UNIVERSITI PUTRA MALAYSIA

CHARACTERIZATION AND MECHANICAL PROPERTIES OF INSITU TITANIUM DIBORIDE REINFORCED ALUMINIUM-COPPER ALLOY COMPOSITES

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CHARACTERIZATION AND MECHANICAL PROPERTIES OF *IN-SITU* TITANIUM DIBORIDE REINFORCED ALUMINIUM-COPPER ALLOY COMPOSITES

By

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

March 2016

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

CHARACTERIZATION AND MECHANICAL PROPERTIES OF *IN-SITU* TITANIUM DIBORIDE FIBRE REINFORCED ALUMINIUM-COPPER ALLOY COMPOSITES

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March 2016

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Aluminium (Al) based *in-situ* metal matrix composites (MMCs) have better properties and performance when compared to *ex-situ* MMCs. Al-MMCs alloys are quite attractive due to their low density, their capability to be strengthened by precipitation, their good corrosion resistance, high thermal and electrical conductivity. Aluminium-copper (Al-Cu) alloys is the one of most MMCs have important high-strength Al alloys. The Al casting alloys, based on the Al-Cu system are widely used in light-weight constructions and transport applications requiring a combination of high strength and ductility. Recently, *in-situ* techniques have been developed to fabricate Al-based MMCs, which can lead to better adhesion at the interface and hence better mechanical properties. *These in-situ* routes provide many advantages such as the *in-situ* formed reinforcement phases are thermodynamically stable, disperse more uniformly in matrix, free of surface contamination and leading to stronger particle matrix bonding.

In this research, Al-Cu master alloy was reinforced with 1 to 6wt.% titanium diboride (TiB_2) obtained from salts route reactions which were potassium hexafluorotitanate (K_2TiF_6) and potassium tetrafluoroborate (KBF_4) salts. The salts route reaction process done at 800 °C. Then the Al-Cu alloy has characterized on the mechanical properties and microstructure characterization. The Instron tensile machine, Vickers and Rockwell hardness tester, and pin on-disc machine were used to characterize the tensile, hardness and wear properties of Al-Cu alloys respectively. Salts spray fog test and Gamry-electrode potentiometer were used to determine the corrosion rate of this alloys. From results obtained, the increasement of TiB_2 contents will increased the value of tensile and hardness properties to Al-Cu alloy. The study also indicates that TiB_2 particles have giving improvement the wear performance of the Al–6wt.%Cu alloy. For a constant load and sliding speed, the wear rate decreases as a function of amount of TiB_2 in the composite. However, addition of TiB_2 particle to the Al–6 wt%.Cu matrix has show the coefficient value of wear decreases regardless of applied load. Microstructure from scanning electron microscope (SEM) shows the composites synthesized using *in-situ* techniques exhibit the presence a uniform distribution of reinforcement that tends to be fine, and associated with a clean interface with the metallic matrix. Morphology observed that the particles of the TiB_2 phase show a hexagonal morphology with straight and sharp edges. In order to achieve a good mechanical and wear properties it is important to control Al_3Ti phase formation during

the synthesis of *in-situ* Al-Cu/TiB_2 composites. In corrosion test that conducted by salt spray fog and Gamry-electrode potentiometer, Al-Cu with composition of 3wt.%TiB_2 gave the good properties in corrosion characterization compare to cast Al-Cu alloy itself. As comparison, Al-Cu with 3wt.%TiB_2 gave the lowest value of corrosion rate, which means alloy has a good properties in corrosion characterization.

The results obtained show that *in-situ* Al-Cu alloy composites containing different weight of TiB_2 phase were synthesized successfully by the salt-metal reaction method and the particles were distributed evenly in the matrix of the composites.

Abstrak tesis yang dikemukan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

PENCIRIAN DAN SIFAT MEKANIK KOMPOSIT ALOI ALUMINIUM-KUPRUM DISEDIAKAN SECARA *IN-SITU* DIPERKUATKAN OLEH TITANIUM DIBORID

