MICROSTRUCTURE, MECHANICAL PROPERTIES AND CORROSION BEHAVIOR OF AA5083 ALUMINUM ALLOY MODIFIED WITH RARE EARTH ELEMENTS

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DEDICATION

To my loved mother and the spirit of my loved father, may 'ALLAH' give him a pleasant place in Paradise

"My Success Is Yours Too"

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ABSTRACT

The commercial AA5083 alloy is a non-heat-treatable alloy with high strength-to-weight ratio. It has good welding properties, ease of machinability and formability, and satisfactory corrosion property resistance in marine and industrial environments. Despite the advantages it possesses, the AA5083 alloy has some limitations such as its moderate strength, and susceptibility to localised corrosion (LC) in the marine environment that increases under high-speed conditions. It is also susceptible to intergranular corrosion (IGC) and intergranular stress corrosion cracking (IGSCC) at the temperature range of 50°C to 200°C. Previous studies showed that the addition of an appropriate amount of rare earth elements (REEs) can improve the microstructure, mechanical properties, and corrosion resistance of Al-alloys. This research was aimed at investigating the effects of REEs (Ce, Pr, and Ce + Pr) additions on the microstructure, mechanical properties, and corrosion behavior of the AA5083 alloy. Ce and Pr were added to AA5083 alloy using in-situ casting process in the amount of 0.1 wt% to 1.0 wt% whilst the combination of Ce-Pr was between 0.1wt% and 0.5wt%. Non-destructive testing (NDT) was conducted to assess the quality of the samples produced whilst the homogenisation process was performed at 450°C for 24 hr to achieve homogeneous microstructure. Characterisation of the modified AA5083 alloys was conducted using optical microscope (OM), variable pressure scanning electron microscope (VPSEM) equipped with energy dispersive spectrometer (EDS) and x-ray diffraction (XRD). Corrosion behavior was investigated by visual assessment (ASSET-Test), IGC susceptibility by nitric acid mass loss test (NAMLT), electrochemical behaviour testing in 3.5% NaCl solution by Tafel extrapolation (TE) and potentiodynamic polarization (PDP), and full immersion corrosion tests in 3.5% NaCl solution under static and high-speed conditions at room temperature. Tensile properties and impact tests were also conducted to determine mechanical properties of the alloy. In addition, erosion prediction test was performed by impingement of solid spherical particle at normal incidence. The findings indicated that addition of Ce, Pr, and Ce + Pr to the AA5083 alloy resulted in the reduction of grain size. The average grain size were reduced from 105μm to 72 μm and 107 μm to 74 μm respectively, as addition of Ce and Pr from 0.1 to 1.0wt%. Addition of Ce-Pr combination in the amount of 0.1% to 0.5% resulted in average grain size reduction from 98 μm to 65 μm. The presence of stable precipitate (REE-rich) phases played a critical role in the grain refinement, thereby improving the mechanical properties. The stable phases reduced the β-phase (Al_3Mg_2) and Fe-rich and Mg-rich phases, which are a major cause of LC, IGC, and IGSCC, thus improving the corrosion resistance. These properties were more prominent in the case of addition of Ce + Pr combined than in individual addition of Ce or Pr. The modified AA5083 alloy with 0.5% Ce + 0.5% Pr addition had the best overall improvement in terms of mechanical properties and corrosion behaviour compared to other modified AA5083 alloys. The average grain size was reduced by 39.8%. Toughness, yield strength, and ultimate strength improved by 21%, 27.5% and, 53.3 % respectively whilst corrosion rate decreased by 31.2%. Thus, it is suggested that, REEs can be promising candidates for the alloying elements of AA5083.

