



CHARACTERIZATION, MODELING AND OPTIMIZATION OF WEAR BEHAVIOR OF DUAL MATRIX AI-CNT COMPOSITES

By

Noha Mohamed Abdeltawab Elsayd

A Thesis Submitted to the
Faculty of Engineering at Cairo University
in Partial Fulfillment of the
Requirements for the Degree of
MASTER OF SCIENCE
in
MECHANICAL DESIGN & PRODUCTION ENGINEERING

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT
2017

**CHARACTERIZATION, MODELING AND OPTIMIZATION
OF WEAR BEHAVIOR OF DUAL MATRIX AI-CNT
COMPOSITES**

By

Noha Mohamed AbdElTawab ElSayd

A Thesis Submitted to
Faculty of Engineering at Cairo University
in Partial Fulfillment of the Requirements for the Degree of
MASTER OF SCIENCE
in
MECHANICAL DESIGN & PRODUCTION ENGINEERING

Under the Supervision of

Prof. Dr. Abdalla Wifi

Professor of Materials and Manufacturing
Engineering, Mechanical Design and
production department, Faculty of
Engineering, Cairo University

Prof. Dr. Amal K. Esawi

Professor of Mechanical Engineering
Mechanical Engineering Department
School of Sciences & Engineering,
The American University in Cairo.

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT
2017

CHARACTERIZATION, MODELING AND OPTIMIZATION OF WEAR BEHAVIOR OF DUAL MATRIX AI-CNT COMPOSITES

By
Noha Mohamed Abdeltawab Elsayd

A Thesis Submitted to the
Faculty of Engineering at Cairo University
Requirements for the Degree of in Partial Fulfillment of the
MASTER OF SCIENCE
In
MECHANICAL DESIGN & PRODUCTION ENGINEERING

Approved by the examining committee

Prof. Dr. Abdalla Shaaban Wifi (Main Advisor)
Department of Mechanical Design and Production
Faculty of Engineering, Cairo University

Prof. Dr. Amal Kamal Esawi (Advisor)
Department of Mechanical Engineering
School of Science and Engineering
The American University in Cairo

Prof. Dr. Iman Salah El-Din El-Mahallawi (Internal Examiner)
Department of Mining, Petroleum and Metallurgy
Faculty of Engineering Cairo University

Prof. Dr. Ashraf Nassef (External Examiner)
Department of Mechanical Engineering
School of Science and Engineering
The American University in Cairo

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT
2017

Engineer's Name: Noha Mohamed Abdeltawab Elsayd
Date of Birth: 16/6/1990
Nationality: Egyptian
E-mail: noha.abdeltwab@aucegypt.edu
Phone: 01229484426
Address: 21-Tarek St. Shoubra
Registration Date: 1/10./2012
Awarding Date: 2017
Degree: Master of Science
Department: Mechanical Design and production Engineering
Supervisors:
Prof. Dr. Abdallah Wifi
Prof. Dr. Amal Kamal Esawi (American university in Cairo)



Examiners:
Prof. Dr. Ashraf Nassef (External examiner) (Mechanical Engineering department -American university in Cairo)
Prof. Dr. Iman Salah El-Din El-Mahallawi (Internal Examiner)
Prof. Dr. Abdalla Shaaban Wifi (Thesis main advisor)
Prof. Dr. Amal Kamal Esawi (Advisor) ((Mechanical Engineering department -American university in Cairo)

