UNIVERSITI PUTRA MALAYSIA

INFLUENCE OF HEAT TREATMENT ON MICROSTRUCTURE, MECHANICAL PROPERTIES, AND CORROSION BEHAVIOUR OF LM6 REINFORCED WITH COPPER POWDER

ALMUKHTAR IB. A. ALJERMI

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By

ALMUKHTAR IB. A. ALJERMI

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

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May 2021

Chairman : Professor Shamsuddin Sulaiman, PhD
Faculty : Engineering

Copper (Cu) is one of the most widely used materials in the world. It has great significance in most industries, especially in the synthesis of alloys. A number of advanced Al-alloys have developed in which Cu is the main alloying element because it makes those alloys more heat treatable. Even though Al-Si eutectic casting alloy (LM6) has outstanding properties such as wear and corrosion resistance (CR), it has medium strength (130MPa) and is considered a non-heat-treatable alloy. Due to the importance of heat treatment for enhancing the mechanical properties of Al-alloys contained Cu, this study aimed to investigate the role of Cu in increase the effectiveness of heat treatment in changing the morphology structure of LM6 which in turn enhances the mechanical properties. In this research, the copper powder (Cu_p) was added to the LM6 to improve the hardness and tensile properties. By using the stir casting technique, Cu_p in the percentages of 3, 6, 9 wt.% has been added into the LM6. Some produced specimens were subjected to solution treatment (T6) to compare with the non-heat treated ones. The T6 was conducted at 490 ^{0}C for 6h followed by quenching in warm water (60 ^{0}C), then aging for 5h at 155 ^{0}C. The microstructures of the samples have been studied through optical microstructure, fracture surfaces of the tensile test samples were analysed via scanning electron microscopy, and X-Ray diffraction was used to identify chemical composition. Hardness and tensile strength tests were conducted for all samples. In order to study corrosion manner, samples were subjected to acidic and alkaline environments using the weight-loss method. The experimental result showed that the mechanical properties of LM6 were changed by adding a certain percentage of Cu and heat treatment through modifying the microstructure. It was observed that the maximum Rockwell hardness value (53.84 HRB) and highest tensile strength (152MPa) were achieved by applying heat treatment on the LM6 composite reinforced with 6wt.%Cu. On the other hand, the corrosion test showed that the LM6 composite reinforced with different Cu content had low CR than the LM6 as-cast. The corrosion test showed that LM6 as-cast and LM6 composite had higher CR in the acidic and alkaline solutions. Analysis of variance (ANOVA) has been used to determine the statistical significance of parameters that influencing the responses. The influence of Cu contents and heat treatment on the mechanical properties of LM6 composite have been evaluated by two-way ANOVA using statistical software Minitab 17. The statistical results revealed that interaction between the two factors, 6wt.%Cu and heat treatment, gave the optimal hardness and tensile properties.

