**UNIVERSITI PUTRA MALAYSIA**

# *EFFECTS OF CuO AND SiO2 AS REINFORCEMENT IN ALUMINIUM (AA6061) CHIPS HYBRID NANOCOMPOSITES USING HOT EXTRUSION PROCESS*

**MSEBAWI MUNTADHER SABAH ABDUL HUSSEIN**

**FK 2022 82**

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By

**MSEBAWI MUNTADHER SABAH ABDUL HUSSEIN**

**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 brothers***
***My family***
**(Huda, Ghadeer and Mahdi)**

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

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**June 2022**

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

Aluminium alloy AA6061 is widely used in various applications which generates a lot of waste in the form of aluminium chips that can be recycled back to the industry. The success of recycling AA6061 alloy chips is highly dependent on extrusion process parameters and the reinforcing materials to enhance its aluminium properties. However, incorporating copper oxide (CuO) and silica oxide ($SiO_2$) nanoparticles into the aluminium matrix is a difficult task particularly due to their agglomeration as well as aggregations in the metal matrix nanoparticle reinforced composites (MMNCs). Furthermore, little quantities of these nanoparticles as reinforcements provides an effective improvement of the thermal, physical and mechanical properties of these nanocomposites. In addition, the literature lacks a comprehensive analysis of the relationship between the strength performance and hybrid composite materials. In this study, the nano silica oxide ($SiO_2$) and nano copper oxide (CuO) were used for reinforcement purposes. Also, the research aimed at optimizing the influences of preheating temperature (PHT), preheating time (PHti), volume fraction of nano silica oxide ($SiO_2$) and nano copper oxide (CuO) on the physical and mechanical properties of the aluminium AA6061 hybrid composite samples through hot extrusion treatment. Furthermore, the comparison and investigation analysis of physical, mechanical, and morphology between the single reinforced Al6061-$SiO_2$ and Al6061-CuO nanocomposites were done. The three parameters mentioned above were varied in the range of 450 - 550 ºC, 1 - 3 h, and 1 - 3 vol%. The optimum values of PHT, PHti, and VF to obtain the maximum tensile strength value was established to be at 541ºC, 2.25 h, as well as 1 vol.% of $SiO_2$ and 2.13 vol.% CuO volume fraction respectively. On top of that, the response surface design (RSM) showed that the interaction between the reinforcements and PHT values contributed significantly to the strength and microhardness. While for the density values of the samples of both reinforcements, the PHti value was significant. On the other hand, the peak tensile strength value of 295.97 MPa was observed in the heat-treated extrudes which was improved to about 27% compared to the optimum tensile strength value of 232.66 MPa in nonheat-treated

sample. At the same time, the Random Forest result value of 2.73% error for both validation and prediction showed that indicating the highly accurate results with no significant over-estimation or under-estimation of the targeted values. Finally, the Differential Scanning Calorimetry (DSC) profiles were employed to explain the weight loss, heat flow and crystallization temperature. Scanning Electron Microscope (SEM) and Field Emission Scanning Electron Microscope (FESEM) showed that the fractured surface in tensile samples differed in contours due to the proper distribution of stress in the composite samples. Lastly, the analysis of Atomic Force Microscopy (AFM) and X-Ray Diffraction (XRD) revealed that the distribution of CuO and $SiO_2$ reinforced particles in specimens were slightly uniform. The results showed that the use of $SiO_2$ and CuO as reinforcements in AA6061 recycling could avoid the possibility of particles aggregation in the recycled composites.

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

**KESAN CuO DAN SiO2 SEBAGAI PENGUKUHAN DALAM ALUMINIUM (AA6061) CIP KOMPOSIT-NANO HIBRID MENGGUNAKAN PROSES PENYEMPERITAN PANAS**

