UNIVERSITI PUTRA MALAYSIA

ZIRCONIUM OXIDE NANOPARTICLE-REINFORCED ALUMINIUM ALLOY (AA7075) MATRIX COMPOSITES VIA HOT EXTRUSION AND EQUAL CHANNEL ANGULAR PRESSING

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By

AL RUBAIAWI HUDA MOHAMMED SABBAR

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

June 2022

DEDICATION

TO
My father
My mother
My brother & sisters
My family
(My husband, Ghadeer & Mahdi)

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

ZIRCONIUM OXIDE NANOPARTICLE-REINFORCED ALUMINIUM ALLOY (AA7075) MATRIX COMPOSITES VIA HOT EXTRUSION AND EQUAL CHANNEL ANGULAR PRESSING

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June 2022

Chairman : Associate Professor Zulkiflle bin Leman, PhD
Faculty : Engineering

Direct solid-state process such as hot extrusion and equal channel angular pressing (ECAP) are alternative and efficient solid-state processes for recycling aluminium alloy AA7075 scrap. These processes utilize less energy and are eco-friendly. Ceramic particles such as zirconium oxide (ZrO_2) have favourable mechanical and electrical behaviours, good wear resistance and a wide bandgap. Therefore, ZrO_2 is suggested as reinforcement in the production of aluminium alloy AA7075 matrix composites (AMCs). Aluminium alloy AA7075 recycling have limitations on achieving good mechanical and physical properties and the products of the direct recycling process are still struggling with parameters optimization. Moreover, the combination of hot extrusion and ECAP metal forming has gained acceptability, but there are extreme challenges through the quality issues and enhanced composite alloy with a cost-effective. This study investigated and optimized through the response surface methodology (RSM) the effect of the volume fraction (VF), preheating temperature (T), and preheating time (t) on the mechanical and physical properties of the AA7075-ZrO_2 composite produced by hot extrusion. Additionally, the effect of heat treatment (T6) on the optimal sample was investigated. In addition, examine the elemental components the ECAP process. Moreover, developed a machine learning model based on extra trees (ET) to predict the properties and optimise the parameters. Each parameter was evaluated at varying magnitudes, i.e., 450, 500, and 550 °C for T; 1, 2, and 3 h for t, and 1, 3, and 5 % for VF. The effects of the process variables on the responses were examined using the factorial design with centre point analysis. A total of 28 experimental runs were performed through the hot extrusion process. The optimum sample was heat treated to investigate the effect on ultimate tensile strength (UTS), compressive test, microhardness, and density before and after the heat treatment condition as well as after ECAP. The recorded datasets were used for training and testing of Artificial Intelligence (AI) models were executed using machine learning methods. The AI models applied in this study was Extra Trees (ET). T and VF were crucial for attaining the maximum tensile strength 490 MPa, was attained at 550 °C, 1.58 h, and 1 vol% ZrO_2 with a microhardness

95.2 HV, compressive strength 545 MPa and density of 2.89 g/cm^3. Also, the hot extrusion and ECAP followed by heat treatment strengthened the microhardness by 64%, compressive strength by 17% and density by 3%. The results exhibited that the preheating temperature and volume fraction are the most important factor that was needed to be controlled to obtain the optimum UTS and microhardness. Preheating time has a big effect on density. The accuracy of mechanical and physical properties (ultimate tensile strength (UTS), microhardness and density) prediction of AI models along with RSM model. The obtained results revealed that the extra trees (ET) model showed outstanding performance amongst the model for training, testing, and overall datasets with coefficient of correlation (R^2), mean absolute error (MAE) and mean squared error (MSE) value of 97.6, 10.8 and 2.32, respectively. The impact of hot extrusion parameters and ECAP followed by heat treatment on the average grain sizes and microstructural analysis of the recycled samples were equally investigated and discussed in detail. Thus, it concluded that the ZrO_2, ECAP and heat treatment have a significant effect on recycled AA7075 chips. Ideationally, the ET machine learning model can minimize the experimental complexities, time, and expense in the manufacture

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

ZARAH NANO ZIRKONIUM OKSIDA DIPERKUKUH DENGAN ALOI ALUMINIUM (AA7075) KOMPOSIT MATRIKS MELALUI PENYEMPERITAN PANAS DAN SALURAN SAMA PENEKANAN SUDUT