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

PENGARUH RAWATAN HABA KEATAS STRUKTUR MICRO, SIFAT MEKANIKAL, DAN KELAKUAN KARAT LM6 DIPERKUAT DENGAN SERBUK TEMBAGA

Oleh

ALMUKHTAR IB. A. ALJERMI

Mei 2021

Pengerusi : Profesor Shamsuddin Sulaiman, PhD
Fakulti : Kejuruteraan

Tembaga (Cu) adalah bahan yang digunakan secara meluas di dunia. Ia banyak memberikan kelebihan ketara dalam kebanyakan industri terutama aloi-aloi sintesis. Beberapa aloi aluminium termaju telah dibangunkan dengan kandungan tembaga sebagai elemen utama sebab ia menjadikan aloi tersebut lebih berkebolehan dirawat. Walaupun aloi tuangan eutektik Al-Si (LM6) mempunyai sifat cemerlang seperti haus dan ketahanan karat (CR), ia adalah dikira kekuatan sederhana (130MPa) dan dianggap aloi yang tidak boleh dirawat haba. Berdasarkan kepada kepentingan rawatan haba untuk menambahbaikan sifat mekanikal Al-aloi yang mengandungi Cu, maka kajian ini bertujuan melihat peranan Cu dalam menambahkan keberkesanan rawatan haba, menukarkan sturuktur mofologi LM6 untuk memperbaiki sifat kekerasan dan ketegangan. Dalam penyelidikan ini serbuk tembaga (Cu) telah dimasukkan ke dalam LM6 untuk menambahkan sifat kekerasan dan ketegangan aloi. Dengan bantuan teknik kacau, Cu dalam jurang 3, 6, 9 % berat telah ditambah dalam LM6. Spesimen yang dihasilkan bergantung kepada jenis rawatan (T6) untuk dibandingkan dengan specimen tanpa rawatan. T6 telah dijalankan pada 490 ^{0}C selama 6 jam diikuti dengan memasukkan ke dalam air suam (60 ^{0}C) kemudian 5 jam penuaan pada 155 ^{0}C. Sampel-sampel struktur mikro telah dikaji menggunakan *optical microstructure* (OM), dan sampel ujian tegangan keretakan permukaan telah dibuat menggunakan *scanning electron microscopy* dan *X-Ray defraction* digunakan untuk mengenalpasti kandungan kimia. Ujian kekerasan dan ketegangan telah dijalankan untuk semua sampel. Untuk mengkaji kelakuan karat, ujian secara kehilangan-berat sampel yang tertakluk kepada larutan asid dan alkali telah dijalankan. Keputusan menunjukkan sifat mekanikal LM6 berubah dengan penambahan peratus Cu sementara rawatan haba mengubah sturuktur mikro bahan. Pemerhatian mendapati nilai kekerasan Rockwell yang dicapai bagi sampel komposit LM6 yang dirawat beserta dengan tembaga adalah 53.84 HRB dan ujian ketegangan jugamenunjukan pertambahan dengan nilai tetinggi 152MPa untuk sampel yang dirawat dan mengandungi 6wt.%Cu. Walaubagaimanapun, ujian kekaratan, menunjukkan komposit LM6 dengan kandungan tembaga yang berbeza menghasilkan ketahan karat yang rendah berbanding LM6 asal. Ujian karatan menunjukkan LM6 asal dan LM6 komposit mempunyai ketahanan karat yang tinggi dalam cairan asid berbanding cairan alkali. Analisa ANOVA telah digunakan untuk menentukan statistik parameter yang mempengaruhi keberkesanan keputusan. Pengaruh kandungan tembaga Cu dan rawatan haba keatas sifat mekanikal komposit LM6 telah dinilai oleh ANOVA dua-hala menggunakan perisian statistik Minitab 17. Keputusan statistik mendapati interaksi antara dua factor, 6wt.%Cu dan rawatan haba memberikan sifat kekerasan dan ketegangan yang optimum.

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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:

Shamsuddin bin Sulaiman, PhD
Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Mohd Idris Shah bin Ismail, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Azmah Hanim binti Mohamed Ariff, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Member)

ZALILAH MOHD SHARIFF, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

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Signature:	
Name of Chairman of Supervisory Committee:	Professor Dr. Shamsuddin bin Sulaiman

Signature:	
Name of Member of Supervisory Committee:	Associate Professor Dr. Mohd Idris Shah bin Ismail

Signature:	
Name of Member of Supervisory Committee:	Associate Professor Dr. Azmah Hanim binti Mohamed Ariff

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LIST OF ABBREVIATIONS

AA	Aluminium Association
Al	Aluminium
Al-Si	Aluminium Silicon
AMCs	Aluminium Matrix Composites
AMMCs	Aluminium Metal matrix composites
ASTM	American Society for Testing and Material
C	Carbon
CO_2	Carbon dioxide
Cu	Copper
COF	Coefficient of Friction
LM6	Light Metal (Aluminium 1 1.8% Silicon alloy)
MPa	Mega Pascal
MYP	mils penetration per year
HF	Hydrofluoric acid
Mg	Magnesium
Mn	Manganese
MMCs	Metal matrix composites
mm	millimeter
ml	milliliter
mg	milligram
min	Minute
NaOH	Sodium hydroxide
H_2SO_4	Sulphuric acid

HNO_3	Nitric acid
NaCl	Sodium chloride
h	Hour
µm	Micrometer (micron)
ρ	Density
%	Percentage
^0C	Degree centigrade
T	Temperature
PAMCs	Particulate Aluminium Matrix Composites
pH	Power of hydrogen ion
OM	Optical Microscopy
RXD	X-Ray Diffraction
SEM	Scanning electron microscope
Sr	Strontium
Wt.%	Weight percentage
WVL	Wear Volume Loss
Zn	Zinc