Title of Thesis:
CHARACTERIZATION, MODELING AND OPTIMIZATION OF WEAR BEHAVIOR OF DUAL MATRIX Al-CNT COMPOSITES

Key Words:
Wear, Carbon nanotube, Design of experiments, Optimization

Summary:
This thesis studies the tribological behavior of dual matrix Al-CNT composites where the optimum values of significant parameters are determined to decrease the wear rate and increase hardness. The study considers the wear behavior as affected by the main control parameters; 1) wt.% CNT content in reinforcement particles (C), 2) the mixing ratio between reinforcement particles to soft aluminum matrix (M), 3) sliding speed (S), 4) applied load (L) and 5) covered distance (D). The study also considers the hardness affected by the main control parameters; 1) wt.% CNT content in reinforcement particles (C), 2) the mixing ratio between reinforcement particles to soft aluminum matrix (M). Response surface methodology including statistical design of experiments, modeling and optimization is applied. The optimized multi objective model and the validating experiments show that optimal levels at minimum wear and maximum hardness can be reached at about (C) 5%, (M) 56%, (D) 0.6km, (L) 8N, and (S) 0.8m/s.

Acknowledgments

I would like to express my appreciation and thankfulness to my supervisors Prof. Dr. Abdalla Wifi and Prof. Amal Esawi for their limitless help and inspiration to present this thesis. Their widespread knowledge and great expertise supported me greatly to face difficulties and obstacles during my work.

I also express a great appreciation to the American University in Cairo, YOUSEF JAMEEL Science Research Centre and TRIBOLOGY AND SPARE PARTS Research Center in Cairo University where the experimental work was implemented.

I am very grateful to Eng. Ehab Salama, Eng. Ahmed Amin Genied and Eng. Hussien Abdelaziz Hegab whose support and motivation helped me to present this thesis.

My family is the source of tenderness and support all along. They are all the time there whenever I needed encouragement.

Dedication

To my family.....

Table of Contents

ACKNOWLEDGMENTS.....	I
DEDICATION	II
LIST OF TABLES.....	V
LIST OF FIGURES.....	VI
NOMENCLATURE	VIII
ABSTRACT	IX
CHAPTER 1 INTRODUCTION.....	1
1.1. WEAR.....	1
1.1.1. Wear Mechanisms.....	1
1.1.2. Characterization of wear mechanisms	3
1.2. LUBRICATION	3
1.3. METAL MATRIX COMPOSITES	4
1.4. POWDER METALLURGY.....	4
1.5. DESIGN OF EXPERIMENTS AND TAGUCHI APPROACH	5
1.5.1. Taguchi's robust design project steps.....	6
1.5.2. The response surface mathematical modeling (RSM)	9
1.6. OPTIMIZATION.....	9
1.6.1. Sequential Quadratic Programming.....	9
1.6.2. Multi objective Genetic Algorithm	10
1.7. THESIS OBJECTIVE.....	10
1.8. THESIS ORGANIZATION	11
CHAPTER 2 LITERATURE REVIEW	12
2.1. FABRICATION AL-CNT COMPOSITE	12
2.1.1. Roll bonding technique	12
2.1.2. Spark plasma sintering technique	12
2.1.3. High pressure torsion technique.....	13
2.1.4. Nano-Scale Dispersion.....	13
2.1.5. AL-CNT composites fabricated using mechanical alloying	14
2.2. DUAL ALUMINUM/ CARBON NANOTUBE MATRIX COMPOSITE	15
2.3. TRIBOLOGICAL EFFECT OF ADDING CNT IN METAL MATRIX COMPOSITE...	17
2.4. APPLICATIONS OF DOE ANALYSIS ON PIN ON DISK WEAR TEST.....	22
2.5. OPTIMIZATION	24
2.6. SUMMARY	24
CHAPTER 3 EXPERIMENTAL PROCEDURE AND DESIGN OF EXPERIMENTS.....	25
3.1. DUAL MATRIX AL-CNT COMPOSITE SYNTHESSES	25
3.1.1. Preparing of reinforcement matrix powder	25
3.1.2. Preparing dual matrix powder mixture	26
3.1.3. Consolidation of composite	27