ABSTRAK

Aloi AA5083 komersial merupakan aloi tidak-boleh-rawat haba dengan sifat nisbah kekuatan-berat yang tinggi. Ia mempunyai sifat kimpalan yang baik, mudah dimesin dan dibentuk, dan ketahanan kakisan yang memuaskan dalam persekitaran marin dan industri. Walaupun mempunyai kelebihan, aloi tersebut mempunyai beberapa kelemahan seperti kekuatan yang sederhana, kerentanan terhadap kakisan setempat (LC) dalam persekitaran marin yang meningkat dalam keadaan kelajuan tinggi. Ia juga rentan terhadap kakisan antara butir (IGC) dan kakisan tegasan retak antara butir (IGSCC) pada julat suhu dari 50°C hingga 200°C. Kajian sebelum ini menunjukkan penambahan unsur nadir bumi (REEs) dengan jumlah yang sesuai boleh menambahbaik struktur mikro, sifat mekanik dan ketahanan kakisan aloi Al. Penyelidikan ini dilaksanakan bertujuan untuk mengkaji kesan mencampurkan REE (Ce, Pr, dan Ce + Pr) terhadap struktur mikro, sifat mekanik dan kelakuan kakisan aloi AA5083. Ce dan Pr dicampur pada aloi AA5083 dengan menggunakan proses penuangan in-situ dalam julat dari 0.1 hingga 1.0% berat manakala campuran kombinasi Ce-Pr adalah antara 0.1% berat hingga 1% berat. Ujian Tanpa Musnah (NDT) dilakukan untuk menilai kualiti sampel yang dihasilkan manakala proses penghomogenan dilakukan pada suhu 450°C selama 24 jam untuk mendapatkan struktur mikro yang homogen. Pencirian aloi AA5083 yang diubahsuai dilaksanakan dengan menggunakan mikroskop optik (OM), mikroskop imbasan elektron tekanan berubah-ubah (VPSEM) yang dilengkapi dengan spektrometer serakan tenaga (EDS) dan pembelauan sinar-x, (XRD). Kelakuan kakisan dikaji melalui penilaian visual pengelupasan dan ujian kerentanan kakisan lekuk (ASSET), kerentanan IGC dengan ujian kehilangan jisim asid nitrik (NAMLT), ujian kelakuan elektrokimia dalam larutan NaCl 3.5% oleh penetuluaran Tafel (TE) dan polarisasi potensiodinamik (PDP), dan ujian kakisan rendaman penuh dalam larutan 3.5% NaCl dalam keadaan statik dan kelajuan tinggi pada suhu bilik. Sifat tegangan, dan ujian hentaman juga dilaksanakan bagi menentukan sifat mekanikal aloi tersebut. Selain itu, ujian ramalan hakisan dilaksanakan dengan menghentam partikel pepejal sfera pada sudut normal. Hasil kajian menunjukkan, mencampurkan Ce, Pr, dan Ce + Pr pada aloi AA5083 mengurangkan saiz butiran. Saiz butiran berkurang dari 105µm ke 72 µm dan 107 µm ke 74 µm masing-masing bilamana Ce dan Pr ditambah dari 0.1 ke 1.0% berat. Penambahan kombinasi Ce + Pr dalam jumlah 0.1 to 0.5% berat menghasilkan pengurangan saiz butiran purata dari 98µm ke 65µm. Kewujudan fasa endapan stabil (REE diperkaya) memainkan peranan penting dalam penghalusan butiran, dengan itu menambahbaik sifat mekanik. Fasa stabil tersebut mengurangkan fasa-β (Al_3Mg_2) dan fasa Fe dan Mg diperkaya, yang merupakan penyebab utama LC, IGC dan IGSCC, dengan itu menambahbaik rintangan kakisan. Sifat-sifat ini lebih terserlah bagi kombinasi Ce + Pr berbanding campuran secara individu Ce atau Pr. Aloi AA5083 yang diubahsuai dengan campuran 0.5% Ce + 0.5% Pr menghasilkan penambaikbaikan keseluruhan yang terbaik dari segi sifat mekanik dan kelakuan kakisan berbanding dengan aloi AA5083 lain yang diubahsuai. Saiz butiran purata berkurang sebanyak 39.8%. Keliatan, kekuatan alah dan tegangan utama ditambahbaik masing-masing sebanyak 21%, 27.5% dan 53.3% manakala kadar kakisan berkurang sebanyak 31.2%. Dengan itu, REEs boleh dicadangkan sebagai calon yang sesuai sebagai unsur paduan bagi AA5083.

