

CHARACTERIZATION OF MARBLE WASTE AND
GRAPHENE OXIDE REINFORCED LM6 ALUMINUM
COMPOSITES FABRICATED BY STIR SQUEEZE
CASTING PROCESS

BY

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ABSTRACT

Applications of aluminum matrix composites (AMC) in automobile and aerospace industries have been increasing due to their attractive properties such as lightweight, high specific modulus, stiffness and good corrosion resistance. However, the cost is the key factor in making composite materials due to the higher cost of reinforcement materials. The reuse of industrial waste, which is otherwise dumped as landfills, can reduce the composite production costs and the pollution levels posed by landfills. Therefore, this research aims to use industrial waste and graphene oxide as reinforcement phase materials to develop new and cost-effective AMC materials. In this study, LM6 aluminum alloy was used as a matrix phase, and industrial waste material of marble waste (MW) and a distinct novel material of graphene oxide (GO) with 5 wt % of each reinforcement were used for composite development. The hybrid stir and squeeze casting method was used to develop the new composites with the use of optimum casting process parameters derived from the DoE (Taguchi coupled with ANOVA) technique, integrated with Statistical Process Control (SPC) Excel software. Mechanical testing of developed AMC such as tensile, impact and hardness were performed according to ASTM- E8/EM8-13, ASTM- E23-16b and ASTM-E18 standards, respectively. The microstructural and morphological analysis were done using optical microscopy (OM), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD) techniques. Further, fractography analysis was performed on the tensile fractured surface of both composite materials using SEM equipped with JED-2300 Analysis Station Plus. Finally, a cost model for the composite fabrication process was developed and compared with fly ash and SiC-reinforced composite materials in order to draw valuable insights on the production cost of the new composites. From the evaluation of the DoE-ANOVA results, it was confirmed that stirring speed of 600 rpm, stirring time of 10 min, and melting temperature of 740 °C have had a significant influence on the response variables of LM6 composites. Mechanical test results showed that there was an increase of 16% in UTS for LM6+5%MW and 41.8% for LM6+5%GO as compared to LM6 aluminum alloy. For hardness property values, around 25.34% increase was witnessed in both composites equally compared to LM6 with the hardness value of 75.52, 94.66 and 94.68 (HRF) for LM6, LM6+5%MW and LM6+5%GO respectively. The enhanced mechanical properties were duly supported by the microstructures of both composites captured using OM, SEM, EDS and XRD. The degree of strengthening of LM6 aluminum alloy was determined by micrographs, chemical composition, particle size, and concentration of reinforcing phase materials. Subsequently, fractography analysis on tensile fractured composite samples showed no or minor cracks, thus observed that fracture was caused by matrix cracking, matrix cavitation, interface separation and rupture. The cost analysis demonstrated that the total reinforcement cost decreased by 44% due to the use of natural and industrial waste materials compared to fly ash and SiC reinforced composite materials. The current research reflected the concept of circular economy as applicable to the LM6 composites to maximize the usefulness of the waste materials. Therefore, the research concluded that MW and GO are prospective alternate candidates as reinforcements in the LM6 matrix phase to develop composites with enhanced mechanical properties suitable for automobile, aerospace and maritime industries.