3.1.4.	Sintering.....	28
3.1.5.	Hot Extrusion	28
3.1.6.	Machining of composite specimens	28
3.2.	MICROSTRUCTURE INVESTIGATION	28
3.3.	HARDNESS TEST	29
3.4.	PIN-ON-DISK TEST	29
3.5.	DESIGN OF EXPERIMENTS ACCORDING TO TAGUCHI APPROACH.....	30
3.5.1.	Hardness Investigation experiments Taguchi plan	30
3.5.2.	Wear Investigation experiment Taguchi plan	31
3.6.	SUMMARY	33
CHAPTER 4 RESULTS, STATISTICAL ANALYSIS AND MODELING.....		34
4.1.	MICROSTRUCTURE OF DUAL AL-CNT MATRIX COMPOSITE.....	34
4.2.	VICKERS HARDNESS INVESTIGATION	34
4.3.	HARDNESS RESULTS STATISTICAL ANALYSIS AND MODELING.....	37
4.3.1.	Analysis Table.....	38
4.3.2.	Proposed Mathematical Model for Vickers Hardness.....	38
4.4.	TRIBOLOGICAL INVESTIGATION.....	39
4.4.1.	Wear rate Results	39
4.5.	WEAR RESULTS STATISTICAL ANALYSIS AND MODELING	42
4.5.1.	Wear rate ANOVA.....	44
4.5.2.	Proposed Mathematical Model for wear rate	44
4.5.3.	Response surface and trend graphs	46
4.6.	COEFFICIENT OF FRICTION.....	49
4.7.	WORN SURFACE SEM INVESTIGATION	54
4.8.	SUMMARY	62
CHAPTER 5 OPTIMIZATION AND RESULTS VALIDATION.....		63
5.1.	OPTIMIZATION	63
5.1.1.	A single objective optimization of hardness and wear rate.....	63
5.1.2.	Multi objective functions to minimize wear rate and maximize Hardness	65
5.2.	RESULTS VALIDATION	67
5.2.1.	Validation of wear rate model.....	67
5.3.	SUMMARY	68
CHAPTER 6 CONCLUSION AND FURTHER WORK		69
6.1.	DISCUSSION	69
6.2.	FURTHER WORK.....	71
6.3.	SUMMARY	72
REFERENCES		73

List of Tables

Table 1-1: L ₈ OA and its linear graph [11].....	7
Table 1-2: Example of ANOVA Table	8
Table 2-1: Parameter and levels of Al6061T6/SiC/Al ₂ O ₃ wear investigation.....	22
Table 2-2: Parameters Investigation of Al/TiO ₂	23
Table 2-3: Process parameters with their different levels	23
Table 3-1: Weight ratio, amount of CNT, Al and ethyl alcohol amount.....	26
Table 3-2: Mixing ratio, CNT wt.% in reinforcement and the overall wt.% of CNT	27
Table 3-3: Hardness Investigation Factors Levels	31
Table 3-4: L ₉ OA orthogonal array	31
Table 3-5: Assignment of levels to control the parameters of wear investigation	31
Table 3-5: L ₂₇ OA Orthogonal array	32
Table 3-6: control variables and assigned array column	32
Table 3-7: experimental settings for the 27 experiments	33
Table 4-1: Hardness investigation results.....	36
Table 4-2: experimental settings, hardness and S/N ratio	37
Table 4-3: Effect of parameters in Vickers hardness	37
Table 4-4: ANOVA of S/N ratio maximum the better Hardness table	38
Table 4-5: model predicted value and relative error of Vickers Hardness	38
Table 4-6: the adequacy test results of the model	39
Table 4-7: Wear rate of dual Al-CNT composite.....	40
Table 4-8: Wear rate S/N ratio for different material composition and friction condition	42
Table 4-9: Effect of parameters in wear rate	43
Table 4-10: ANOVA table to S/N minimum the better	44
Table 4-11: experimental and predicted value of wear rate	44
Table 4-12: Average coefficient of friction of the 27 sample	49
Table 5-1: parameters levels and function values of optimum solution.....	66
Table 5-2: Validation hardness results	67
Table 5-3: Wear rate validation results	68

