूरी तरह स्लाइड होजाती है लेकिन प्रिस्मैटिक ग्रेन्स में स्लाइडिंग उसी जगह होती है जिस जगह स्लिप लाइन ग्रेन

बाउंड्री से मिलती है। भिन्न ग्रेन्स मे इंडेंटेशन करने के पश्चात यह बात सामने आयी कि हर ग्रेन कि हार्डनेस, यंग'स मॉड्युलस एवं डेफोर्मेशन बिहेवियर अलग है, और इनका ग्रेन्स की उन्मुखीकरण से बहुत गहरा सम्बन्ध है। इन परिणामो से प्रेरित होकर इंडेंटेशन क्रीप का प्रयोग किया गया। इंडेंटेशन क्रीप के प्रयोग के बाद यह मालूम पड़ा कि सभी ग्रेन्स का क्रीप रिस्पांस अलग है और इनका ग्रेन्स की उन्मुखीकरण से सम्बन्ध है। प्रिस्मैटिक ग्रेन्स की तुलना मे बेसल ग्रेन ज्यादा क्रीप डिस्प्लेसमेंट प्रदर्शित करती है। लेकिन, इन्डेण्टर के हटाने के पश्चात, इम्प्रेशन की रिकवरी, बेसल ग्रेन मे, लगभग ५० % तक पायी गयी। क्रीप स्ट्रेस प्रतिपादक का सटीक एवं विश्वसनीय अनुमान दो भिन्न तरीको से किया गया। अंततः, एक डायनामिक प्रयोग किया, जिसमे इन्डेण्टर कि मदद से जिंक मे स्क्रेच किया गया। स्क्रेच परीक्षण करने से पूर्व जिंक नमूनों का टेक्सचर का ख़ास ध्यान रखा गया था। टेक्सचर का प्रभाव स्क्रेच वियर पर कैसे होता है यह इस प्रयोग का केंद्र बिंदु था। इस प्रयोग से ये देखा गया कि टेक्सचर, स्क्रेच वियर प्रतिक्रिया पर प्रभावशाली है। बेसल ग्रेन युक्त सतह का स्क्रेच वियर प्रतिरोध, प्रिस्मैटिक ग्रेन्स युक्त सतह कि तुलना में ज्यादा होता है। जिंक में स्क्रेच के दौरान रिक्रिस्टलैजेशन पाया गया और ये बहुत दिलचस्प परिणाम था। इंडेंटेशन स्क्रेच के सहयोग से रीक्रिस्टलॉइजेशन बिहेवियर का और अध्ययन किया गया। शोध के पश्चात ये नतीजा सामने आया कि चाहे जिंक की सतह का कोई भी टेक्सचर हो, रीक्रिस्टलाइज़्ड ग्रेन्स का उन्मुखीकरण हमेशा बेसल ही होता है।

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