خلاصة البحث

تتزايد تطبيقات مركبات مصفوفة الألومنيوم (AMC) في صناعات السيارات والطيران بسبب خصائصها الجذابة مثل الوزن الخفيف والمعامل النوعي العالي والصلابة والمقاومة الجيدة للتآكل. ومع ذلك ، فإن التكلفة هي العامل الرئيسي في صنع المواد المركبة بسبب ارتفاع تكلفة مواد التعزيز. يمكن أن تؤدي إعادة استخدام النفايات الصناعية ، التي يتم إلغاؤها كمداخن للقمامة ، إلى تقليل تكاليف الإنتاج المركبة ومستويات التلوث التي تشكلها مدافن النفايات. لذلك ، يهدف هذا البحث إلى استخدام النفايات الصناعية وأكسيد الجرافين كمادة لمرحلة تقوية لتطوير مواد AMC جديدة وفعالة من حيث التكلفة. في هذه الدراسة ، تم استخدام سبائك الألومنيوم LM6 كمرحلة مصفوفة ، وتم استخدام مادة النفايات الصناعية من نفايات الرخام (MW) ومادة جديدة متميزة من أكسيد الجرافين (GO) مع 5٪ بالوزن من كل تقوية لتطوير المركب. تم استخدام طريقة الصب المجهن والضغط لتطوير المركبات الجديدة باستخدام معلمات عملية الصب المثلى المستمدة من تقنية DoE (Taguchi المقتترنة بـ ANOVA) ، المدججة مع برنامج Excel للتحكم في العمليات الإحصائية (SPC). تم إجراء الاختبارات الميكانيكية لـ AMC المطورة مثل الشد والتأثير والصلابة وفقاً لمعايير ASTM- E8/EM8-13 و ASTM- E23-16b و ASTM-E18 ، على التوالي. تم إجراء التحليل المجهرى والمورفولوجي باستخدام تقنيات المجهر الضوئي (OM) والمسح المجهرى الإلكتروني (SEM) والتحليل الطيفي المشتت للطاقة (EDS) وتقنيات حيود الأشعة السينية (XRD). علاوة على ذلك ، تم إجراء تحليل الكسور على السطح المكسور الشد لكلا المادتين المركبتين باستخدام SEM المجهز بمحطة تحليل JED-2300 Plus. أخيراً ، تم تطوير نموذج تكلفة لعملية التصنيع المركب ومقارنته بنموذجين آخرين معززين مثل الرماد المتطاير والمواد المركبة SiC من أجل استخلاص رؤية قيمة حول تكلفة إنتاج المركبات الجديدة. من تقييم نتائج DoE-ANOVA ، تم التأكيد على أن سرعة التحريك البالغة 600 دورة في الدقيقة ، ووقت التحريك 10 دقائق ، ودرجة حرارة الانصهار البالغة 740 درجة مئوية كان لها تأثير كبير على متغيرات الاستجابة لمركبات LM6. أظهرت نتائج الاختبارات الميكانيكية أن هناك زيادة بنسبة 16٪ في UTS لـ LM6 + 5 و 41.8٪ لـ GO/LM6 + 5 مقارنة بسبيكة الألومنيوم LM6. بالنسبة لقيم خصائص الصلابة ، لوحظ زيادة حوالي 25.34٪ في كلا المركبين بالتساوي مقارنة بـ LM6 بقيمة صلابة 75.52 و 94.66 و 94.68 (HRF) لكل من LM6 و LM6 + 5 و GO/LM6 + 5 على التوالي. تم دعم الخواص الميكانيكية المحسنة على النحو الواجب من خلال الهياكل الدقيقة لكلا المركبين اللذين تم التقاطهما باستخدام OM و SEM و EDS و XRD. لوحظ أن الصور المجهرية والتركيب الكيميائي وحجم الجسيمات وتركيز مواد مرحلة التعزيز تحدد درجة تقوية سبائك الألومنيوم LM6. بعد ذلك ، أظهر تحليل الكسور على عينات المركبة المكسورة الشد عدم وجود شروخ صغيرة أو شقوق طفيفة ، وبالتالي لوحظ أن الكسر كان ناتجاً عن تكسير المصفوفة ، وتجويف المصفوفة ، وفصل السطح البيني ، والتمزق. أظهر تحليل التكلفة أن التكلفة الإجمالية للتعزيزات انخفضت بنسبة 44٪ بسبب استخدام مواد النفايات الطبيعية والصناعية مقارنة بالرماد المتطاير والمواد المركبة المقواة بالكربيد. عكس البحث الحالي مفهوم الاقتصاد الدائري كما ينطبق على المركبات LM6 لتعظيم فائدة مواد النفايات. لذلك ، خلص البحث إلى أن MW و GO هما مرشحان بديلان محتملان كتعزيزات في مرحلة مصفوفة LM6 لتطوير مركبات ذات خواص ميكانيكية محسنة مناسبة للسيارات والطيران والصناعات البحرية.

APPROVAL PAGE

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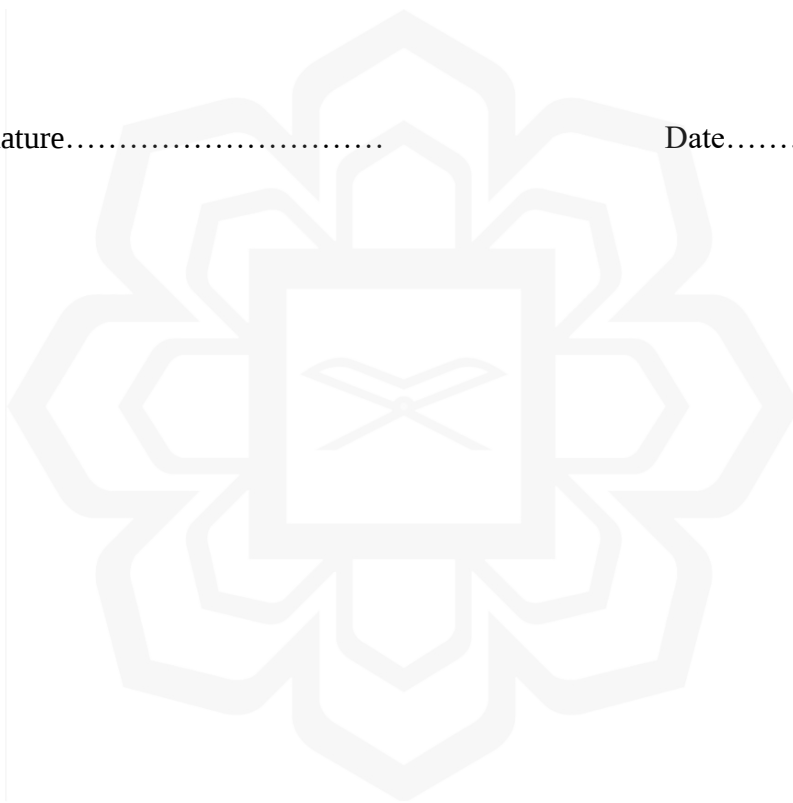
DECLARATION