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Aluminium alloys are widely used in several engineering applications, such as transportation and aerospace due to their outstanding properties. Aluminium silicon cast alloys are extensively used in the casting industries because they have a low melting point, low thermal expansion coefficient, and high strength to weight ratio. Those properties led to the use of these alloys in the automobile industry, especially for cylinder blocks, cylinder heads, pistons, and valve lifters (Ejiofor and Reddy, 1997). Some applications of Al-Si components in automobile components are shown in Figure 1.1.

Addition of major alloying elements such as silicon, copper,magnesium, and zinc is useful to improve pure Al properties through composing its alloys. In Al-Si alloys, silicon is considered as the essential alloying element which controls in improves most ofpure Al properties. It counts primarily responsible for cast-ability where it possesses high fluidity and low shrinkage because it reduces the thermal expansion coefficient of Al. Moreover, silicon has lowsolubility in Al solid solution that leads to improving abrasion resistance (Rana et al., 2012).

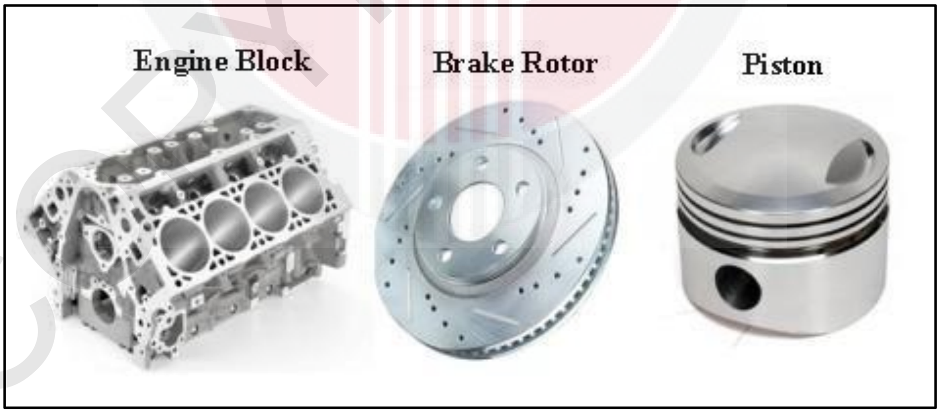

Figure 1.1 : Some applications of Al-Si alloys for automobile component

The Al-Si alloys are formed by adding silicon element to pure aluminium, which are considered the most used Al-alloys because of its lightweight, good specific strength, high corrosion resistance, good cast-ability, and low thermal expansion coefficient. However, in some engineering applications, the increased strength by adding the silicon is insufficient. Furthermore, adding Si to Al reduces ductility as well as machinability. In such cases, incorporating other elements is necessary to improve such properties. Copper and magnesium are preferred using as alloying elements due to their capability to make alloys responded to precipitation and aging hardening. In order to enhance tensile strength, hardness, and machinability, copper is introduced into Al-alloys. Copper can, however, cause stress susceptibility to corrosion in particular compositions, metallurgical conditions, and service environments (Zor et al., 2010).

The mechanical properties of eutectic Al-Si cast alloys mainly depend on microstructure features, e.g. grain size, eutectic and silicon morphology, and other intermetallics that exist in the microstructure. During heat treatment, the precipitation hardening phases often exert an important effect on the mechanical properties (Mohamed et al., 2009). The presence of some additional elements in the Al-Si alloys leads to forming several complex intermetallics.One of the active precipitation-strengthening agents in aluminium is copper. In natural or artificial ageing, copper additions of up to about 5 per cent result in alloys with high strength and strong durability. Due to precipitation of the dispersed Al_2Cu phase during ageing, the addition of Cu increased the strengthof Al-Si alloys (Abdulsahib, 2014).