Oleh

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Proses keadaan pepejal langsung seperti penyemperitan panas dan penekan sudut saluran sama (ECAP) ialah proses dimana keadaan pepejal alternatif dan cekap untuk mengitar semula skrap aloi aluminium AA7075. Proses ini menggunakan tenaga yang kurang dan mesra alam. Zarah seramik seperti zirkonium oksida (ZrO_2) mempunyai kelakuan mekanikal dan elektrik yang menggalakkan, rintangan haus yang baik dan jurang jalur yang luas. Oleh itu, ZrO_2 dicadangkan sebagai pengukuhan dalam pengeluaran aloi aluminium komposit matriks AA7075 (AMC). Kitar semula aloi aluminium AA7075 mempunyai had untuk mencapai sifat mekanikal dan fizikal yang baik dan produk proses kitar semula langsung masih bergelut dengan pengoptimuman parameter. Selain itu, gabungan penyemperitan panas dan pembentukan logam ECAP telah mendapat penerimaan, tetapi terdapat cabaran yang melampau melalui isu kualiti dan aloi komposit yang dipertingkatkan dengan kos yang efektif. Kajian ini menyiasat dan mengoptimumkan melalui metodologi permukaan tindak balas (RSM) kesan pecahan isipadu (VF), suhu prapanas (T), dan masa prapemanasan (t) ke atas sifat mekanikal dan fizikal komposit AA7075-ZrO_2 yang dihasilkan oleh penyemperitan panas. Selain itu, kesan rawatan haba (T6) ke atas sampel optimum telah disiasat. Di samping itu, periksa komponen unsur proses ECAP. Selain itu, membangunkan model pembelajaran mesin berdasarkan pepohon tambahan (ET) untuk meramalkan sifat dan mengoptimumkan parameter. Setiap parameter dinilai pada magnitud yang berbeza-beza, iaitu, 450, 500, dan 550 °C untuk T; 1, 2, dan 3 jam untuk t, dan 1, 3, dan 5 % untuk VF. Kesan pembolehubah proses ke atas tindak balas telah diperiksa menggunakan reka bentuk faktorial dengan menganalisis titik pusat. Sebanyak 28 eksperimen telah dilakukan melalui proses penyemperitan panas. Sampel optimum telah dirawat haba untuk menyiasat kesan ke atas kekuatan tegangan (UTS), ujian mampatan, kekerasan mikro, dan ketumpatan sebelum dan selepas keadaan rawatan haba serta selepas ECAP. Data yang direkodkan digunakan untuk latihan dan ujian model ‘’Artificial Intelligence’’ (AI) telah dilaksanakan menggunakan kaedah pembelajaran mesin. Model AI yang digunakan dalam kajian ini ialah Pokok Tambahan (ET). T dan VF adalah penting untuk

mencapai kekuatan tegangan maksimum 490 MPa, dicapai pada 550 °C, 1.58 jam, dan 1 vol% ZrO_2 dengan kekerasan mikro 95.2 HV, kekuatan mampatan 545 MPa dan ketumpatan 2.89 g/cm^3. Selain itu, penyemperitan panas dan ECAP diikuti dengan rawatan haba menguatkan kekerasan mikro sebanyak 64%, kekuatan mampatan sebanyak 17% dan ketumpatan sebanyak 3%. Keputusan menunjukkan bahawa suhu prapemanasan dan pecahan isipadu adalah faktor terpenting yang perlu dikawal untuk mendapatkan UTS dan kekerasan mikro yang optimum. Masa prapemanasan mempunyai kesan yang besar pada ketumpatan. Ketepatan ramalan sifat mekanikal dan fizikal (kekuatan tegangan muktamad (UTS), kekerasan mikro dan ketumpatan) model AI bersama-sama model RSM. Keputusan yang diperolehi mendedahkan bahawa model pokok tambahan (ET) menunjukkan prestasi cemerlang di kalangan model untuk latihan, ujian, dan set data keseluruhan dengan nilai pekali korelasi (R^2), ralat mutlak min (MAE) dan nilai ralat kuasa dua (MSE) sebanyak 97.6 , 10.8 dan 2.32, masing-masing. Kesan parameter penyemperitan panas dan ECAP diikuti oleh rawatan haba ke atas saiz butiran purata dan analisis mikrostruktur bagi sampel kitar semula telah disiasat dan dibincangkan secara terperinci. Oleh itu, ia membuat kesimpulan bahawa ZrO_2, ECAP dan rawatan haba mempunyai kesan yang ketara ke atas cip AA7075 kitar semula. Secara idealnya, model pembelajaran mesin ET boleh meminimumkan kerumitan eksperimen, masa dan perbelanjaan dalam pembuatan.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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Date: 13 October 2022

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I hereby confirm that:

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LIST OF ABBREVIATIONS

AA	Aluminium Alloys
AFM	Atomic Force Microscopy
AMC	Aluminium Matrix Composites
AMMC	Aluminium Metal Matrix Composite
AR	As Received
ASTM	American Standard Testing Material
BSE	Back Scattered Electrons
CAE	Channel Angular Extrusion
CMC	Ceramic Matrix Composites
CNC	Computer Numerical Control
CPE	Curved Profile Extrusion
DOE	Design of Experiment
DT	Decision Trees
ECAP	Equal Channel Angular Pressing
EDS	Energy Dispersive Spectroscopy
EDX	Energy Dispersive X-ray
ET	Extra Trees
FEM	Finite Element Method
FESEM	Field Emission Scanning Electron Microscopy
GWP	Global Warming Potential
HT	Heat Treatment
LM	liquid Metallurgy
MAE	Mean Absolute Error

