MULTI-AXIAL FORGING OF AA6082/B_4C_P NANOCOMPOSITE DEVELOPED THROUGH ULTRASONIC ASSISTED STIR CASTING

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by

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Submitted
In fulfillment of the requirements of the degree of Doctor of Philosophy
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Dedicated to

My Lovely Wife Sakshi

and

My Beloved Parents

CERTIFICATE

This is to certify that the thesis entitled "**Multi-Axial Forging of AA6082/B_4C_p Nanocomposite Developed Through Ultrasonic Assisted Stir Casting**" being submitted by Mr. Srijan Prabhakar to the Indian Institute of Technology Delhi for the award of the degree of Doctor of Philosophy in Department of Mechanical Engineering is a bonafide research work carried out by him under my supervision and guidance. To the best of my knowledge, the thesis has reached the requisite standard. The research reports and the results presented in this thesis have not been submitted in parts or in full to any other University or Institute for the award of any degree or diploma.

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ABSTRACT

Aluminium matrix composites (AMCs) with nanoparticle reinforcement exhibit superior mechanical properties compared to conventional AMCs. Stir casting, a widely used fabrication method for AMCs, faces challenges like agglomeration and poor wettability of nanoparticles. This study focuses on AA6082/B_4C_p metal matrix nanocomposites developed through ultrasonic-assisted stir casting, incorporating B_4C nanoparticles and Ti-based flux (K_2TiF_6) as reinforcement. Ultrasonic vibration, applied post-mechanical stirring, ensures uniform distribution of nanoparticle using a custom-made setup. The formation of secondary phases (Al_3Ti, $TiSi_2$) refines the matrix microstructure and enhances B_4C wettability.

The effect of processing temperature, % wt. of 'B_4C and K_2TiF_6' and vibration time and interaction of these three parameters on mechanical properties have been characterized. Box Behnken design and Response Surface Methodology optimize parameters for balanced compressive strength and failure strain. Under optimal conditions, a 22% improvement in compressive strength and a 144% increase in fracture strain are achieved, with significant grain refinement (342 μm to 78 μm).

To further enhance AMC properties, multi-axial forging (MAF), a severe plastic deformation process, is applied. MAF involves subjecting a material to a large plastic strain by application of sequential compression in all the three principal axes. A true strain of 0.1 (approx.) was imparted in each direction, leading to a total effective strain of 0.3 in each cycle. The process has also been simulated using finite element method. The strain inhomogeneity from centre to surface has been predicted by finite element simulation of multi-axial forging with Voce hardening model and it correlated well with the experimentally determined microhardness variation. The strain inhomogeneity factor (IF) was highest after cycle 1 and it decreased significantly later in 2^{nd} and 3^{rd} cycles for the composite. The peak load predicted through FE simulation for each pass agreed well with the experimental values for both the

alloy and the composite. In MAF experiments, it was found that a total true strain of 0.894 (three cycles) could be imparted to the composite in MAF before failure whereas only a total true strain of 0.293 (one cycle) could be imparted to the AA6082 alloy.

Microstructural changes due to MAF are examined through SEM, EDS, and EBSD. After three MAF cycles, the composite exhibits a refined grain structure (154 µm to 52 µm) and a bimodal grain distribution. B_4C nanoparticle distribution improves with more MAF cycles. Dislocation density varies with cycles, influenced by dislocation annihilation and recovery. An intermetallic phase (Al_5FeSi) with a Chinese script pattern emerges with increasing strain. Crystallographic texture evolution correlates with observed variations in yield strength during MAF.

Keywords: Aluminium Matrix Composites, Ultrasonic Assisted Stir Casting, Optimization, Ultimate Compressive Strength, Failure Strain, Multi-Axial Forging, Finite Element Simulation, Strain Inhomogeneity, Microstructure, Crystallographic Texture.