Oleh

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Aluminium (Al) berasaskan *in-situ* komposit matrik logam (MMCs) mempunyai sifat dan prestasi yang agak baik jika dibandingkan dengan MMC disediakan secara *ek-situ*. Aloi Al-MMC menarik perhatian kerana memiliki ketumpatan yang rendah, berkebolehan untuk penguatan dalam pemendakan, kerintangan pengaratan yang baik dan kekonduksian terma dan elektrik yang tinggi. Aloi aluminium-kuprum (Al-Cu) adalah salah satu komposit matrik logam yang memiliki kekuatan yang tinggi dalam aloi Al. Aloi Al yang disedia secara tuangan berasaskan kepada sistem Al-Cu telah diguna meluas dalam penggunaan pembinaan berat yang ringan dan penggunaan untuk pengangkutan disebabkan memiliki kombinasi sifat kekuatan yang tinggi dan mulur. Kebelakangan ini, teknik *in-situ* telah dibangunkan untuk memfrabikasikan Al komposit berasaskan matrik logam yang mana membawa kepada lekatan pada antara muka yang baik dan juga meningkatkan sifat mekanik yang baik. Teknik ini memberikan banyak kelebihan seperti pembentukan *in-situ* yang terhasil daripada fasa penguat stabil dari segi termodinamik, berserakan secara seragam dalam matrik, bebas daripada kekotoran dan memberikan ikatan zarah terhadap matrik.

Dalam penyelidikan ini, aloi Al-Cu diperkuatkan dengan dengan 1 hingga 6 %bt. titanium diborid (TiB_2) yang diperoleh daripada tindakbalas laluan garam. Tindakbalas laluan garam dilakukan pada suhu 800 °C di antara garam kalium heksaflorotitanat (K_2TiF_6) dan kalium tetrafloroborat (KBF_4) yang kemudiannya dicirikan oleh sifat mekanik dan mikrostruktur. Mesin Universal Instron, alat penguji kekerasan Vickers dan Rockwell dan mesin ujian haus masing-masing digunakan bagi mencirikan sifat regangan, kekerasan dan sifat haus aloi Al-Cu. Teknik semburan garam dan Gamry-elektrod potentiometer pula digunakan bagi menentukan kadar pengaratan bagi aloi ini. Daripada keputusan yang diperoleh, didapati peningkatan kandungan TiB_2 akan meningkatkan nilai sifat regangan dan kekerasan aloi Al-Cu. Kajian juga menunjukkan bahawa partikel TiB_2 memberikan peningkatan dalam prestasi haus aloi Al–6%bt. Cu. Dengan menetapkan beban, dan kelajuan sliding, kadar haus menurun berbanding dengan kandungan TiB_2 dalam komposit. Walau bagaimanapun, dengan penambahan kandungan TiB_2 terhadap Al-6%bt. Cu menunjukkan nilai pekali berkurangan berbanding dengan beban yang digunakan. Mikrostruktur daripada mikroskop imbasan elektron (SEM) pula menunjukkan komposit yang disintesiskan melalui teknik *in-situ* menghasilkan pembahagian bahan penguat yang seragam dan cenderung untuk

menjadi halus dan berlakunya pergabungan antara muka dengan bahan matrik logam. Morfologi yang dilihat pada fasa TiB_2 menunjukkan bentuk heksagon berkeadaan lurus dengan bucu yang tajam. Untuk mencapai sifat mekanik dan haus yang terbaik ia penting untuk mengawal pembentukan fasa Al_3Ti semasa penghasilan komposit Al-Cu/TiB_2 secara *in-situ*. Ujian pengaratan menggunakan semburan garam dan Gamry-elektrod potentiometer menunjukkan Al-Cu dengan komposisi 3%bt. TiB_2 menghasilkan sifat yang terbaik bagi pencirian pengaratan berbanding dengan aloi Al-Cu itu sendiri. Sebagai perbandingannya, aloi Al-Cu dengan 3%bt. TiB_2 menghasilkan nilai kadar pengaratan yang rendah, bermaksud sesuatu logam itu mempunyai sifat pengaratan yang baik.

Keputusan-keputusan yang diperoleh menunjukkan bahawa aloi komposit Al-Cu disediakan secara *in-situ* dengan kandungan peratusan berat TiB_2 yang berbeza berjaya disintesiskan menggunakan teknik tindak balas laluan garam di mana partikel diserakkan secara seragam dalam matrik komposit.

ACKNOWLEDGEMENTS

In the name of Allah, most gracious, most merciful, all praise and thanks are due to Allah, and peace and blessings be upon His Messenger, Muhammad SAW. I would like to express the most sincere appreciation to those who made this work possible; advisor, member of advisory committee, technicians, my family and friends.