Oleh

**MSEBAWI MUNTADHER SABAH ABDUL HUSSEIN**

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**Pengerusi : Profesor Madya Zulkiflle bin Leman, PhD**
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Aloi aluminium AA6061 digunakan secara meluas dalam pelbagai aplikasi yang menjana banyak sisa dalam bentuk cip aluminium yang boleh dikitar semula ke industri. Kejayaan mengitar semula cip aloi AA6061 sangat bergantung pada parameter proses penyemperitan dan bahan penguat untuk meningkatkan sifat aluminiumnya. Walau bagaimanapun, menggabungkan nanozarah kuprum oksida (CuO) dan silika oksida ($SiO_2$) ke dalam matriks aluminium adalah tugas yang sukar terutamanya disebabkan oleh pengagregatannya serta pengagregatan dalam komposit bertetulang nanozarah logam (MMNCs). Tambahan pula, kuantiti kecil nanozarah ini sebagai tetulang memberikan penambahbaikan yang berkesan terhadap sifat terma, fizikal dan mekanikal komposit nano ini Selain itu, kesusasteraan tidak mempunyai analisis yang komprehensif tentang hubungan antara prestasi kekuatan dan bahan komposit hibrid. Dalam kajian ini, nano silika oksida ($SiO_2$) dan nano kuprum oksida (CuO) digunakan untuk tujuan tetulang. Selain itu, penyelidikan bertujuan untuk mengoptimumkan pengaruh suhu prapemanasan (PHT), masa prapemanasan (PHti), pecahan isipadu nano silika oksida ($SiO_2$) dan nano kuprum oksida (CuO) ke atas sifat fizikal dan mekanikal komposit hibrid aluminium AA6061. sampel melalui rawatan penyemperitan panas. Tambahan pula, perbandingan dan analisis penyiasatan fizikal, mekanikal dan morfologi antara nanokomposit Al6061-$SiO_2$ dan Al6061-CuO bertetulang tunggal telah dilakukan. Tiga parameter yang dinyatakan di atas telah diubah dalam julat 450 - 550 ºC, 1 - 3 jam, dan 1 - 3 vol%. Nilai optimum PHT, PHti, dan VF untuk mendapatkan nilai kekuatan tegangan maksimum telah ditetapkan pada 541 ºC, 2.25 jam, serta 1 vol.% daripada $SiO_2$ dan 2.13 vol.% pecahan isipadu CuO masing-masing. Selain itu, reka bentuk permukaan tindak balas (RSM) menunjukkan bahawa interaksi antara tetulang dan nilai PHT menyumbang dengan ketara kepada kekuatan dan kekerasan mikro. Manakala bagi nilai ketumpatan sampel kedua-dua tetulang, nilai PHti adalah signifikan. Sebaliknya, nilai kekuatan tegangan puncak 295.97 MPa diperhatikan dalam penyemperitan dirawat haba yang bertambah baik kepada kira-kira 27% berbanding nilai kekuatan tegangan optimum 232.66 MPa dalam sampel tidak dirawat haba. Pada masa yang sama, nilai hasil Hutan

Rawak sebanyak 2.73% ralat untuk kedua-dua pengesahan dan ramalan menunjukkan bahawa menunjukkan keputusan yang sangat tepat tanpa anggaran berlebihan atau anggaran terkurang yang ketara bagi nilai yang disasarkan. Akhirnya, profil Pengimbasan Kalorimetri Berbeza (DSC) digunakan untuk menerangkan penurunan berat badan, aliran haba dan suhu penghabluran. Mikroskop Elektron Pengimbasan (SEM) dan Mikroskop Elektron Pengimbasan Pelepasan Medan (FESEM) menunjukkan bahawa permukaan patah dalam sampel tegangan berbeza dalam kontur disebabkan oleh pengagihan tegasan yang betul dalam sampel komposit. Akhir sekali, analisis Mikroskopi Daya Atom (AFM) dan Difraksi Sinar-X (XRD) mendedahkan bahawa taburan zarah bertetulang CuO dan $SiO_2$ dalam spesimen adalah seragam sedikit. Keputusan menunjukkan bahawa penggunaan $SiO_2$ dan CuO sebagai tetulang dalam kitar semula AA6061 dapat mengelakkan kemungkinan pengagregatan zarah dalam komposit kitar semula.