सारांश

नैनोकण सुदृढीकरण के साथ एल्यूमीनियम मैट्रिक्स कंपोजिट (एएमसी) पारंपरिक एएमसी की तुलना में बेहतर यांत्रिक गुण प्रदर्शित करते हैं। स्टिर कास्टिंग, एएमसी के लिए व्यापक रूप से उपयोग की जाने वाली निर्माण पद्धति, नैनोकणों के एकत्रीकरण और खराब अस्थिरता जैसी चुनौतियों का सामना करती है। यह अध्ययन अल्ट्रासोनिक-असिस्टेड स्टिर कास्टिंग के माध्यम से विकसित AA6082/B_4C_p धातु मैट्रिक्स नैनोकम्पोजिट पर केंद्रित है, जिसमें सुदृढीकरण के रूप में B_4C नैनोकणों और Ti-आधारित फ्लक्स (K_2TiF_6) को शामिल किया गया है। अल्ट्रासोनिक कंपन, यांत्रिक स्टिरिंग के बाद लागू, एक कस्टम-निर्मित सेटअप का उपयोग करके नैनोकणों का समान वितरण सुनिश्चित करता है। द्वितीयक फेजेज (Al_3Ti, TiSi2) का निर्माण मैट्रिक्स माइक्रोस्ट्रक्चर को संशोधित करता है और B_4C वेटेबिलिटी को बढ़ाता है। प्रसंस्करण तापमान, 'B_4C और K_2TiF_6' का प्रतिशत भार और कंपन समय और इन तीन मापदंडों के संयोग का प्रभाव मैकेनिकल गुणों पर चरित्रित किया गया है। बॉक्स बेनकेन डिज़ाइन और रिस्पांस सरफेस मेथडोलॉजी संतुलित संपीड़न शक्ति और विफलता तनाव के लिए मापदंडों का अनुकूलन करती है। आदर्श परिस्थितियों में, संपीड़न शक्ति में 22% सुधार और फ्रैक्चर तनाव में 144% की वृद्धि हासिल की जाती है, जिसमें महत्वपूर्ण दाना शोधन (342 माइक्रोन से 78 माइक्रोन) होता है।

एएमसी गुणों को और बढ़ाने के लिए, मल्टी-एक्सियल फोर्जिंग (एमएएफ), एक गंभीर प्लास्टिक विरूपण प्रक्रिया, लागू की जाती है। एमएएफ में सभी तीन प्रमुख अक्षों में अनुक्रमिक संपीड़न के अनुप्रयोग द्वारा एक सामग्री को बड़े प्लास्टिक तनाव के अधीन करना शामिल है। प्रत्येक दिशा में 0.1 (लगभग) का वास्तविक स्ट्रेन प्रदान किया गया, जिससे प्रत्येक चक्र में कुल प्रभावी स्ट्रेन 0.3 हो गया। इस प्रक्रिया को फाइनाइट एलिमेंट विधि का उपयोग करके भी अनुकरण किया गया है। फाइनाइट तत्व सिमुलेशन के माध्यम से केंद्र से सतह तक की विकृति असमानता का पूर्वानुमान किया गया है और यह प्रयोगात्मक रूप से निर्धारित माइक्रोहार्डनेस विविधता के साथ अच्छी तरह से संगत होता है। तनाव अमानवीयता कारक (आईएफ) चक्र 1 के बाद उच्चतम था और बाद में कम्पोजिट के लिए दूसरे और तीसरे चक्र में यह काफी कम हो गया। प्रत्येक पास के लिए एफई सिमुलेशन के माध्यम से अनुमानित अधिकतम भार मिश्र धातु और मिश्रित दोनों के लिए प्रयोगात्मक मूल्यों से अच्छी तरह सहमत है। एमएएफ प्रयोगों में, यह पाया गया कि विफलता से पहले एमएएफ में कम्पोजिट को 0.894 (तीन चक्र) का कुल वास्तविक तनाव प्रदान किया जा सकता था, जबकि AA6082 मिश्र धातु को केवल 0.293 (एक चक्र) का कुल वास्तविक तनाव प्रदान किया जा सकता था।

एमएएफ के कारण होने वाले सूक्ष्म संरचनात्मक परिवर्तनों की जांच एसईएम, ईडीएस और ईबीएसडी के माध्यम से जांचा गया है। तीन एमएएफ चक्रों के बाद, कम्पोजिट एक परिष्कृत दाना संरचना (154 μm से 52 μm) और एक द्विमोडल दाना वितरण प्रदर्शित करता है। अधिक एमएएफ चक्रों के साथ बी4सी नैनोकण वितरण में सुधार होता है। अव्यवस्था का घनत्व चक्रों के साथ बदलता रहता है, जो अव्यवस्था के विनाश और पुनर्प्राप्ति से प्रभावित होता है। चीनी लिपि पैटर्न के साथ एक इंटरमेटेलिक चरण (Al_5FeSi) बढ़ते तनाव के साथ उभरता है। क्रिस्टलोग्राफिक बनावट का विकास एमएएफ के दौरान उपज शक्ति में देखी गई विविधताओं से संबंधित है।

मुख्य शब्द: एल्यूमिनियम मैट्रिक्स कंपोजिट, अल्ट्रासोनिक असिस्टेड स्टिर कास्टिंग, ऑप्टिमाइजेशन, अल्टीमेट कंप्रेसिव स्ट्रेंथ, फेल्योर स्ट्रेन, मल्टी-एक्सियल फोर्जिंग, परिमित तत्व सिमुलेशन, स्ट्रेन इनहोमोजेनिटी, माइक्रोस्ट्रक्चर, क्रिस्टलोग्राफिक टेक्सचर।