I would like to thank Prof. Dr. Shamsuddin bin Sulaiman for providing me the opportunity to complete my PhD studies under his valuable guidance, for the many useful advice and discussions, for his constant encouragement and guidance, and for co-authoring and reviewing some of my publications, where his practical experience and technical knowledge made this research and those publications more interesting and relevant. In addition, special thanks extend to the supervisory committee members from Universiti Putra Malaysia, Dr. Mohd Idris Shah bin Ismail and Dr. Azmah Hanim binti Mohamed Ariff and also from Universiti Teknologi Mara, Shah Alam, Dr. Mahesh Kumar Talari. I am grateful for their willingness to serve on my supervisory committee, constant encouragement, helpful advice and many fruitful discussions.

Special thanks dedicate to Dr. Mohamad bin Harun, a Research Officer and their Laboratory Assistant, En. Sharif bin Sattar, a technician from Agensi Nuklear Malaysia, Kajang to guide how to use wear test machine of ‘pin-on-disk’, and Pn. Sabrina Mohd. Yahya (PhD student from Faculty of Applied Sciences UiTM Shah Alam), to guide a corrosion test used Gamry-electrode potentiometer by Linear Polarization Resistance (LPR) technique in Universiti Teknologi Mara, Jalan Othman, campus Petaling Jaya, Selangor.

To the sponsors, I am grateful to Ministry of Education and Universiti Teknologi Mara for offering me the scholarship and FRGS grant for pursuing the PhD degree and for funding this research at Universiti Putra Malaysia.

To all my family, thanks and acknowledgements to my mother, Maznah binti Mat Jusoh, my wife, Rozihan binti Mahmood and childrens, Rafie’uddin (12), Nur Wirdani (10), Suci Ramadhani (6), Nur Saffiyah Irdina (5) and Adam Addeney (2) who deserve my deepest appreciation. I am grateful for the countless sacrifices they made to ensure that I could pursue my ambition and always being there for me.

I certify that a Thesis Examination Committee has met on 9 March 2016 to conduct the final examination of Rosmamuhamadani bin Ramli on his thesis entitled "**CHARACTERIZATION AND MECHANICAL PROPERTIES OF *IN-SITU* TITANIUM DIBORIDE FIBRE REINFORCED ALUMINIUM-COPPER ALLOY COMPOSITES**" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the (insert the name of relevant degree).

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AgCl	Argentum chloride
Al	Aluminium
Al-Cu	Aluminium-copper
Al-Cu-Fe	Aluminium-copper-ferum
Al-Cu-Mg	Aluminium-copper-magnesium
Al/TiB_2	Aluminium-titanium diborde
Al–Ti–B	Aluminium-titanium-boron
Al/SiC	Aluminium-silicon carbide
Al-Zn-Mg-Cu	Aluminium-zinc-magnesium-copper
Al/Mg$(ZnCu)_2$	Aluminium/magnesium(zinc(II) copper)
Al_2O_3	Aluminium oxide
Al_3Ti	Aluminium titanium
AMCs	Aluminium Matrix Composites
ASTM	American Society for Testing and Materials
B	Boron
BC	Boron carbide
Bap	Bagasse ash
CeO_2	Cerium oxide
ClO_4^-	Perchlorate ion
C_S	Solid formed
Cu	Copper
$CuAl_2$	Copper (II) aluminium
Ds	Self diffusion coefficient
E_{corr}	Corrosion potential
EDS	Energy Dispersive Spectroscopy
EDX	Energy-Dispersive X-Ray
ELTA	Electrolytic Low-Titanium Aluminum
F	Faraday constant
GPa	Gega Pascal
HCl	Hydrochloric acid
HIP	Hot isostatic pressing
HNO_3	Nitric acid
H_3BO_3	Boric acid
Hv	Vickers hardness
I_{corr}	Corrosion current
K	Kelvin
KBF_4	Potassium tetrafluoroborate
K_2TiF_6	Potassium hexafluorotitanate
$KFAlF_3$	Cryolite
KF	Kalium flouride
kgf	Kilogram force
L	Liquid phase
LPR	Linear Polarization Resistance
M	Atomic weight of the metal
mA/cm^2	Mili Ampere/centimeter square
mm/y	Millimeter per year
mV	mili Volt
MPa	Mega Pascal