I hereby declare that this dissertation is the result of my own investigations, except where otherwise stated. I also declare that it has not been previously or concurrently submitted as a whole for any other degrees at IIUM or other institutions.

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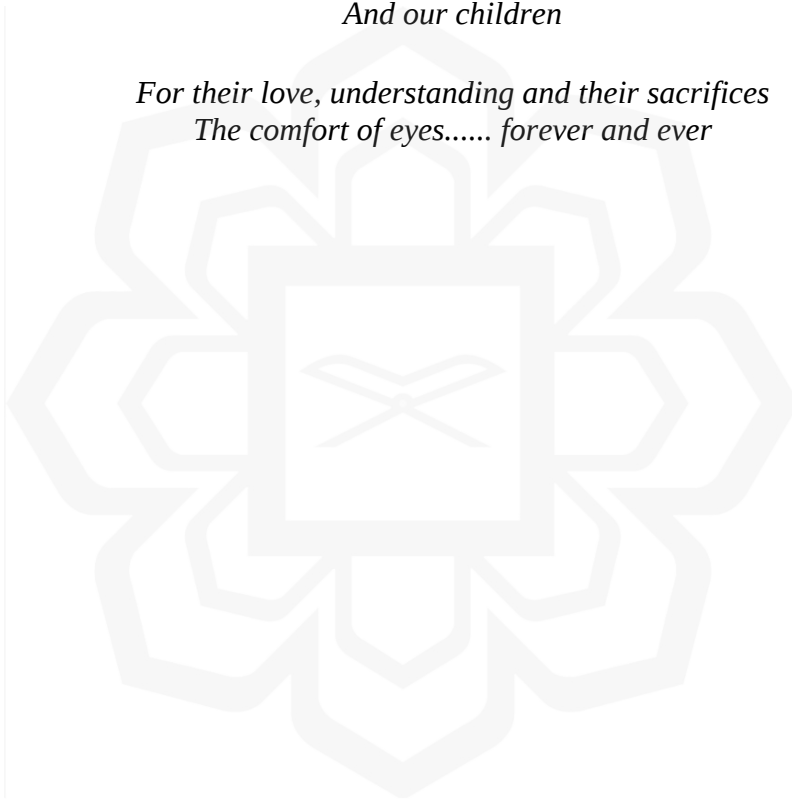
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My parents, they are forgiven God willing,

*My sweetheart wife
And our children*

*For their love, understanding and their sacrifices
The comfort of eyes..... forever and ever*



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TABLE OF CONTENTS

Abstract.....	ii
Approval Page.....	iv
Declaration.....	v
Copyright Page.....	vi
Acknowledgements.....	viii
Table of contents.....	ix
List of Tables.....	xii
List of Figures.....	xiv
List of Symbols, Abbreviations and Notations.....	xviii
CHAPTER ONE: INTRODUCTION	1
1.1 Background.....	1
1.2 Problem Statement and Its Significance	5
1.3 Research Philosophy.....	8
1.4 Research Objectives.....	8
1.5 Research Methodology	9
1.6 Research Scope.....	11
1.7 Thesis Organization	13
CHAPTER TWO: LITERATURE REVIEW	15
2.1 Introduction.....	15
2.2 Aluminum Matrix Materials	19
2.2.1 Aluminum Silicon Alloys	20
2.2.2 LM6 Alloy.....	22
2.2.2.1 Physical and Mechanical Properties of LM6 Alloy.....	23
2.2.2.2 Castability of LM6 alloy.....	24
2.3 AMC With Ceramic Reinforcement.....	24
2.4 Strengthening Mechanisms In AMCS	28
2.5 Industrial Waste Reinforcements.....	31
2.5.1 Refinery Waste (Spent Alumina Catalyst).....	31
2.5.2 Red Mud.....	32
2.5.3 Fly Ash.....	33
2.5.4 Electric Arc Furnace Dust.....	34
2.5.5 Slag.....	35
2.5.6 Waste Glass.....	36
2.5.7 Mines Waste.....	37
2.5.8 Marble Waste	38
2.6 Graphene Building Materials.....	39
2.7 Fabrication of Aluminum Matrix Composites.....	41
2.7.1 Solid Phase Processing Technique.....	42
2.7.2 Liquid Phase Processing Techniques.....	43
2.7.2.1 Liquid Metal Infiltration	44
2.7.2.2 Stir Casting	45
2.7.2.3 Stir Squeeze Casting Process	46
2.7.3 Semi-Solid Phase Processing Technique	47