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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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| | |
|---|---|
| AA | Aluminium Association |
| AMCs | Aluminium Matrix Composites |
| MMCs | Metal Matrix Composites |
| PMCs | Polymer Matrix Composites |
| CMCs | Ceramic Matrix Composites |
| MMNCs | Metal Matrix Nanocomposites |
| HMMCs | Hybrid Metal Matrix Composites |
| GFRPs | Glass Fibre Reinforced Polymers |
| °C | Degree Centigrade |
| ANOVA | Analysis of Variance |
| DOE | Design of Experimental |
| PHT | Preheating Temperature |
| PHti | Preheating Time |
| vol.% | Volume Fraction Percentage |
| wt.% | Weight Percentage |
| RF | Random Forest |
| PSO | Particle Swarm Optimization |
| DT | Decision Tree |
| SDGs | Sustainable Development Goals |
| MSE | Mean Square Error |
| MAE | Mean Absolute Error |
| $R^2$ | Correlation Coefficient |
| S | swarm |
| SEM | Scanning Electron Microscope |

| | |
|---|---|
| UPM | University Putra Malaysia |
| UTHM | Universiti Tun Hussein onn Malaysia |
| TS | Tensile Strength |
| XRD | X-ray Diffraction |
| DSC | Differential Scanning Calorimetry |
| TGA | Thermal Gravimetric Analysis |
| SEM | Scanning Electron Microscope |
| FESEM-EDS | Field Emission Scanning Electron Microscopy |
| AFM | Atomic Force Microscopy |

# CHAPTER 1

# INTRODUCTION

## 1.1 Introduction to Hybrid Composite

Composite materials have useful properties due to their constituent matrix and reinforced materials [1, 2]. One of the latest generations of composites to date is the heterogeneous hybrid composite that comprises at least three distinct components or phases with various shapes and compositions.

The conventional approach for aluminium production from ores requires approximately 113 GJ per tonne of aluminium, while the secondary fabrication and production from conventional aluminium recycling methods from scrap requires around 13.6 GJ per tonne of energy [3, 4]. In contrast, the production of recycled aluminium can save up to 88% of the energy used in extracting aluminium from ores [4]. However, due to the difference in properties, the recycled aluminium alloy chips are not frequently used in different applications, such as in the automotive industry. The properties of recycled aluminium can be significantly improved through the utilisation of reinforced materials. The successful application of reinforcements to achieve the desired properties, including the tensile strength, with an increase in yield was recently reported [5].

The solid-state recycling is used to convert the metallic scraps into bulk material, consequently eliminating the remelting process that is commonly found in the conventional recycling approach [6, 7, 8]. Moreover, solid-state recycling via the hot extrusion process produces less waste and lower environmental implications [9].

The quality and properties of hot extruded aluminium alloy chips are determined by several extrusion factors, such as process temperature, extrusion ratio, die geometry, chip morphology, and extrusion speed. These factors play a vital role in determining the final properties and microstructure in the extruded products [10]. The formation of hybrid composites with the addition of two different reinforcing ceramic particles in the aluminium matrix improved the mechanical properties [11]. The use of hybrid ceramic reinforcements was also employed to produce cheaper final products [12].

Previously, aluminium silica oxide ($Al\text{-}SiO_2$) nanocomposites prepared by powder metallurgy and subsequent hot extrusion resulted in an enhanced tensile strength and compressive mechanical properties [13, 14, 15]. In other studies, the addition of copper oxide (CuO) reinforcement reduced the energy consumption by changing the preheating temperature to 550 °C and preheating time to 3 h for optimum mechanical properties [15,16] and reduced melting point [17.18]. The tribological properties are considered to be one of the major factors controlling the performance and mechanical properties of composites [18,19,20]. A good balance between the mechanical properties, thermal properties, and production cost is the key performance indicators for the successful development of the hybrid composites. However, incorporating CuO and silica oxide $SiO_2$ nanoparticles into the aluminium matrix was difficult due to the agglomeration phenomena in the nanoparticle-reinforced metal matrix composite MMCs [21]. MMC production show a significant potential of this material to be applied in the automobile engineering industries Figure 1.1. [22].