List of Figures

Figure 1-1: Sketch showing deformation at the points of real contact [1]	1
Figure 1-2: Two-body and three-body wear abrasion mechanisms [3].....	2
Figure 1-3: Mechanism of crash and debris formation in surface contact fatigue [4]	2
Figure 1-4: Fatigue cracks on the worn surface [3].....	2
Figure 1-5: Corrosion wear damage in silver-plated disconnect switch blade. [2].....	3
Figure 1-6: Schematic drawing of process of ball milling [10].....	4
Figure 1-7: Refinement of particle and grain sizes with milling time [10].....	5
Figure 1-8: Process diagram with input-output responses [11].....	5
Figure 1-9: Nominal the best Quality loss function (when the response level $y = T$, the quality loss is zero) [11]	6
Figure 1-10: Sequential Quadratic Programming [13].....	10
Figure 1-11: Flow chart of GA [14]	10
Figure 2-1: AL-CNT composite production using Roll-bonding technique [16].....	12
Figure 2-2: Schematic diagram HPT process [18]	13
Figure 2-3: Stress-strain curves in compression tests for MWCNT/Al composites [15]	14
Figure 2-4: Stress strain behavior of pure Dual Al-CNT 5-50% wt. tested	17
Figure 2-5: COF and wear amounts	19
Figure 2-6: Wear rate for Al6061 and Al606 of 1 wt% CNTs as a function of load	20
Figure 2-7: Wear rate of the Al-CNT composite 5 wt% CNT at different loads	21
Figure 2-8: Wear rate of the Al-CNT composite 5 wt% CNT at different speeds	21
Figure 2-9: The theoretical and experimental wear rate.....	23
Figure 3-1: Controlled atmosphere chamber Glove Box	25
Figure 3-2: a) Retsch® high energy planetary ball mill b) Milling Jars	26
Figure 3-3: Retsch® turbula mixer.....	26
Figure 3-4: FOSTER® hydraulic press	27
Figure 3-5: hardened tool steel die	27
Figure 3-6: LEO SUPRA 55-Field Emission Scanning Electron Microscope.....	28
Figure 3-7: Vickers hardness indentation.....	29
Figure 3-8: Macro Vickers Hardness Tester	29
Figure 3-9: Schematic diagram of ASTM G99-95 pin on disk apparatus.....	30
Figure 3-10: Plint TE/P indexing Pin-on-Disk Apparatus	30
Figure 3-11: Linear Graph for L9.....	31
Figure 3-12: Linear Graph L27OA.....	32
Figure 4-1: Dual Al-CNT composite microstructure of 4%wt. CNT and 75% Mixing ratio.....	34
Figure 4-2: Macro Vickers hardness	35
Figure 4-3: Relationship between Macro hardness and mixing ratio.....	35
Figure 4-4: Hardness experimental data representation	36
Figure 4-5: Macro Vickers hardness variation with overall CNT wt.% in dual Matrix.....	37
Figure 4-6: Hardness parameters main effects	38
Figure 4-7: Run experimental and Predicted Value Macro Hardness Analysis.....	39
Figure 4-8: Response surface of hardness plotted through derived model	39
Figure 4-9: Wear rate with different composition and friction condition	40
Figure 4-10: Wear Rate of different Friction Condition for different CNT wt. %.....	42
Figure 4-11: wear rate parameters effect graph.....	44