2.7.3.1 Compocasting Process	47
2.7.3.2 In Situ Processing	48
2.7.3.3 Spray Deposition Method	49
2.8 Influence Of Process Parameters In Stir Squeeze Casting	50
2.8.1 Effect of Squeeze Pressure.....	50
2.8.2 Effect of Stirring Speed.....	50
2.8.3 Effect of Stirring Time	51
2.8.4 Effect of Particle Size and Volume Variation.....	51
2.8.5 Effect of Metal Casting Temperature.....	51
2.8.6 Optimization Parameter for Stir Squeeze Casting	52
2.9 Porosity In Alumnium Composite Materials.....	54
2.10 Methods For Optimizing Process Parameters.....	56
2.11 Mechanical Characteristicof AMC	59
2.12 Failure Characteristics of AMC.....	62
2.12.1 Failure Due to Reinforcement Cracking	63
2.12.2 Failure Due to Void Nucleation	64
2.12.3 Failure Due to Debonding.....	65
2.12.4 Failure Due to Matrix Cavitation.....	66
2.13 Cost Analysis	67
2.14 Summary.....	69
CHAPTER THREE: EXPERIMENTAL DETAILS.....	71
3.1 Introduction.....	71
3.2 Laboratory Equipment Used.....	72
3.3 Design of Laboratory Experiments.....	74
3.4 Constituent Materials.....	77
3.4.1 Matrix Material	77
3.4.2 Reinforcement Materials.....	78
3.5 Fabrication of Mw and Go Reinforced LM6 Composites	82
3.6 Sample Preparation of AMC	86
3.7 Characterization of Composite Samples.....	87
3.7.1 Mechanical Testing	88
3.7.2 Microstructural and Morphological Examination.....	91
3.8 Composite Production Cost Analysis	95
3.9 Summary.....	97
CHAPTER FOUR: RESULTS AND DISCUSSION.....	98
4.1 Introduction.....	98
4.2 Mechanical Characterisation of Composites	98
4.2.1 Tensile Properties.....	98
4.2.2 Hardness Properties.....	103
4.2.3 Impact Properties	104
4.3 Physical Characterization of Composites	106
4.3.1 Density and Porosity	106
4.4 Discussion on Doe Results	110
4.5 Microstructural Morphological Characterization	113
4.5.1 Optical Microscopy.....	113
4.5.2 Scanning Electron Microscopic Results	115
4.5.2.1 LM6 Aluminum Alloy	115

4.5.2.2 LM6+5%MW Composite	118
4.5.2.3 LM6+5%GO Composite.....	120
4.5.3 XRD Analysis LM6 Alloy and LM6 Composites	124
4.5.4 Fractography Analysis	126
4.5.5 Fracture Surface Characteristics	126
4.5.5.1 LM6+5%MW.....	126
4.5.5.2 Fracture Surface Characteristics of LM6+5%GO	128
4.6 Cost Analysis of Composite Fabrication	129
4.6.1 Fixed Costs.....	130
4.6.2 Variable Costs	130
4.6.2.1 Matrix Materials Costs.....	130
4.6.2.2 Reinforcing Material Costs	131
4.6.2.3 Storage Costs	132
4.6.2.4 Environmental Costs.....	132
4.6.3 Total Cost of Manufacturing of LM6 Composites.....	132
4.7 Circular Economy For LM6 Composites	135
4.8 Summary	136
CHAPTER FIVE: CONCLUSION AND RECOMMENDATION	138
5.1 Conclusion	138
5.2 Recommendations For Future Research.....	140
5.3 Contribution To Knowledge.....	141
REFERENCES.....	143
LIST OF PUBLICATIONS.....	157
APPENDICES	143
APPENDIX A: Microstructural Morphology of Lm6, Lm6+5%Mw and LM6+5%Go Composites.	158
APPENDIX B: Tensile Fracture Surface of Lm6+5%Mw and LM6+5%Go Composites	164
APPENDIX C: EDS & XRD of LM6, LM6+5%MW and LM6+5%GO Composites	166
APPENDIX D: Tensile of LM6, LM6+5%MW and LM6+5%GO.....	170
APPENDIX E: Hardness of LM6, LM6+5%MW and LM6+5%GO	171
APPENDIX F: Impact Property of LM6, LM6+5%MW and LM6+5%GO.....	172
APPENDIX G: Density and Porosity of LM6, LM6+5%MW and LM6+5%GO	173
APPENDIX H: Desgin of Exprement	174
APPENDIX I: Cost Analysis of Composite Fabrication	176