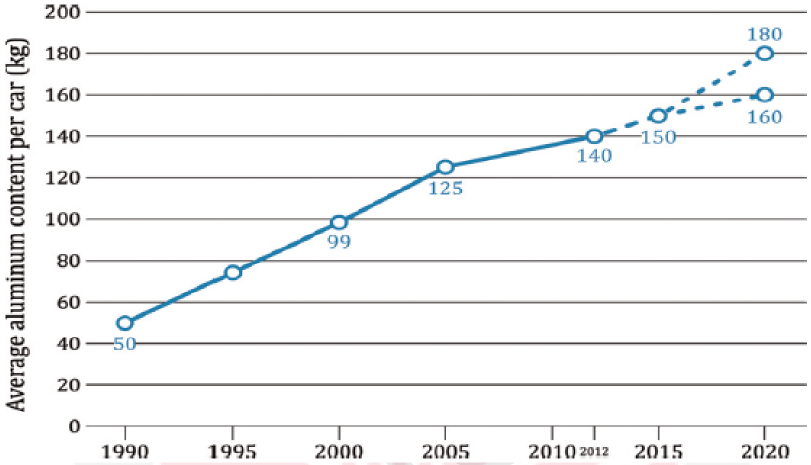


**Figure 1.1 : The evolution in average aluminium content per car produced in Europe. [22]**

The mechanical properties of the composite materials could be optimised through the development of an efficient model based on machine learning. Currently, algorithms are widely used in tree-based machine learning for different applications, such as agricultural processes [23], transportation sector [24], materials science research [25], and energy processes [26]. Tree-based algorithms are well-known and studied for the production and prediction of practical and convenient performing end results [25,26]. These algorithms are used effectively by maintaining the interactions automatically, even in the presence of various large coefficients [28]. The Random Forest (RF) model is an effective tree-based ensemble technique to carry out regression and classification studies.

## 1.2 Problem Statement

Pure AA6061 recycling metals and alloys have limitations in obtaining good mechanical and physical properties such as strength, wear resistance, corrosion resistance, toughness and high temperature performance. Nowadays, the exceptional properties of CuO and $SiO_2$ and commercially available and low cost nanoparticles [29], they have incorporated into the aluminium matrix as a strengthening to fabricate copper nano oxide blended aluminium matrix in the nanocomposites (Al-CuO) and silica nano oxide reinforced aluminium matrix composites (Al-$SiO_2$). Incorporating a little quantity of these nanoparticles as reinforcements provides an effective improvement of the thermal, physical, and mechanical properties of these nanocomposites. Incorporating CuO and $SiO_2$ nanoparticles into the aluminium matrix is difficult task particularly due to agglomeration as well as aggregations in the nanoparticle-reinforced MMNCs [21]. While, increasing the silica oxide contain led to a decrease in the tensile properties [15] Moreover, the presence of too much brittle $Al_2Cu$ phase and large Si phase caused by Si clustering in the aluminium composite debase the mechanical properties remarkably. Blending Cu in the composites reduces the melting point and resulting in the formation of $Al_2Cu$ phase, thus reducing the consolidation temperature and enhancing the strength of Al matrix [18]. Furthermore, the ceramic reinforcement in the composites reduce the heat emitted by changing the thermal behaviour to the endothermic phase [30]. As well as silica supported weldability and improved mechanical properties and form compounds that make the alloys heat treatable [14].

Aluminium is abundant on earths; the primary aluminium source is depleting due to massive mining of ores. A lot of wastes are generated from conventional manufacturing and machining processes, and all these are recyclable. Environmentalists are also greatly concerned about $CO_2$ emissions and carbon footprint especially in Asia as it becomes among the top in the global emission list. All these justify the use of other resources to be explored as alternatives to overcome the stated problems. The alternative technique can be proposed is solid-state recycling via the hot extrusion process was recommended as a novel recycling technique for machining chips. It is comparatively requiring low cost and the technique is also environmentally friendly. This process involves a direct use of metal scraps to produce a bulk material for engineering applications. An excessive plastic work that is induced during the hot extrusion process can efficiently consolidate the chips to produce composite of various shapes.

Today, the development of new composites gained high recognition, more rapidly than ever before. The reasons are due to:

(i) The transport industry is always in a quest for reduction of weight for which aluminium is a better candidate to achieve this aim

(ii) Environmental pollution is at the fore of the Sustainable Development Goals (SDGs) for which almost all the countries of the world are signatories to its success.