One of the advanced materials is known to be metal matrix composites (MMCs) that are used to improve the strength of Al and its alloys by adding ceramic particles such as carbides and oxides into Al. Eventhough these ceramics increase the strength, the ductility is reduced due to the low thermal expansion coefficient that leads to missing bonding with the matrix (Yadav and Bauri, 2015). Metallic particles are also used as reinforcements to Al matrix to improve the strength and ductility. However, metallic particles might form some harmful intermetallic if their solid solubility is low (Thakur and Gupta, 2007). Copper has a relatively high solid solubility in Al which has reduced with decreasing temperature (Mahmood et al., 2016).

The sand casting method is one of the adopted processes used to produce MMCs. It is considered relatively low-cost, simple, and versatile compared to other methods. It also makes it possible to produce large and intricate parts and is also used in mass production (Reddy and Ztioun, 2011). Meanwhile, the key disadvantages of the sand cast method are the complexity of regulating the distribution of reinforcement and achieving a uniform microstructure of the matrix, therefore using stir casting technique enhances the constituent's homogenization. In addition, adverse reactions are more likely to occur at high temperatures at the interface between the matrix and the reinforcement. These resulted in the creation of a harmful intermetallic compound, which adversely affect the mechanical properties (Balaji et al., 2015). Figure 1.2 shows typical sand casting steps, while Figure 1.3 shows stir technique casting process.

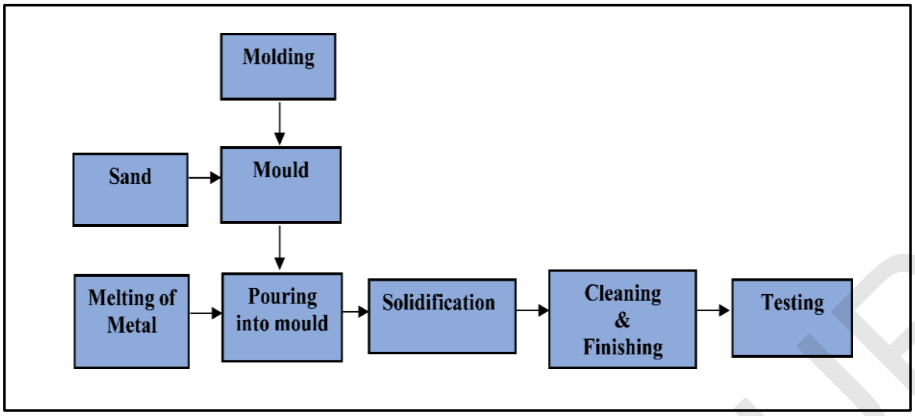

Figure 1.2 : Sand casting steps

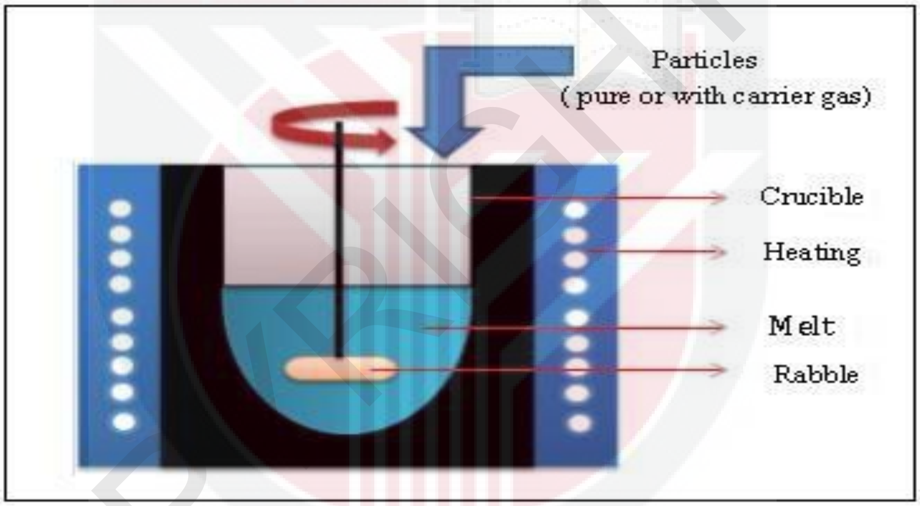

Figure 1.3 : Stir casting technique
(Park and Seo, 2011)

Aluminium alloys are categorised into alloys which are heat-treatable and non-heat-treatable. Heat-treatable alloys are those which copper, magnesium, and zinc are the main alloying components. Only by cold working, the properties of the remaining aluminium alloys could be enhanced since there is no precipitation hardening process in them. The primary requirement for an alloy system to respond to the heat treatment is a substantial decrease in the solid solubility of one or more alloying elements with decreasing the temperature. One of the few elements with relatively high solubility in Al is copper (Aravind et al., 2004).