MMC	Metal Matrix Composites
MSE	Mean Squared Error
NP	Nanoparticles
PM	Powder Metallurgy
PMC	Polymer Matrix Composites
PRMMC	Particle-Reinforced Metal Matrix Composites
Ra	The arithmetic roughness
RF	Random Forest
Rq	The root means square
RSM	Response Surface Methodology
SEM	Scanning Electron Microscopy
SPD	Sever Plastic Deformation
SPM	Scanning Probe Microscope
T	Preheating Temperature
t	Preheating time
TS	Tensile Strength
UTM	Universal Testing Machine
UTS	Ultimate Tensile Strength
VF	Volume Fraction
XRD	X-Ray Diffraction
ZrO_2	Zirconium oxide

CHAPTER 1

INTRODUCTION

1.1 Research Background

Recently, the potentials of using composite materials have gained wider acceptability due to enhanced mechanical properties. The improved properties are obtained by combining different materials, such as metallic alloys and ceramics. The ease of making composites even with oxides, borides, nitrides and carbon nanotubes is equally an advantage [1]. The composite materials are heterogeneous at microscale but homogeneous at macro-scale. Some composite materials exist in nature while others are fabricated. Take the instances of wood, teeth, bones etc. as natural forms of composite. On the fabricated composites, there are 3 types of composites material available when classified based on matrix material. These are; Polymer Matrix Composites (PMC), Metal Matrix Composites (MMC) and Ceramic Matrix Composites (CMC) [2]. Parameters affecting the properties of composite include the manufacturing techniques, this entails the processing and finishing process. The other factors are the types of reinforcements which provide various distinctive profiles even with the adoption of the same composition and amounts of the components are used [3]. Like other types of MMCs, properties of Aluminium Matrix Composites (AMCs) are associated with the processing technique and its corresponding parameters. Depending on the type of reinforcement used, different processing techniques can also be used. Aluminium Alloy AA7075 is considered a high strength alloy because its yield strength is above 500 MPa in the optimal aging conditions [4].

With excellent mechanical properties it is justifiable that alloy for structural is used applications and also for the development of Metal Matrix Composites (MMCs). The AA7075 is the most common in the 7xxx series. It has one of the highest strength, good corrosion resistance, good electrical and thermal conductivity among the aluminium alloy family [5]. The fabrication of composites from the aluminium is through the direct recycling by means of hot extrusion. In contrast to the conventional recycling process where melting of scraps is mandatory. This method has been applied in the industry, owing to the potential of saving energy, hot extrusion is an innovative process where low energy and less labour is required. This method delivered improved mechanical properties but ECAP is at present, the most developed severe plastic deformation technique producing bulk, porosity-free ultrafine grained materials [6].

1.2 Problem Statement

Pure AA7075 aluminium recycling metals and alloys have limitations on achieving good mechanical and physical properties such as strength, corrosion resistance, wear resistance, toughness's, and high-temperature performance. Nowadays, researchers from all over the world are focusing on the improvement of light metal alloys from monolithic to reinforced composite material. Recent efforts were targeted at converting as much waste to useful materials. The current work shows significant support in healthy environment for humans because it reduces the emission of carbon dioxide, which is increasing globally. However, once the manufacturing industries minimized the effect of global warming, then the direct recycling of aluminium alloys would be in the right way [7]. The general idea behind the introducing the recycling aluminium was to reduce the greenhouse gases. In addition, recycling aluminium scrap support in a great extent to the reduction of energy consumption and reduce CO_2 emissions during manufacturing as against the aluminium mining route [6]. The results of the analysis indicate that hot extrusion process obviously gives the significant environmental benefits compared to conventional re-melting technique where the Global Warming Potential (GWP) is reduced up to 69.2%. In order to produce a recycled product for engineering applications with a good mechanical property, Zirconium oxide (ZrO_2) nanoparticles are used as reinforcement in AA7075 based chips. ZrO_2 is considered as one of the promising reinforcing materials in recycling metal matrix composites [6].