Figure 4-12: Model predicted values and experimental values of wear rate.....	45
Figure 4-13: Response surface of wear rate	46
Figure 4-14: wear rate vs. mixing ratio at different load.....	47
Figure 4-15: wear rate vs. mixing ratio at different speeds	48
Figure 4-16: wear rate vs. speed for different mixing ratio.....	48
Figure 4-17: wear rate vs. load for different mixing ratio	49
Figure 4-18: Average coefficient of friction at different wear condition	51
Figure 4-19: Average COF of different condition of different CNT wt.%	52
Figure 4-20: a) sample 1 (3%C, 25%M, S0.5m/s, L8N and D 0.6Km), b) sample 2(3%C, 50%M, S0.5m/s, L14N and D 1Km), c) Sample 3(3%C, 75% M, S0.5m/s, L 20N and D1.4Km), d) Sample 4(3%C, 25%M, S1m/s, L 8N and D1Km)	55
Figure 4-21: a) Sample 5(3%C, 25%M, S1m/s, L14N and D 1.4Km), b) Sample 6(3% C, 25%M, S1m/s, L14N and D1.4Km)	56
Figure 4-22: of sample 7(3% C, 50% M, S1.5m/s, L8N and D1.4Km)	56
Figure 4-23: a)Sample 8(3% C, 75% M, S1.5m/s, L14N and D0.6Km), b) sample 9 (3% C, 25% M, S1.5m/s, L20N and D1Km), c) sample 10(4%C, 25% M, S0.5m/s, L8N and D 0.6Km), d) sample 11(4%C, 50% M, S0.5m/s, L 14N and D 1Km).....	57
Figure 4-24: a) sample 12(4% C, 75% M, S0.5m/s, L20N and D1.4Km), b) Sample 13(4% C, 75% M, S1m/s, L8N and D 1Km), c) Sample 14(4% C, 25%M, S 1m/s, L14N and D1.4Km), d) sample 15(4% C, 25% M, S1m/s, L 20N and D 0.6Km).....	58
Figure 4-25: a) sample 16(4% C, 50% M, S1.5m/s, L8N and D1.4Km), b) sample 17(4% C, 75% M, S1.5m/s, L14N and D0.6Km), c) sample 18(4% C, 25% M, S1.5m/s, L20N and D 1Km), d) Sample 19 (5%C, 25%M, S0.5m/s, L8N and 0.6Km).....	59
Figure 4-26: a) sample 20(5% C, 50% M, S0.5m/s, L14N and D 1Km), b) sample 21(5% C, 75% M, S 0.5m/s, L20N and D 1.4Km), c) Sample 22 (5%C, 75%M, S1m/s, L8N and D1Km, d) sample 23(5% C, 25% M, S1m/s, L14N and D 1.4Km).....	60
Figure 4-27: a) Sample 24(5%C, 50% M, S1m/s, L20N and D0.6Km), b) Sample 25(5%C, 50%M, S1.5m/s, L8N and D1.4Km), c) Sample 26(5%C, 50%M, S1.5m/s, L14N and 0.6Km).....	61
Figure 4-28: Sample 27 (5%C, 25%M, S1.5m/s, L20N and D1Km).....	61
Figure 5-1: iteration and hardness value enhancement	64
Figure 5-2: wear rate values at different iteration	64
Figure 5-3: objective function value reached stability after 17 iteration.....	65
Figure 5-4: Pareto front graph for multi objective function of wear rate and hardness .66	
Figure 5-5: Hardness validation results	67
Figure 5-6: Wear rate validation results	68
Figure 5-7: Worn surface of sample of optimum parameters values (C5%-M57%- S0.8m/s-L8N-D0.6m).....	68

Nomenclature

- CNTs: Carbon Nano Tubes
- AL-CNT: Aluminum Carbon Nanotubes Composites
- SWCNT: Single wall carbon nanotube
- MWCNT: Multiple wall carbon nanotube
- MMCs: Metal Matrix Composites
- DOE: Design of experiments
- DOF: Degree Of Freedom
- SQP: Sequential Quadratic Programming
- GA :Genetic Algorithm
- CVD :chemical vapor deposition
- BPR: Ball to Powder Ratio
- PCA: Process control agent
- % wt. : weight ratio percent
- MA: Mechanical alloying
- COF: Coefficient of friction
- DOE: Design of Experiments
- SS: Statistical Sum
- MS: Mean Sum
- SEM: Scanning electron microscope
- TEM: Transmission electron microscope
- FEM: Force electron microscope
- RSM: Response Surface Methodology
- GA: Genetic Algorithm
- S/N: Signal To Noise Ratio
- OA: Orthogonal Array
- ANOVA: Analysis of Variance
- SS: Statistical Sum
- USL: Upper Specified Limit
- LSL: Lower Specified Limit
- NR: Nature Rubber
- SPD: severe plastic deformation
- HPT: high pressure torsion
- SPE : spark plasma extrusion
- HP: Hot Pressing
- SPS: Spark Plasma Sintering