Aluminium and Al-Si alloys have good corrosion resistance in a neutral aqueous solution due to the formation of a thin isolated film. One way to enhance mechanical properties is to add alloying elements such as copper, however, this might result in a negative side effect on the electrochemical properties. The corrosion behaviour is affected by many factors connected to the circumference and the metal, where the design of the corrosive medium has an effective role. (Kaiser, 2019).

1.2 Study Motivation

The first primary motivation of this work is the potential to fabricate LM6 composite reinforced with copper powder using sand casting mould which satisfies the requirement of intricate-shaped parts such as impellers, and manifolds of pumps. The second major motive is to investigate the effect of copper addition with different percentages on the mechanical properties of LM6 alloy. The third main motivation of this work is to search for the impact of heat treatment on the range of alteration of the morphology structure. The fourth motivation is to study the effect of both copper addition and heat treatment on the corrosion behaviour of produced composite to bear working conditions such as acidic and alkaline solutions which may be facing in the water treatment stations.

1.3 Problem statement

Adding silicon to pure aluminium improves physical and mechanical properties such as reducing density, increasing hardness, improving cast-ability, and enhancing specific strength. In some applications, the strength produced by adding silicon is still relatively low due to a lack of homogeneity (Zor et al., 2010). Some previous studies have indicated that Al-Si alloy (LM6) has a medium strength such as; (Bhandwale et al., 2016), (Anoopsai & Pradheep, 2017), and (Sadiq et al., 2019). On the other hand, some works that conducted tensile tests on the LM6 samples have gotten low tensile results for example; (Prakash et al., 2020) detected LM6 tensile strength was 111.6 MPa, (Sayuti et al., 2016) discovered LM6 tensile strength was 118 MPa, (Akhil, 2018) found LM6 tensile strength was 119 MPa, (Bera et al., 2018) recorded LM6 tensile strength was 112 MPa, and (Sulaiman & Zulkifli, 2018) found LM6 tensile strength was 89.11MPa.

The mechanical properties of Al-Si alloys rely on the characteristics of the microstructure. The microstructure can be adjusted by adding alloying elements and/or by applying heat treatment, which in turn leads to enhance the mechanical properties. Alloying elements added to Al-Si Alloys such as; Cu, Mg, or Ni, might dissolve in the solid solution or form intermetallic phases. The presence of copper in the alloy as an alloying element leads to the formation of an intermetallic compound (AL_2Cu) which considers the common precipitation hardening phase.

The maximum solubility of Cu in Al at 548 ^{0}C is 5.65wt.% and decreases at room temperatureto 0.1wt.%. (Mahmood et al., 2016).

Some studies used Cu as a reinforcement element to improve the mechanical properties of Al-Si eutectic alloy but got reverse results. Sayuti et al. (2020) studied the mechanical properties of LM6 reinforced with different Cu contents using sand mold followed by mechanical vibration. The result revealed that LM6 as-cast under mechanical vibration had better mechanical properties than reinforced composite. The tensile strength was reduced with increasing copper percentages, especially with applying mechanical vibration. They concluded the vibration has good effectiveness during applying on the LM6 as-cast, but it gave reverse results during applying on the LM6 reinforced with copper.

Previous studies extremely did not focus on the use of Cu as a reinforcing element for Al-Si composite, even though Cu is used to enhancing the bonding and the wettability between Al matrix and reinforcing materials such as steel, and carbon. The role of copper in this process has been investigated in some previous studies such as; (Wang et al., 2017), and (Chelladurai et al., 2017). Even the few works that focused on adding copper as a reinforcing element to Al alloys such as (Sayuti et al., 2020), did not take into consideration the solubility of Cu in aluminum which needs heat treatment to have a more impact on the microstructure, which in turn lead to enhance the mechanical properties.

