

**DEVELOPMENT OF GRAIN BOUNDARY
PRECIPITATE FREE ZONE (PFZ) AND ITS
EFFECT ON MECHANICAL PROPERTIES OF
A Mg-8Al-0.5Zn ALLOY**

ANUZ ZINDAL



**DEPARTMENT OF APPLIED MECHANICS
INDIAN INSTITUTE OF TECHNOLOGY DELHI
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by

Anuz Zindal

DEPARTMENT OF APPLIED MECHANICS

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CERTIFICATE

This is to certify that the thesis entitled “**DEVELOPMENT OF GRAIN BOUNDARY PRECIPITATE FREE ZONE (PFZ) AND ITS EFFECT ON MECHANICAL PROPERTIES OF A Mg-8Al-0.5Zn ALLOY**” being submitted by **Mr. ANUZ ZINDAL** 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.

Prof. Rajesh Prasad

Professor

Department of Applied Mechanics
Indian Institute of Technology Delhi

Dr. Jayant Jain

Assistant Professor

Department of Applied Mechanics
Indian Institute of Technology Delhi

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Abstract

Precipitation hardening is considered to be an important mechanism to alter the mechanical properties of age hardenable alloys. However, a precipitate free zone (PFZ) along grain boundary is formed during precipitation. The occurrence of PFZ in various age hardenable alloys is a well-known phenomenon. The grain boundary microstructural features (PFZ and precipitates) can significantly affect the mechanical properties and corrosion resistance of the alloys. However, in magnesium alloys, the development of PFZ in terms of formation mechanisms and the evolution of its width with different process parameters have not been investigated extensively. In the present work, the effect of different process variables such as solutionizing temperature, quenching medium, aging temperature, aging time, grain size and pre-strain on the evolution of PFZ in an AZ80 magnesium alloy has been systematically studied. Moreover, the influence of the PFZ and grain boundary precipitate characteristics on the fracture and fracture toughness properties of AZ80 magnesium alloy has also been investigated.

The results suggest that the PFZ width decreases with the increase in the solutionizing temperature and the severity of quenching rate, whereas it increases with aging temperature and time. This is explained in terms of vacancy and solute depletion along the grain boundary. The solute depletion has been noticed to be the dominant mechanism in the development of PFZ as compared to vacancy depletion. The nanoindentation and electron probe micro-analysis (EPMA) have been performed within PFZ to verify the solute depletion experimentally. From the EPMA analysis, the width of PFZ and solute depletion has been found comparable with each other at all the different aging temperatures. It has also been observed that the PFZ and grain boundary precipitates nucleate and grow in tandem with each other during aging treatment. The width of PFZ is found to be varied linearly in the decreasing trend with the severity of pre-strain. This was attributed to the pile-up of dislocations at the grain boundary. The PFZ width is also found

to be increased with the grain size. This was rationalised by considering the variation of grain boundary solute concentration and precipitate characteristics.

The mechanical properties of the AZ80 magnesium alloy has been found to be greatly affected with the aging temperature. This can be explained in terms of the variation in precipitate characteristics at the grain boundaries as well as inside the grains and evolution of PFZ. The higher area fraction of grain boundary precipitates, larger precipitate size and strain localization within PFZ increases the tendency of intergranular fracture. On the other hand, the higher number density of precipitates with finer distribution promotes the occurrence of transgranular fracture. The model proposed by Hornbogen and Graf (1977) to correlate the fracture toughness with the microstructural characteristics for aluminium alloys has been successfully validated for the AZ80 magnesium alloy. The results suggest a good agreement between the measured and predicted fracture toughness. Nonetheless, this consequently emphasizes the role of microstructure in controlling the fracture toughness of the aged AZ80 alloy.