Abstract

Metal – carbon nanotube (CNT) composites are developing materials with superior tribological behavior. A new dual matrix Al-CNT composite structure design manufactured by mechanical milling is investigated and characterized. A pin on disk wear test is conducted according to L27OA fractional factorial Taguchi orthogonal array. Wear behavior is investigated with different CNT wt.% content in reinforcement particles and different mixing ratio between reinforcement particles to AL matrix under different conditions of sliding speed, applied load and covered distance.

Statistical analysis (ANOVA of signal to noise ratio) and regression mathematical model of wear rate are conducted to study significance and effect of each parameter. Sequential quadratic programming approach is used to optimize single objective functions to minimize the wear rate and maximize the hardness. On the other hand, genetic algorithm is applied to the multi objective function to obtain parameters optimum values that minimize the wear rate and maximize the hardness of the composite material. An independent set of experiments is used to validate the optimization results.

Results show that increasing CNT wt.% and mixing ratio between reinforcement particles to soft Al particles lead to increase the hardness. Mixing ratio of reinforcement particles to soft Al matrix, sliding speed, and applied load are found to be the most significant parameters affecting on the wear rate. Experimental results show that lowest wear rate occurs at 3 CNT wt.%, mixing ratio 50%, covered distance 1.4Km, applied load of 8N, and sliding velocity 1.5m/s. The optimized multi objective model show that optimal values of minimum wear and maximum hardness occurs at 5 CNT wt.%, mixing ratio 56%, covered distance 0.6km, applied load of 8N, and sliding velocity 0.8m/s.

Chapter 1 Introduction

This chapter covers an introduction about wear mechanisms in metals and lubrication techniques. Metal matrix composites' properties are discussed and their production using powder metallurgy method. Finally response surface methodology including statistical design using Taguchi approach optimization algorithms is reviewed and thesis objective statement and thesis organization are presented.

1.1. Wear

Wear is the removal of material from solid surfaces in a solid-state contact where wear rate is the amount of material removed concerning time or distance. In this thesis wear rate is defined as mass loss per unit time. Wear resistivity is a measure of the resistivity of a body to lose material by wear, the reciprocal of wear rate.

1.1.1. Wear Mechanisms

a. Adhesive

When two solid materials are in rubbing contact, adhesion takes place in the mating faces, and debris are pulled off from one face to stick to the other. These debris may fall off the face on which they are stuck and either be relocated to the original face or be loose wear particles as shown in Figure 1-1 [1]

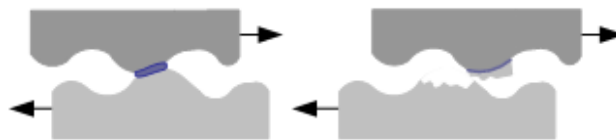


Figure 1-1: Sketch showing deformation at the points of real contact [1]

If two similar materials are in friction, both faces will be roughened, the asperities on both surfaces scratch the other, so fast wear will take place. But if two unlike materials are rubbing each other, shear will be subjected, and fine fragments of the ductile material will be pulled off and stick to the surface of the harder material. Accordingly, damage will be mainly restricted to the ductile surface, so that if adhesion is the leading friction mechanism, it is preferable to slide dissimilar materials together mainly if they are soft materials.[2]

b. Abrasive

Abrasive wear occurs due to hard asperities enforced against and sliding along a surface. Figure 1-2Error! Reference source not found.. Shows that the asperities may either be surrounded in the counter surface (two-body mechanism), or gone within the rubbing region (three-body mechanism).[3]