LIST OF TABLES

Table 1.1	Comparison of Estimated Material Parameters for Al and AMC	3
Table 2.1	Nationally Recognized Designations for Al-Si alloys (LM6)	22
Table 2.2	Physical and Mechanical Properties of LM6 Aluminum Alloy	24
Table 2.3	Casting Characteristics of LM6	24
Table 2.4	AMC Materials with Various Reinforcement, Properties and Applications	26
Table 2.5	Strengthening Modes of AMCs	29
Table 3.1	Levels of The Parameters Used in The Experiment of Hybrid Casting Process	74
Table 3.2	Experimental Design Matrix for L9 Orthogonal Array	75
Table 3.3	DoE - Experimental Conditions	76
Table 3.4	Chemical Composition of LM6	78
Table 3.5	Mechanical Properties of LM6	78
Table 3.6	Chemical Composition of Marble Wastes	81
Table 3.7	Specification of GO Powder	82
Table 3.8	Chemical Composition of GO Powder	82
Table 3.9	General Formulation Scheme for Composites Fabrication	84
Table 3.10	Process parameters of Stir Squeeze Casting Process	85
Table 3.11	Impact Machine Specifications	90
Table 4.1	UTS and Ductility Values of Samples from Tensile Testing	99
Table 4.2	Results of density and porosity of samples A, B and C	108
Table 4.3	ANOVA Design Table Based on All Factors and Interactions	110
Table 4.4	Factors and Responses from DoE Analysis	111



LIST OF FIGURES

Figure 1.1 Basic Factors Relative to Cost for A Material	2
Figure 1.2 Block Diagram Show the Flow of the Thesis	12
Figure 2.1 2020 Corvette Stingray Car	16
Figure 2.2 LCA TEJAS Indian Navy (2018-19) (Rajagopal, 2018)	17
Figure 2.3 Aircraft A380 Fuselage Structure (Prasad Rambabu et al., 2017)	17
Figure 2.4 Cutting Tools	18
Figure 2.5 Aluminum Composite Panels	18
Figure 2.6 Industrial Safety Shoes	19
Figure 2.7 Equilibrium Diagram of Aluminum-Silicon Alloys	21
Figure 2.8 Microstructure of Al-Si alloys, (a) Hypoeutectic (1.65-12.6 wt. % Si) 150x. (b) Eutectic (12.6% Si). 400x, (c) Hypereutectic (> 12.6% Si) 150x.	22
Figure 2.9 Microstructure of LM6 Alloy	23
Figure 2.10 Optical microstructure and SEM images of SAC as reinforcement	32
Figure 2.11 SEM and Optical Microstructure of Red Mud as a Reinforcement	33
Figure 2.12 SEM Image of Fly Ash Particles, Along With Size Representation and LM25 Ingots	34
Figure 2.13 Optical Microstructure and SEM Images of EAFD as Reinforcement	35
Figure 2.14 Optical Microstructure and SEM Images of Slag as Reinforcement	36
Figure 2.15 SEM Images of Waste Glass as Reinforcement	37
Figure 2. 16 SEM Images of Mines Waste as Reinforcement	38
Figure 2.17 Strategies of Graphene Oxide by Hummer Method	39
Figure 2.18 (a, b and c) SEM Al ₅ Mg Alloy Powder Micrographs, (d and e) GO, and (f) TEM GO Image	41
Figure 2.19 AMC fabrication using a powder metallurgical method	43

Figure 2.20 (a) Liquid Metal Infiltration Process, (b) Pressure Infiltration Process	44
Figure 2.21 Stir Casting Process	45
Figure 2.22 Stir Squeeze Casting Setup	47
Figure 2.23 Compocasting of AMC	48
Figure 2.24 Spray Deposition Process	49
Figure 2.25 Recommended Stir-Squeeze Process Parameters	54
Figure 2.26 Stress-Strain Curve of Al and Al Based Composite with Various	62
Figure 2.27 SEM of Boron Carbide Cracking	64
Figure 2.28 SEM Image of Fracture Surface Scrap Aluminum Alloy Wheel and Al_2O_3	65
Figure 2.29 Interfacial Debonding of Matrix Reinforcement	66
Figure 2.30 Cross-Section Images Of The Fractured Specimen: (A) General View; (B) Enlarged View Of The Fractured Surface; (C) Enlarged View Of The Short Cracks, and (D) Fractured Surface Predicted By The Microstructure-Based Model	67
Figure 3.1 Hacksaw for Cutting Cast LM6	77
Figure 3.2 Cleaning The LM6 Pieces	78
Figure 3.3 Marble Wastes in The Landfills	79
Figure 3.4 Vibrating Sieve Analyser	80
Figure 3.5 MW Particles	80
Figure 3.6 GO Powder	81
Figure 3.7 Cross-Sectional View of Stir Squeeze Casting Machine	83
Figure 3.8 Stir Squeeze Casting Machine Photograph	83
Figure 3.9 Molten Aluminum Matrix Composite	85
Figure 3.10 Hydraulic Pressing Method for AMC Production	85
Figure 3.11 AMC samples (a) LM6 (b) LM6+5%MW and (c) LM6+5%GO	86
Figure 3.12 AMC Samples (a) LM6 (b) LM6+5%MW (c) LM6+5%GO Wire Cut With WEDM	87