सार

प्रिसिपिटेशन हार्डनिंग एज हार्डनिंग मिश्र धातुओं के यांत्रिक गुणों को बदलने के लिए एक महत्वपूर्ण तंत्र माना जाता है। हालांकि, ग्रेन बाउंड्री के साथ एक प्रेसिपिटेट फ्री जोन (पीएफजेड) प्रिसिपिटेशन के दौरान बनता है। पीएफजेड का गठन विभिन्न एज हार्डनिंग मिश्र धातुओं में एक प्रसिद्ध घटना है। ग्रेन बाउंड्री के सूक्ष्म संरचनात्मक गुण (पीएफजेड और प्रेसिपिटेट) मिश्र धातुओं के यांत्रिक गुणों और संक्षारण प्रतिरोध को काफी प्रभावित कर सकते हैं। हालांकि, मैग्नीशियम मिश्र धातुओं में, गठन तंत्र के संदर्भ में पीएफजेड के विकास और विभिन्न प्रक्रिया मापदंडों के साथ उसकी चौड़ाई के विकास में बड़े पैमाने पर जांच नहीं की गई है। वर्तमान कार्य में, मैग्नीशियम मिश्र धातु में पीएफजेड के विकास पर विभिन्न प्रक्रिया चर का प्रभाव जैसे कि सल्यूशनाइजिंग टेंपरेचर, क्वेनचिंग मीडिया, एजिंग टेंपरेचर, एजिंग टाइम, ग्रेन साइज ओर पूर्व-तनाव को व्यवस्थित रूप से अध्ययन किया गया है। इसके अलावा, पीएफजेड और ग्रेन बाउंड्री का प्रभाव एजेड 80 मैग्नीशियम मिश्र धातु के फ्रैक्चर और फ्रैक्चर टफनेस पदार्थ गुणों की विशेषताओं पर भी जांचा गया है।

परिणाम बताते हैं कि पीएफजेड की चौड़ाई सल्यूशनाइजिंग टेंपरेचर में वृद्धि और क्वेनचिंग मीडिया की तीव्रता के साथ घट जाती है, जबकि यह एजिंग टेंपरेचर और एजिंग टाइम से बढ़ जाती है। ग्रेन बाउंड्री के साथ रिक्ति और सल्यूट विलेपन के संदर्भ में इसे समझाया गया है। रिक्ति की कमी के मुकाबले पीएफजेड के विकास में सल्यूट विलेपन की कमी को प्रमुख तंत्र माना गया है। नैनोइंटेंडेंशन और इलेक्ट्रॉन जांच सूक्ष्म-विश्लेषण (ईपीएमए) को पीएफजे के भीतर सल्यूट विलेपन को अनुभूतिपूर्वक जाचने के लिए प्रयोग किया जाता है। ईपीएमए विश्लेषण से, पीएफजेड की चौड़ाई और सल्यूट विलेपन अलग-अलग एजिंग टेंपरेचर पर एक-दूसरे के साथ तुलनीय पाय गये हैं। यह भी पाया गया है कि पीएफजेड और ग्रेन बाउंड्री एजिंग उपचार के दौरान एक साथ निर्मित होती और बढ़ती है। पूर्व-तनाव की तीव्रता के साथ पीएफजेड की चौड़ाई घटती प्रवृत्ति में रैखिक रूप से परिवर्तित होती है। इसे ग्रेन बाउंड्री पर डिसलोकेशन के संग्रह को जिम्मेदार ठहराया गया था। ग्रेन बाउंड्री के आकार के साथ पीएफजेड की चौड़ाई भी बढ़ी है। यह ग्रेन बाउंड्री सल्यूट एकाग्रता और प्रेसिपिटेट लक्षणों की परिवर्तन पर विचार करके युक्तिसंगत था।

एजेड 80 मैग्नीशियम मिश्र धातु के यांत्रिक गुणों को एजिंग टेंपरेचर से काफी प्रभावित पाया गया है। इसे ग्रेन बाउंड्री के साथ-साथ ग्रेन के अंदर के प्रेसिपिटेड लक्षणों और पीएफजे में परिवर्तन के संदर्भ में समझाया जा सकता है। ग्रेन बाउंड्री के प्रेसिपिटेड का उच्च क्षेत्र अंश, बड़े आकार का प्रेसिपिटेड और पीएफजे के भीतर तनाव स्थानीयकरण इंटरग्रेनुलर फ्रैक्चर की प्रवृत्ति को बढ़ाता है। दूसरी ओर, बेहतर वितरण के साथ प्रेसिपिटेड की उच्च संख्या घनत्व ट्रांसग्रानुलर फ्रैक्चर की घटना को बढ़ावा देता है। एल्यूमीनियम मिश्र धातुओं के लिए माइक्रोस्ट्रोकल विशेषताओं के साथ फ्रैक्चर टफनेस को सहसंबंधी बनाने के लिए हॉर्नबोजेन और ग्राफ (1977) द्वारा प्रस्तावित मॉडल को एजेड 80 मैग्नीशियम मिश्र धातु के लिए सफलतापूर्वक मान्य किया गया है। परिणाम बताते हैं कि मापा और भविष्यवाणी फ्रैक्चर टफनेस के बीच एक अच्छा समझौता है। यह फलस्वरूप एजेड 80 मिश्र धातु के फ्रैक्चर टफनेस को नियंत्रित करने में सूक्ष्म संरचना की भूमिका पर जोर देती है।

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