

**DEFORMATION AND STATIC
RECRYSTALLIZATION BEHAVIOR OF
EXTRUDED PURE MAGNESIUM UNDER
STRAIN PATH CHANGE**

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

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EXTRUDED PURE MAGNESIUM UNDER
STRAIN PATH CHANGE**

by

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DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING

Submitted

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Certificate

This is to certify that the thesis entitled “DEFORMATION AND STATIC RECRYSTALLIZATION BEHAVIOR OF EXTRUDED PURE MAGNESIUM UNDER STRAIN PATH CHANGE” being submitted by Mrs. SHANTA MOHAPATRA 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 her 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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Abstract

The present work has examined the influence of strain path change on deformation behaviour, texture evolution, and recrystallization of pure Mg. The samples were subjected to compression at room temperature along ED, ND, ND-ED, and ED-ND directions to the same total strain (ED: Extrusion direction, ND: Normal direction). Electron back-scattered diffraction (EBSD), hardness testing, and X-ray diffraction were utilized to examine the deformed and annealed samples at different conditions. The texture evolution in the ND-ED and ED-ND samples shows a significant difference than the samples independently deformed along ED and ND directions. As-deformed microstructures illustrate the presence of lenticular shaped twins in the ND-ED and ED-ND samples. Simultaneously, complete flipping of original grains due to twinning was observed in the ED10 and ND10 samples. Annealing was done both isothermally and isochronally from 150 °C-300 °C for different times. There was a negligible reduction of hardness values in ED-ND samples upon annealing. However, the recrystallization of the ED deformed sample showed the fastest kinetics, followed by ND-ED and ND samples, respectively. This behavior is rationalized based on the variations in deformation mechanisms. For all three conditions, the formation of new grains at prior grain boundaries appears to be the prevalent mechanism for recrystallization. Additionally, for the ND-ED deformed samples, evidence of recrystallization within $\{10\bar{1}2\}$ twin is reported.

In the grain growth study, as-extruded pure magnesium characteristics were investigated for samples annealed at temperatures ranging from 300 °C to 450 °C for different times. The grain growth kinetics was determined following the Burke-Turnbull model. The grain growth exponent was estimated as $n = 3.7$, indicating rapid coarsening. A normal grain growth behavior was observed both at low and high annealing temperatures.

However, at intermediate annealing temperature, abnormal grain growth was recorded. This temperature dependence of grain growth behavior is attributed to competition between dislocation density gradient and grain boundary mobility.

सार

वर्तमान कार्य ने विकृति व्यवहार, टेक्सचर विकास और शुद्ध एमजी के पुनः क्रिस्टलीकरण पर तनाव पथ परिवर्तन के प्रभाव की जांच की है। नमूने ईडी, एनडी, एनडी-ईडी, और ईडी-एनडी दिशाओं के साथ कमरे के तापमान पर एक ही कुल तनाव (ईडी: एक्सट्रूज़न दिशा, एनडी: सामान्य दिशा) के लिए संपीड़न के अधीन थे। इलेक्ट्रॉन बैक-बिखरे हुए विवर्तन (ईबीएसडी), कठोरता परीक्षण और एक्स-रे विवर्तन का उपयोग विभिन्न परिस्थितियों में विकृत और अनिल्ड नमूनों की जांच के लिए किया गया था। एनडी-ईडी और ईडी-एनडी नमूनों में टेक्सचर विकास ईडी और एनडी दिशाओं के साथ स्वतंत्र रूप से विकृत नमूनों की तुलना में महत्वपूर्ण अंतर दिखाता है। विकृत माइक्रोस्ट्रक्चर एनडी-ईडी और ईडी-एनडी नमूनों में लैटिकुलर के आकार के द्विन की उपस्थिति का वर्णन करते हैं। इसके साथ ही, ईडी₁₀ और एनडी₁₀ नमूनों में द्विनिंग के कारण मूल ग्रेन का पूर्ण रूप से पलटना देखा गया। एनीलिंग अलग-अलग समय के लिए १५० डिग्री सेल्सियस-३०० डिग्री सेल्सियस से समतापी रूप से और समकालिक रूप से दोनों तरह से किया गया था। एनीलिंग करने पर ईडी-एनडी नमूनों में कठोरता मूल्यों में नगण्य कमी आई थी। हालांकि, ईडी विकृत नमूने के पुनः क्रिस्टलीकरण ने क्रमशः एनडी-ईडी और एनडी नमूनों के बाद सबसे तेज गतिज दिखाया। विरूपण तंत्र में भिन्नता के आधार पर इस व्यवहार को युक्तिसंगत बनाया गया है। तीनों स्थितियों के लिए, पूर्व ग्रेस की सीमाओं पर नए ग्रेस का निर्माण पुनर्क्रिस्टलीकरण के लिए प्रचलित तंत्र प्रतीत होता है। इसके अतिरिक्त, एनडी-ईडी विकृत नमूनों के लिए, {१०१₂} द्विन के भीतर पुनर्क्रिस्टलीकरण के साक्ष्य की सूचना दी गई है।

ग्रेन वृद्धि अध्ययन में, अलग-अलग समय के लिए ३०० डिग्री सेल्सियस से ४५० डिग्री सेल्सियस तक के तापमान पर अनिल्ड नमूनों के लिए एक्सट्रूडेड शुद्ध मैग्नीशियम विशेषताओं की जांच की गई थी। बर्क-टर्नबुल मॉडल के बाद ग्रेन वृद्धि गतिज निर्धारित किया गया था। ग्रेन की वृद्धि

का घातांक एन = ३. ७ के रूप में अनुमानित किया गया था, जो तेजी से मोटे होने का संकेत देता है। एक सामान्य ग्रेन वृद्धि व्यवहार कम और उच्च एनीलिंग तापमान दोनों पर देखा गया था। हालांकि, मध्यवर्ती एनीलिंग तापमान पर, असामान्य ग्रेन वृद्धि दर्ज की गई थी। ग्रेन वृद्धि व्यवहार की यह तापमान निर्भरता अव्यवस्था घनत्व ढाल और ग्रेन सीमा गतिशीलता के बीच प्रतिस्पर्धा के लिए जिम्मेदार है।

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Symbols and Abbreviations

ED	Extrusion direction
ND	Normal direction
TD	Transverse direction
JMAK model	Johnson–Mehl–Avrami– Kolmogorov model
BSE	Back scattered electron
FESEM	Field Emission Scanning Electron Microscope
KAM	Kernel’s average misorientation
HCP	Hexagonal close-packed
FCC	Face-centered cubic
CRSS	Critical resolved shear stress
TEM	Transmission electron microscopy
RT	Room temperature
SFE	Stacking fault energy
RD	Rolling direction
SIBM	Strain-induced boundary migration
HAGB	High angle grain boundary
LAGB	Low angle grain boundary