Figure 3.13	Tensile Testing Specimen Dimensions (as per ASTM- E8/EM8-13)	88
Figure 3.14	Principle of Rockwell Hardness Testing and (b) Apparatus and Samples	89
Figure 3.15	(a) Impact Testing Apparatus (b) Specimen Dimensions (as per ASTM- E23-16b)	90
Figure 3.16	Density Measuring Setup	91
Figure 3.17	Mounting Materials	92
Figure 3.18	Grinding & Polishing Machine	92
Figure 3.19	Polishing Steps (a) 9 μm , (b) 3 μm , (c) 1 μm	93
Figure 3.20	Optical Microscope	94
Figure 3.21	Cross-sectional View of SEM	94
Figure 3. 22	Cost Elements of Composite Production	96
Figure 4.1	Stress-Strain Curves for 3 Types of Samples A, B and C	99
Figure 4.2	UTS Values of LM6 Composites of Five Different Reinforcement Materials (with 5% Reinforcement). 1. ZrO ₂ , 2. Glass Particles, 3. Redmud, 4. & 5. MW & GO (Current Research)	102
Figure 4.3	Results of Rockwell Hardness Testing	103
Figure 4.4	Energy Absorbed by Samples During Impact Testing	105
Figure 4.5	Experimental and Theoretical Density Values for Samples A, B and C	108
Figure 4.6	% Porosity Values for Samples A, B and C	108
Figure 4.7	Response Plots for UTS (a) X vs Y (b) X vs Z (c) Y vs Z (d) All Plots	112
Figure 4.8	Optical Micrographs (200X) (a) LM6 (b) LM6+5%MW and (c) LM6+5%GO	115
Figure 4.9	SEM Image of LM6	116
Figure 4.10	EDS Map of LM6	116
Figure 4.11	EDS Image of LM6	117
Figure 4.12	SEM Image of LM6+5%MW Composite	118
Figure 4.13	EDS Map of LM6+5%MW Composite	119

Figure 4.14 EDS Image of LM6+5%MW Composite	119
Figure 4.15 SEM Image of LM6+5%GO Composite	121
Figure 4.16 EDS Map of LM6+5%GO Composite	122
Figure 4.17 EDS Image of LM6+5%GO Composite	123
Figure 4.118 XRD Images of (a) LM6, (b) LM6+5%MW and (c) LM6+5%GO Composite	125
Figure 4.19 SEM Micrograph of Tensile Fractured Surface of LM6+5%MW Composite	127
Figure 4.20 (a) Visible Crack in LM6+5%MW Sample (b) Unfragmented MW Particles in LM6 Matrix	127
Figure 4.21 SEM Micrograph of Tensile Fractured Surface of LM6+5%GO Composite	129
Figure 4.22 Total Cost (in 1000) of Manufacturing LM6 Composites (in USD)	134

LIST OF SYMBOLS, ABBREVIATIONS AND NOTATIONS

Al	Aluminum
Al ₂ O ₃	Aluminum oxide
AMC	Aluminum matrix composite
AMMC	Aluminum metal matrix composites
B ₄ C	Boron carbide
BN	Boron nitride
CMCs	Ceramic matrix composites
CNT	Carbon nanotubes
Cu	Copper
HB	Brinell hardness
Mg	Magnesium
MMCs	Metal matrix composites
Si	Silicon
Si ₃ N ₄	Silicon nitride
SiC	Silicon carbide
SiO ₂	Silicon dioxide
MW	Marble wastes
LM6	Aluminum alloy A413
GO	Graphene oxide
Zn	Zinc
SAAW	Scrap aluminum alloy wheels
SAC	Spent aluminum catalyzes
WEDM	Wire electrical discharge machining
CRT	Cathode ray tube
EAFD	Electric arc furnaces dust
WS ₂	Tungsten disulfide
FW	Friction welding
FRP	Fibre-reinforced plastic
SEM	Scanning electron microscope
EDS	Energy dispersive x-ray analysis
UTS	Ultimate tensile strength
TCM	Total cost modelling
PM	Powder metallurgy
SQU	Sultan Qaboos University
ASTM	American Society for Testing and Materials

CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND

In light of varying design criteria and threshold values of crucial design parameters due to dynamic environmental and operating conditions, any engineering system demands its structures and components to be made up of adequately strong materials. A single metal/alloy fails to meet all the specifications and requirements in a harsh engineering environment. This aspect stimulates the need for developing new materials possessing specific unique properties to meet or exceed the engineering requirements and overcome technical challenges.