A hot extrusion preceding the ECAP method was used to reduce specimens size [8]. This made the investigation on consolidation of reinforced composite particles that directly affect the metal physical and mechanical properties worthwhile [9]. The combination of hot extrusion and ECAP metal forming has gained acceptability, but there are extreme challenges to produce the composites. There are insufficient studies of optimum parameters to obtain the best mechanical properties in these composites [10]. The design of experimented (DOE) technique applied to optimise the parameters by a few authors which shows a lack of a systematic approach to process parameters. Moreover, the machine learning is more robust and accurate than DOE [11]. Products of the direct recycling process are still struggling with quality issues. Energy conservation is at the fore front of environmentalists, a study of this nature supports the low energy consumption and reduces financial burden for practical applications in industry as well as in transport industry. The conventional direct recycling is also faced with the challenge of optimum use of raw metal. The appropriate management of solid waste are equally gaining the attention of city managers as landfills are on increase. The relevance of this study in offering suggestions to all aforementioned challenges can never be over emphasized.

1.3 Aim and Objectives

The aim of this study was to convert the waste aluminium alloy AA7075 into useful products, by incorporating ZrO_2 nanoparticles as promising reinforcement in the AA7075 composite produced by solid-state recycling with enhanced mechanical and physical properties. The specific objectives were:

1. To determine tensile strength, compressive strength, microhardness and density of the AA7075-ZrO_2 nanocomposite after producing it by using a combination of severe plastic deformation process cold press and hot extrusion.

2. To optimize the parameters that affect on the physical and mechanical properties of the AA7075-ZrO_2 nanocomposites by full factorial method followed by response surface methodology (RSM).
3. To assess the maximum mechanical and physical behaviour of the developed chip based nanocomposite after the heat treatment (T6) condition as well as after ECAP.
4. To examine the components of the recycled AA7075-ZrO_2 consolidated via hot extrusion and ECAP principle using SEM, FESEM, XRD, and AFM.
5. To develop a machine learning model based on extra trees (ET) to predict the properties of AA7075-ZrO_2.

1.4 Scope of Research

The scopes of this research cover the following limitation:

1. Recycling aluminium alloy AA7075 chips were produced by a computer numerical control (CNC) machining process.
2. The experiments were carried out by a cold press machine compacted the billet with a maximum of 50-ton capacity followed by a hot extrusion.
3. The main investigated parameters were preheating time (1 h, 2 h and 3h) preheating temperature (450°C, 500°C and 550°C) and the reinforced particles of (ZrO_2) contents.
4. Conducting experimental investigations and evaluate the following responses by hot extrusion process.
5. Tensile strength and compressive strength test using a universal testing machine (The limitation of ECAPed samples size restricts the experiment to the determination of compressive strength only).
6. Microhardness using the Vickers hardness tests and density test.
7. Optical Characterization and fracture surface using scanning electron microscope (SEM) and field emission scanning electron microscopy (FE-SEM), atomic force microscope analysis (AFM) and X-Ray diffraction (XRD).
8. Modelling and optimization of the extrusion quality characteristics using (3 factors x 3 levels) face centre, CCD Response Surface of RSM optimization method.
9. Predicting and optimization of the extrusion quality characteristics using machine learning model based on extra trees (ET).

1.5 Significance of Study

The research intends to propose a new approach to improve the performance of aluminium composites made of chips with the addition of ZrO_2 nanoparticles. The addition of reinforced particulates is expected to improve the chip-based composite's mechanical and physical properties compared to composite with 100% pure AA7075 chips. This research is also aimed at further investigations on aluminium chips reinforced by ZrO_2 processed through the solid-state recycling technique performed without the melting phase. Therefore, hot extrusion is introduced as the solid-state recycling method in this research. The significance of this research is to perform a comprehensive evaluation of the hot extrusion potential indirect aluminium chips recycling by incorporating the RSM method for process optimization.

In addition, this research investigates the quality of preparing scraps in machining the depth of cut, cleaning, drying, mixing composites, and hot extrusion influences such as preheating temperature and preheating time. However, installing the heating supply to the die avoids materials fatigue and broken tools. Furthermore, the study aims to access the maximum behaviour of the developed chip-based nanocomposite before and after the heat treatment condition as well as after ECAP to be used in the existing automotive components. The study also includes a novel simulation by developed a machine learning model based on extra trees (ET) to predict the mechanical properties of AA7075-ZrO_2 and optimize this model performance.

1.6 Thesis Organization

The thesis has been structured into five chapters:

Chapter 1 covers the basic research background and problems that necessitate the aim and objectives. In addition, this chapter also covers the objectives, scope and significance of the study.

Chapter 2 deals with a review of the major topics related to this thesis in a logical manner. This includes the previous work on modifications in properties and formation of aluminium metal matrix composite (AMMCs) characterizations and recycling techniques. Further work on the literature review includes previous studies on principles of severe plastic deformation (SPD), fundamentals of hot extrusion practice equal channel angular pressing (ECAP), post heat treatment and the effects of zirconium oxide (ZrO_2) reinforced aluminium matrix composite.

Chapter 3 thesis covers the materials, process and methodology used in the thesis.

Chapter 4 explained the results and discusses the findings.

Chapter 5 presents the conclusions of this study and the overall summary of the findings and recommendations for future works.

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