Therefore, the purpose of this study is to investigate the extent to which the microstructure and mechanical properties of LM6 are affected by adding different percentages of copper followed by applying heat treatment. In this experimental work, LM6 alloy matrix composite was reinforced with various copper powder contents using carbon dioxide sand casting mould supported with stir technique. The produced specimens were subjected to solution heat treatment followed by an artificial ageing process. The microstructure characteristics of reinforced and heat-treated LM6 were analysed, the hardness and tensile strength were tested, and the corrosion behaviour was studied, then the results were compared with the unreinforced alloy.

1.4 Research objectives

The main objective of this experimental work is to investigate the influence of copper content and heat treatment process on the mechanical properties, microstructure, and corrosion behavior of Al-Si eutectic alloy (LM6) using carbon dioxide sand casting mold with a supporting stir technique. The specific objectives of this study include:

1. To develop a composite material that has represented in reinforcing the LM6 matrix with different copper powder contents using sand casting mould.

2. To investigate the effectiveness of heat treatment on strengthening the composites containing copper by precipitation hardening.
3. To test the mechanical properties and to analyse the microstructure of the samples with the different copper contents that were either heat-treated or not.
4. To examine the corrosion behaviour of the produced composite by subjected the samples to acidic and alkaline solutions.

1.5 Scope and limitation

In this experimental study, aluminum-silicon eutectic alloy (LM6) used as a matrix material and different contents of copper powder (3wt.%, 6wt.%, and 9wt.%) were used as reinforcement materials to improve the hardness and strength of LM6. Sulphuric acid and sodium hydroxide were used in the corrosion test as different mediums test. The method adopted to prepare tensile test specimens was CO_2 sand casting supported with a stir casting technique. The weight-loss method was used to study the corrosion behavior of the composite. The T6 solution treatment conducted to modify the morphology structure. Hardness and tensile tests were conducted to investigate their changes due to adding the different copper contents and applying heat treatment. The microstructure, surface fracture, and compositions were analyzed using an optical microscope, a scanning electron microscope, and X- Ray diffraction, respectively. All tests have conducted at room temperature.

1.6 Overview and layout of the thesis

This thesis consists of five chapters that illustrate the fabrication of aluminium silicon composite reinforced with copper powder using sand casting mould.

Chapter 1 is the introduction. To accomplish the aim and boundary of this work, the problem statements, objectives and scope of the study were specified. The chapter ends with summary that clarifies the chapter description in general.

Chapter 2 presents a comprehensive literature review about aluminium metal matrix composites. It lists some related works about aluminium silicon alloys, alloying elements, and copper importance. Sand casting mould, heat treatment, hardness, tensile properties, microstructure analysis, and corrosion behaviour were explained in this chapter.

Chapter 3 deals with the research methodology, in which the sand casting mould and heat treatment were mentioned in this chapter. It lists and describes some tests; hardness, tensile, and corrosion test and analyses of the microstructure and surface fracture using OM, SEM, and XRD.

Chapter 4 describes the findings of this research, including the analysis and description of tables and figures and the discussion of the study's outcomes that often apply to the significance of the effects of the experimental work carried out.

Chapter 5 discusses the conclusion of the research and possible recommendations for improvements of this work

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BIODATA OF STUDENT

The student name is Almukhtar Ib. A. Aljermi who was born on 23rd of March 1965 in Zawia city, Libya. He obtained his Bachelor in Industrial Engineering from Faculty of Engineering, Benghazi University, Libya 1987.After finishing his study, he has worked for twenty years (1989- 2009) in Research Technology Center / Tripoli /Libya. He has pursued his Master of Science, specialized in Manufacturing Engineering, Belarusian National Technology University (2002-2005). He continued work in Research Technology Center until (2009). Then he joined ministry of higher education in Libya at (2009) as a lecturer in Zawia Engineering Faculty. In 2013, he got a scholarship from the Ministry of Higher Education, Libya. In2015, he enrolled in a PhD program under supervision of **Professor Dr. Shamsuddin bin Sulaiman, Associate Professor Dr. Mohd Idris Shah bin Ismail,** and **Associate Professor Dr. Azmah Hanim binti Mohamed Ariff,** Department of Mechanical and Manufacturing Engineering, Faculty of Engineering, Universiti Putra Malaysia,

LIST OF PUBLICATIONS

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