The development of innovative materials is almost like a panacea for all mission-critical problems and requirements. However, innovation lies in how the new materials are developed without much complications in processing, infrastructure, life expectancy (longevity) and cost-effectiveness. In the aerospace, transportation, automotive, and construction industries, there has been a growing demand for newer, stronger, and stiffer yet lightweight materials over the last few decades. Composite materials are produced largely to meet the growing technological needs of the automotive and aerospace sectors. (Chawla et al., 2013). Materials scientists constantly explore ways and means by which innovative and advanced materials with enhanced material properties can be developed to meet the ever-changing requirements.

Because of its improved mechanical and physical qualities, composite materials have proven to be a viable alternative material to many conventional metals and alloys since their inception. Composite materials are multiphase materials made up of metallic and nonmetallic materials that are immiscible within each other. The composites have

more enhanced strengths and moduli compared to the properties of individual constituent materials. Thus, applications of composite materials have been gradually growing, constantly entering and conquering new markets. Modern composite materials make up a substantial proportion of the demand for engineered materials, ranging from consumer goods to specialized niche applications. Types of composites include polymer matrix-based, ceramic matrix-based and metal matrix-based composites (Rathod, Kumar & Jain, 2017; Sommers et al., 2010) Figure 1.1 shows the essential factors relative to the cost of the material.

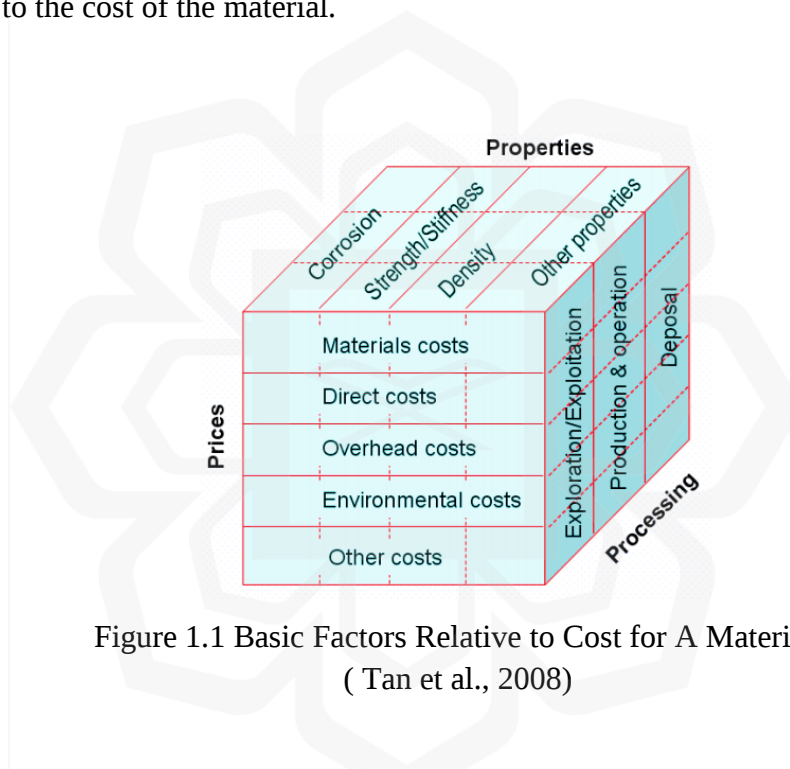


Figure 1.1 Basic Factors Relative to Cost for A Material
(Tan et al., 2008)

Due to the challenges of materials inspection, problem-solving, and constituent material reuse, processing and manufacturing composite components are more expensive with present technology. The cost, qualities, and availability related to the reinforcing phase would also contribute to the greater overall cost of creating the composite. It is estimated that the average price of aluminum alloys is 6 USD/kg, and the average price of composites is 80 USD/kg (Tan et al., 2008). For example, in a typical aircraft application (wing part), Table1.1 shows a comparison between the use

of conventional aluminum alloys and AMC materials in terms of costs and component weights. Although there is a significant weight reduction (86%) and reduction in the number of pieces used (64%), it is obvious that the cost of composites has nearly doubled (93%) and is no longer comparable to that of aluminium alloys. However, increasing the mechanical qualities of composites will result in lower operating costs over time and a quicker rate of return on investment (Tan et al., 2008).

Table 1.1 Comparison of Estimated Material Parameters for Al and AMC

Materials used	Number of parts	% Decrease in the number of parts	Weight in kg	% Decrease in weight	Estimated Cost \$	% Increase in cost
Aluminum	117	64%	12959.35	86%	91857.08	93%
AMC	42		1873.30		178141.63	

Therefore, it is crucial for high-intensive composite users to carefully select the materials before pressing them into manufacturing. Researchers continue to give their best to reduce the cost of composites through a range of solutions, including the reduction in the costs of constituent phase materials besides designing the processes efficiently. Several approaches could be taken to reduce composites' cost, such as single-step mixing, opting for selective reinforcements, and using cheaper reinforcements.