These outstanding benefits were the spur of the metal nanocomposite developed has become the most widely investigative research area for industrial applications. The aluminium MMC was developed as an alternative to steel and cast-iron owing to their high density and weight. Economical there is need for further research into the cost effectiveness of the composites has been long inquired and therefore it is worthy to explore. On top of that, the mechanical properties of the reinforced materials as well as the processing technique need thorough review to promote the composite for mass production. Shaping and machining of these materials leftover a challenge due to combination of various dissimilar reinforcements and its abrasive nature advancement in the fabrication of these nanocomposites with the combination of novel materials, there will be always new challenges for engineering of such materials with close tolerance [31]. When preparing the MMC through melting process like stir casting method, there is significant loss of those costly particles. This is due to poor wettability of the particles to melt due to the density difference between the particles within the composites. These waste of particles makes fabrication more expensive [32] Strengthening of metal matrix by nanoparticles underpinning, the particulates attracted many researchers for improving mechanical properties in different application. The presence of nanoparticles in the composites makes a requirement of an analysis of hot workability study of nanocomposites. So far, limited works are available about the impact of nanoparticles reinforcements in the current work, on hot compression properties of nonferrous alloys and nanocomposites synthesised by means of mechanical alloying routes [33]. Decrease in size of nanoparticles reinforcement, and then it's very likely to achieve a finer grain microstructure due to the suppression of grain growth during solidification stage which can result in the enhancement of mechanical properties. The trapping of air and formation of bubbles increase with reduction of the size of particles, which dramatically decrease the mechanical properties of composite. Therefore, substituting micro ceramic particles with nanoparticle reinforcements is also accompanied with some challenges [34]. The ceramic particles dissolve into aluminium matrix, which helps to produce a high bonding strength among the particles and matrix materials [35]. Other than that, the previous studies show less were made on the relationship of directly recycled aluminium alloy AA6061 product by hot extrusion with the hybrid ceramic reinforcement. Therefore, it is important to elucidate this relationship specifically and in the well-planned manner. An agreement between mechanical, thermal properties and production cost may likely to be the key performance indicator effective fabrication of hybrid-reinforced nanocomposites.

The present work focuses on the development of nano silica oxide and nano copper oxide particles reinforced chip-based composite. This composite was processed through the hot extrusion route. The chips in this case are directly recycled without involving the high energy of re-melting operation. The effect of volume fraction of reinforcement particle added to the composite to achieve the desired mechanical properties (tensile strength and Microhardness tests) and thermal properties were also be investigated. Finally, a comparison in terms of mechanical performance with the nano silica oxide and nano copper oxide particle reinforced composite was carried out to investigate the efficiency of the nano silica oxide and nano copper oxide addition into the newly developed chip-based hybrid composite before and after heat treatment.

## 1.3 Aim and Objectives

The aim of this study was to convert the waste aluminium alloy AA6061 to useful products, the proposed $SiO_2$ and CuO nanoparticles promising reinforcing AA6061 composite produced by solid-state recycling by performing various mechanical and physical tests. The specific objectives were:

(i) To determine the feasibility of synthesizing single and hybrid AA6061 nanocomposites using a combination of cold press and hot extrusion

(ii) To optimise the process parameters affecting the physical and mechanical of the synthesised AA6061-$SiO_2$-CuO nanocomposites using response surface methodology (Minitab).

(iii) To compare the analysis of physical, mechanical and morphology between the single reinforced AA6061-$SiO_2$ and AA6061-CuO nanocomposites.

(iv) To establish heat treatment at T6 condition and assess the effect on the morphological, microstructural, physical and mechanical properties of hybridise AA6061-SiO–CuO nanocomposites.

(v) To develop a machine learning model based on random forest (RF) model to predict the mechanical properties of AA6061-$SiO_2$-CuO and optimise this model based on a metaheuristic method to improve the model performance.

## 1.4 Contribution

In brief, this thesis reported significant contributions, which enhance the existing knowledge, and they are remarked as follows:

(i) Fabricate new product aluminium hybrid nanocomposite from waste AA6061 alloy by hot extrusion with high strength and endothermic behaviour. Although is difficult task particularly agglomeration phenomena and various dissimilar reinforcements [21, 31].