Mg	Magnesium
MMCs	Metal Matrix Composites
M/n	Equivalent weight
nm	Nano meter
nm/s	Nano meter per second
N	Newton
Na_2SO_4	Natrium sulphate
NaCl	Sodium chloride
NaBr	Natrium bromide
$NaBH_4$	Sodium borohydride:
Na_3AlF_6	Sodium hexafluoroaluminate
NaI	Natrium iodide
PM	Powder metallurgy
R_M	Corrosion rate
rpm	Revolusion per minute
SEM	Scanning Electron Microscope
SiC	Silicon carbide
Ti	Titanium
TiB_2	Titanium diboride
TiO_2	Titanium carbide
TiO_2-Al-B	Titanium oxide-aluminium-boron
TiO_2-Al-B-CuO	Titanium oxide-aluminium-boron-copper oxide
TiO_2-Al-B_2O_3,	Titanium oxide-aluminium-boron trioxide
$TiCl_4$	Titanium chloride
TiC	Titanium carbide
TiC/Al	Titanium carbide/aluminium
TEM	Transmission electron microscope
UTS	Ultimate tensile stress
wt.%	Percentage of weight
XRD	X-Ray Diffractometer
YS	Yield stress
ZrO_2	Zirconium dioxide
°C	Degree celcius
α	Alpha
$\beta\rho$	Density
µm	Micron meter
η	Charge number
σ_y	Yield stress
σ_o	Friction stress

CHAPTER 1

INTRODUCTION

1.1 Background of Study

In order to meet the demands of the aerospace, automotive and military industries such as guns, ammunition, missiles, military aircraft, military vehicles, ships and electronic systems, the necessity of lightweight and high performance structural materials has provided the necessary momentum for the development and emergence of metal-matrix composites (MMCs). Further, these MMCs are attractive and viable alternatives to the traditional engineering alloys, with majority of them having metallic matrices reinforced with high strength high modulus and brittle ceramic phases (Krishna et. al., 2011). Particulate reinforced MMCs appear to be the most popular choice because they can offer relative ease in processing, lower fabrication cost, and nearly isotropic properties in comparison to fiber reinforced materials (Christy et. al., 2010).

Aluminium-copper (Al−Cu) alloys are one of the most MMCs have important high-strength Al alloys. They have been employed extensively in the aircraft and military industries, in which materials are frequently subjected to elevated temperature. The aluminum (Al) casting alloys, based on the Al−Cu system are widely used in light-weight constructions and transport applications requiring a combination of high strength and ductility (Wang et. al., 2010).

Recently, *in-situ* techniques have been developed to fabricate Al-based MMCs, which can lead to better adhesion at the interface and hence better mechanical properties (Krishna et. al., 2011). Al-MMCs fabricated by *in-situ* routes provide many advantages such as reinforcement phases are thermodynamically stable, disperse more uniformly in matrix, free of surface contamination and leading to stronger particle matrix bonding (Wang et. al., 2009). At the same time, *in-situ* Al-MMCs formed reinforcement with phase finer size. According to Sun and Ahlatci (2011), *in-situ* processes can create a variety of reinforcement morphologies, ranging from discontinuous to continuous, and the reinforcement may be either ductile or ceramic phases.

1.2 Problem Statements

There are two methods to synthesized aluminium matrix composites (AMCs) which are *ex-situ* and *in-situ* synthesis. The difficulties in the development of *ex-situ* particulate MMCs such as poor wettability, inhomogeneous distribution of reinforcement particles, formation of unwanted reaction products at the interface between the matrix and reinforcement, have led to the attempts to synthesize new generation *in-situ* composites (Wang et. al., 2009).

Most of the Al-based *ex-situ* composites have low reinforcement or matrix bonding strength. To overcome this problem, *in-situ* synthesis techniques were developed in the last few years. It was observed during *in-situ* synthesis, unwanted brittle phases can occur resulting in poor mechanical properties (Kumar et. al., 2007).

In-situ processes involve the synthesis of composites such that desirable reinforcements, matrices and interfaces are formed during processing. The successful synthesis of *in-situ* composites involves a good understanding of thermodynamics and reaction kinetics in order to obtain the desirable end product. Besides, the composites synthesized by *in-situ* techniques exhibit the presence of a uniform distribution of reinforcement that tends to be fine and associated with a clean interface with the metallic matrix, which assists in the formation of a stronger bond between the reinforcement and the metallic matrix (Lakshmi et. al., 1998).