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NOMENCLATURE

Hv- Vicker's hardness

Tm- Melting Temperature

σ_y- Yield Strength

σ_0- Overall lattice resistance to dislocation movement

K_{hp}- Hall-Petch Constant

d- Grain Size

Φ- Inner Die Angle

Ψ- Outer Die Angle

ε- Strain applied in each pass

vol.%- Volume Percentage

wt.%- Weight Percentage

θ- Contact Angle

K_h- Mass Matrix

M_h- Mass Matrix

ω_i- Natural Frequency of I^{th} Mode Shape

u_i- i^{th} Eigenvector (Mode Shape)

k- Number of Factors/ Variables

Y- Response Variable

n- Number of Variables

β_i, β_{ii} and β_{ij}- Coefficients of Linear, Square, and Interacting terms

Xi and Xj- Independent Variables or Factors

ε- Random Error

m- Interface Friction Factor

τ- Shear Stres

σ_T- Flow Stress

σ_n- Normal Contact Pressure

ε_1- True Strain in Height for 1st Pass

ε_2- True Strain in Height for 2nd Pass

ε_3- True Strain in Height for 3rd Pass

h_f and h_0- Final and Initial Dimensions of the Sample in the Direction of Compression

$\bar{\varepsilon}$- Effective Starin

ε_f- Failure Strain

σ_{lv}- Surface Energy of Al Melt in Vapour Phase

σ_{sv}- Surface Energy of Reinforcement Particles in Vapour Phase

σ_{sl}- Surface Energy of The Particle in Liquid

u- Maximum Velocity of Acoustic Streaming

f- Frequency of Ultrasonic Vibration

a- Amplitude of Vibration

σ- True Stress

K- Strength Coefficient

ϵ- True Strain

n- Strain Hardening Index

A, B, C, K, ϵ_0- Material Constants

h_i- Initial Thickness

h_f- Final Thickness

Δh- Change in Thickness

e- Engineering Strain

ρ_{GND}- Dislocation Density

μ- Step Size of EBSD Acquisition Process

b- Burgers Vector of the Material

ABBREVIATIONS

AMCs- Aluminium Matrix Composites

ANOVA- Analysis of Variance

ARB- Accumulative Roll Bonding

BBD- Box Behnken Design

CCD- Central Composite Design

CG- Coarse-grained

CNT- Containing Carbon Nanotubes

DLS- Dynamic Light Scattering

DoE- Design of Experiment

DRX- Dynamic Recrystallization

EBSD- Electron Backscatter Diffraction

ECAP- Equal Channel Angular Pressing

ECAP- Equal Channel Angular Pressing

ECAP- Equal Channel Angular Pressing

EDS- Energy-Dispersive X-Ray Spectroscopy

EDX- Energy Dispersive X-ray

FCC- Face Centered Cubic

FE- Finite Element

GPB- Guinier–Preston–Bagaryatsky

GRG- Generalized Reduced Gradient

HAGBs- High-Angle Grain Boundaries

HIP- Hot Isostatic Pressing

HMI- Human Machine Interface

HPT- High-Pressure Torsion

HPT- High-Pressure Torsion

IF- Inhomogeneity Factor

IPF- Inverse Pole Figure

IQ- Image Quality

KAM- Kernel Average Misorientation

LAGBs- Low Angle Grain Boundaries

MAF- Multi-axial Forging

MAF- Multi-Axial Forging

MCC- Multiple Constrained Compression

MDF- Multi-Directional Forging

MMCs- Metal Matrix Composites

NI- National Instruments

ODF- Orientation Distribution Function

ODF- orientation distribution function

PCS- Photon Correlation Spectroscopy

PDI- Polydispersity Index

PID- Proportional–Integral–Derivative

PM- Powder Metallurgy

QELS- Quasi-Elastic Light Scattering

RPRF- Repetitive Press Roll Forming

RSM- Response Surface Methodology

RTF- Room Temperature Forging

SEM- Scanning Electron Microscope

SFE- Stacking Fault Energy

SL- Semisolid-liquid

SPD- Severe Plastic Deformation

SS- Semisolid-semisolid

SSE- Sum of Square of Errors

TEM- Transmission Electron Microscope

UCS- Ultimate Compressive Strength

UFG- Ultrafine-grained

UTM- Universal Testing Machine

UTS- Ultimate Tensile Strength

UV- Ultrasonic Vibration

WF- Warm Forging

XRD- X-Ray Diffraction

YS- Yield Strength