(ii) New combination of ceramic reinforcements have used, both of them have different mechanical and physical properties of the particles in the melt and due to the density difference between the particles and the matrix remarkable findings comparing with other reported articles such as [15, 21, 35].

(iii) For Aluminium fabrication that uses machine learning was significant to obtain high efficiency in research studies and manufacturing processes, the new fabricated product can be obtained minimizing the time and energy consumption compared with other reported articles such as [9, 36].

As a result, these contributions led to novel approach to improve the performance of AA6061 aluminium made of chips with the addition of $SiO_2$-CuO without remelting and development of a simulation to predict and optimise the mechanical and physical properties of nanocomposite through machine learning.

### 1.5 Scope of Study

The scope of this research covers the following limitation

(i) Recycling AA-6061 aluminium chips were produced by a high-speed machining process.

(ii) The experiments were carried out by a cold press fallowed by a hot extrusion.

(iii) The main investigated parameters were pre-heating temperature (450 °C, 500 °C and 550 °C), preheating time (1, 2 and 3 h) contents.

(iv) The volume fraction of silica oxide ($SiO_2$) and copper oxide (CuO) particles volume fraction were (1 %, 2 % and 3 %) from each ceramic

(v) The heat treatment at T6 condition considers the effect of different temperatures during solution heat treatment and distinct aging time applied. The optimisation on the heat treatment parameters was carried out using the DOE of factorial design.

(vi) Tensile strength test using a universal testing machine

(vii) The Vickers hardness tests using to microhardness at the deepest layer of the surface.

(viii) The density test using Archimedes principles.

(ix) Surface layer changes by using a scanning electron microscope (SEM), Ray Diffraction XRD, Atomic force microscope (AFM) and Field Emission Scanning Electron Microscopy (FESEM EDS).

### 1.6 Thesis Outlines

This thesis shows how to investigate and analyse the CuO and $SiO_2$ on the AA6061 to employ them for suitable applications. The thesis was organized as follows:

Chapter 1 presents the overview of the solid-state recycling process to produce recycled aluminium based on oxide reinforcement and their performance, mechanical and thermal properties feature for different applications. Additionally, the $SiO_2$, CuO and extrusion factors effect on the properties of the hybrid composite materials coupled with development of an efficient model based on machine learning. Moreover, the problem statement and the main objectives of this thesis are also included.

Chapter 2 introduces the literature review of aluminium recycling techniques information. Nano reinforcement materials, such as copper oxide and silica oxide are reviewed where they play an important role in composite materials. Also, solid-state recycling such as hot extrusion in addition to it is parameters and optimization are discussed in detail where these elements are employed to achieve our goals and objectives of this study.

Chapter 3 highlight the methodology of the AA6061 chips preparation and characterizations. The important steps of hybrid and single reinforcement composite preparation procedures are described here. Starting with mixing sample calculations, followed by fabrication procedure, and aluminium fabrication are clearly explained systematically. The material characterizations by means of mechanical, physical and thermal analysis in addition to the instrumentation are also specified.

Chapter 4 Firstly, to determine the feasibility of synthesizing hybrid AA6061-$SiO_2$-CuO nanocomposites using a combination of cold press and hot extrusion. As a result, optimise the process parameters affecting the physical and mechanical of the synthesised AA6061-$SiO_2$-CuO nanocomposites. and compare analysis of physical, mechanical and morphology investigations between the single reinforced AA6061-SiO and AA6061-CuO nanocomposites. Lastly, develop a machine learning model based on random forest (RF) model to predict the mechanical properties of (the material working on) and optimise this model based on a metaheuristic method to improve the model performance. Finally, to establish heat treatment at T6 condition and assess the effect on the morphological, microstructural, physical and mechanical properties of hybridise AA6061-SiO–CuO nanocomposites.

Chapter 5. concludes the findings of this study. Upon the analysis of the results, each material properties are identified for suitable parameter showed a positive increment in performance indeed process like solid state (hot extrusion) that decreased in energy to produced endothermic material according to the reinforcements that were added.

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