However Al-MMCs that already use in automotive industries are not strong enough to withstand high pressure, low specific stiffness and low strength, and low wear resistance (Wang et. al., 2010). Hence the desire in the engineering community to develop a new material with greater wear resistance, finer grain structure and better mechanical properties, without much compromising on the strength to weight ratio led to the development of MMCs. So, as a solution, fabricate of *in-situ* of Al-6wt.%Cu as matrix and react with TiB_2 as reinforcement was introduced. This is believed, *in-situ* can lead to better adhesion at the interface and hence better mechanical properties. Lu et. al., (2001) stated that TiB_2 is chosen because it is particularly suitable as reinforcement for Al-based due to high exothermic and thermodynamic stability in Al.

1.3 Objectives of Study

The objectives of the study are;

i. To synthesize *in-situ* TiB_2 reinforced Al-Cu alloys with different TiB_2 contents.
ii. To determine mechanical properties of Al-Cu alloys reinforced TiB_2 and the influence of reinforcement, TiB_2 on the aging behaviour of the Al–Cu matrix.
iii. To analyse microstructures and phase distribution characterization of Al-Cu alloys reinforced TiB_2 by Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD).
iv. To determine the corrosion resistance of Al-Cu alloys reinforced TiB_2 and the effect of reinforcements, TiB_2 on Al-Cu alloys.

1.4 Scope of Study

In this research, Al-6 wt.%Cu alloys and TiB_2 as reinforcement will be used. Al-Cu alloys will be reinforced with *in-situ* TiB_2 particles and are synthesized by salt route reaction. TiB_2 was introduced into the Al-Cu MMCs alloy by the reaction of the molten alloy with halide salts which are potassium hexafluorotitanate (K_2TiF_6) and potassium tetrafluoroborate (KBF_4) at 800 °C for 60 minutes. The contents of reinforcement used were 1 to 6 wt. %TiB_2 which are fabricated by this technique. The focus of study will give more attention especially in dynamic and static mechanical properties such as tensile, hardness, wear performance and corrosion properties. Besides that, microstructure and phase distribution also were characterized by SEM and XRD.

1.5 Thesis Layout

The layouts of this thesis are the Introduction, Literature Review, Methodology, Results and Discussions and Conclusions and Recommendations for Future Research. The layouts of the thesis are as discussed and listed below.

In Chapter 1, background study of Al-MMCs alloy and reinforcement TiB_2 used in this research were briefed. Also the general information of *in-situ* technique that will use in this investigation is discussed. Besides that, the statement of the problems and the objectives of research are also being highlighted in this chapter.

In Chapter 2, the discussions from previous study which were related with materials and processing used were highlighted. Besides *in-situ*, other techniques that used in fabrication of alloys also highlighted. It is important to discuss and study the previous result from other researchers especially in mechanical properties such as tensile, hardness and wear properties performance of Al-Cu composites.

In Chapter 3, selected of materials, equipments and instruments used for this investigation are highlight and discussed. Besides, the procedures and the selection of methods also were discuss according to test were applied. Most of mechanical tests follow according to ASTM methods. The mechanical characterization involved tensile properties (ASTM E-345, 2002), wear performance (ASTM G-99, 2010) and hardness properties (ASTM E-92, 2003). The characterization of XRD and SEM also were done to study the phase distribution and microstructure observation of Al-Cu reinforced with TiB_2. For corrosion study, two types of tests are choosing which are salts spray fog tests according to ASTM B-117, (2011) and Gamry-electrode potentiometer by Linear Polarization Resistance (LPR) technique.

In Chapter 4, discussion on research results of Al-Cu alloy reinforced with TiB_2 were discusses. The results mainly can be divided to mechanical properties, such as tensile properties (Tensile strength, yield strength, young's modulus and elongations), hardness and wear performance test. Besides, corrosion tests also were done by salts spray test and Gamry-electrode potentiometer. Microstructures and phase distribution characterization were observed by SEM and XRD. The results obtained then be compared to previous results have been studied by previous researcher.

And lastly, in Chapter 5, a conclusions and recommendations for future research were listed. In this chapter, all the results obtained by data and figures were summarized and concluded. For the general conclusion are *in-situ* Al-Cu alloy composites containing different TiB_2 contents were synthesized successfully by the salt-metal reaction method and the particles were distributed evenly in the matrix of the composites. Besides, the recommendation what the next plan and activities for future study were also highlighted.

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