Because of their superior physical, mechanical, and tribological properties compared to base alloys, the applications of aluminum matrix composite materials in the automotive and aerospace industries are expanding rapidly. Because of their lower density, better wear and corrosion resistance, high strength to weight ratio, good

formability, high hardness, high thermal shock resistance, high modulus, and high fatigue strength, composite materials with metal matrix materials, such as aluminum or magnesium, are finding widespread use in a variety of industries. In the automobile industry, they are used in various parts such as car bodies, pistons, valves, engine blocks, brakes, etc. Aluminum-based composites reinforced with micro/nano SiC, Al₂O₃, B₄C, TiB₂, ZrO₂, SiO₂, and graphite particles alter the microstructure, resulting in superior mechanical and physical properties suitable for automotive/aerospace applications (Koli, Agnihotri & Purohit, 2015).

Waste recycling and waste reuse are the results of the creative and innovative thinking of active researchers. These two phenomena are critical attributes of any organization that wishes to remain competitive and self-reliant in the dynamic market. For example, the Sultanate of Oman is surrounded by continuous mountains, potential natural minerals and metals such as Copper, Gold, Silver, Chromium, Lead, Nickel, Manganese and Zinc, etc. Further, the country is renowned for exporting high-quality non-metallic minerals such as marbles, granite and glass materials. Industrial wastes resulting from processing these materials are dumped in landfills. A rough estimate reports that an average of 1.6 million tons of solid waste were dumped as landfills in 2010 (Hafidh A. et al., 2019). Marble waste (MW) can be one of the potential materials for developing composite, which is also being dumped in landfills.

In summary, the costs involved in preparatory and further treatment processes for the reinforcing phase material also compound the total cost of manufacturing composites and reinforcing materials. Researchers strive hard to minimize the cost incurred in producing AMC using various strategies and techniques, including lowering the amount of reinforcement materials utilized or using low-cost reinforcing materials.

A new class of reinforcing additive called Graphene Oxide (GO), a strong and abundant mono-atomic layered substance from the graphite family, provides good mechanical properties at less wt % reinforcements. GO has unique properties such as lightweight, transparency, and superior mechanical, thermal, and hardness properties due to its honeycomb structure, making it valuable in a range of areas (Nieto et al., 2017). On the other hand, the land-fill material is a challenging issue for the government because of limited land availability and adverse environmental and public health impacts. One of the potential remedies for reducing the costs is identifying low-cost reinforcing materials without impairing the composites' desirable properties and quality. The present attempt aims to solve two issues with one solution, using industrial waste materials as a reinforcing phase in AMC, which can be used in automotive applications. The issues include the higher cost of developing AMC and environmental issues, and greenhouse gas emission due to the landfill of the industrial wastes, which are addressed in this research.

1.2 PROBLEM STATEMENT AND ITS SIGNIFICANCE

Automotive and aerospace industries demand new-age engineering materials to meet the strength, tribological and cost factors. Compared to conventional metals and alloys, composites can meet the requirements with cost challenges due to the higher development cost of composites. However, there are various composites in hand for such applications with a wide range of properties. These properties are achieved through constituent materials (both matrix and reinforcement depend on the field application), whereas the availability of the reinforcement is the key concern as it determines the significant development costs. Therefore, there is a need to identify and select new reinforcement phase materials that can reduce the composites manufacturing costs

without sacrificing the materials' performance properties. Moreover, the reinforcement phase material from a waste source can reduce the cost and solve environmental problems posed by such waste materials.

A technological and environmentally sustainable approach to solid industrial waste management, disposal, reuse, and recycling has emerged as one of the world's most pressing solutions to industrial waste. Most of the wastes are dumped into landfills which take up much space and are a significant source of greenhouse gas emissions. In Oman, for example, all wastes from the marble industry are disposed of as landfills, thus polluting the environment. To date, only a few engineered land-fill sites remain among the Sultanate's more than 300 dump sites (Qureshi et al., 2018). However, MW is a promising and suitable candidate reinforcement material for the development of composites and reduce aluminum composite materials production costs because it will add no any additional costs and can contribute to solving the environmental pollution problems.

The strength of composite materials depends on several factors such as manufacturing, the existence of porosity, properties of constituent materials and wt % reinforcement. When unconventional reinforcing materials (viz. industrial or natural wastes) are used in the stir casting process, it often results in a higher porosity level in the composites, leading to inferior mechanical and physical properties(Arunachalam et al., 2019). Hence, the shift from the conventional stir casting process to a hybrid manufacturing process such as stir squeeze casting would be a promising solution for the current problem as the squeeze pressure exerted during stirring operation for the cessations of inter-atomic voids and pores of the materials, resulting in a reduction of the porosity after solidification.