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LIST OF ABBREVIATIONS

ISO	-	International Organization For Standardization
ASTM	-	American Society For Testing And Materials
REEs	-	Rare Earth Elements
LREEs	-	Light Rare Earth Elements
HREEs	-	Heavy Rare Earth Elements
NDT	-	Non-Destructive Testing
VT	-	Visual Inspection
LT	-	Liquid Penetrant Testing
UT	-	Ultrasonic Testing
IMCs	-	Intermetallic Compounds
NAMLT	-	Nitric Acid Metal Loss Test
ASSET	-	Assessment Of The Exfoliation Test
IGC	-	Intergranular Corrosion
IGSCC	-	Intergranular Stress Corrosion Cracking
PDP	-	Potentiodynamic Polarization
TE	-	Tafel Extrapolation
EFC	-	Exfoliation Corrosion
Ecorr	-	Corrosion Potential
i_{corr}	-	Corrosion Current Density
E_{pit}	-	Pitting Potential
I_{pass}	-	Passivation Current Density
WE	-	Working Electrode
RE	-	Reference Electrode
CE	-	Counter Electrode
SCE	-	Saturated Calomel Electrode
CR	-	Corrosion Rate
CNC	-	Computer Numerical Control
XRD	-	X-Ray Diffraction

OM - Optical Microscopy
LOM - Low Power Magnification Light Optical Microscope
HOM - High Power Magnification Light Optical Microscope
EDS - Energy Dispersive X-Ray Scanning
VPSEM - Variable Pressure-Scanning Electron Microscope
GZ - Grain Size
ASL - Altered Surface Layer
TS - Tensile Strength
UTS - Ultimate Tensile Strength
Wt.% - Weight Percentage
rpm - Rotation per Minute

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CHAPTER 1

INTRODUCTION

1.1 Background of the study

Newly modified alloys with exceptional characteristics are created every day for specific applications. The AA5083 aluminium-magnesium-manganese alloy is generally classified as a non-heatable alloy. It is characterised by certain unique properties that have made it one of the most important and commercially popular alloys. AA5083 has a low density and a high strength-to-weight ratio, has ideal welding properties, is easy to machine and form medium strength, is able to resist corrosion in marine and industrial environments, and is available at a reasonable cost. These particular properties make it a clear choice for a variety of engineering applications such as marine technology (shipbuilding materials), the automotive industry, the aerospace industry, pressure vessels, armoured vehicles, and armour plate (Kaibyshev *et al.*, 2003; Liu *et al.*, 2017; Sunny *et al.*, 2013).

On the other side, AA5083 alloy is susceptible to intergranular corrosion (IGC) and intergranular stress corrosion cracking (IGSCC) when exposed to temperatures between 50°C and 200°C. This is due to sensitisation process, which is a consequence of the formation of β-phase Al_3Mg_2 at the grain boundary. This phenomenon also has an adverse effect on mechanical properties in which the β-phase leads to a decline in the hardness and strength of the material because of a reduction of the Mg concentration in the aluminium solid solution (Oguocha *et al.*, 2008; Searles *et al.*, 2001; Vetrano *et al.*, 1997). In addition, the AA5083 alloy is susceptible to localised corrosion in NaCl solution in the form of pitting corrosion, which occurs as a result of the formation of Fe-rich phases such as $Al_6(Fe, Mn)$ and Al(Mn, Fe, Cr), as well as Mg-rich phases such as Mg_2Si (Aballe *et al.*, 2001; Lucadamo *et al.*, 2006; Yasakau *et al.*, 2007). This phenomenon becomes more pronounced when the alloy is subjected to high-speed conditions (Jafarzadeh *et al.*, 2009; Jo *et al.*, 2002). The above factors limit the use of the AA5083 alloy in several critical applications.

To address the limitations, the microstructure, mechanical properties, and corrosion resistance of aluminum and aluminum alloys, it is improved by adding an appropriate amount of rare earth elements (REEs: Ce, Sc, Pr, La, Er, Y, Nd, Eu, Pm Gd, Dy, Ho, Tb, Tm, Yb, Lu, and Sm), as indicated by various previous studies (Jin, 2012; Umarova, 2017). Therefore, the addition of REEs to AA5083 is a promising topic. Ce has successfully been used on AA5083 and other Al alloys. The addition was made on individual elements in the range of 0.1wt% to 0.6wt%, which enabled the mechanical properties (hardness, tensile strength, and elongation) to be enhanced. Additionally, praseodymium (Pr), may provide a similar effect since it belongs to the light rare-earth elements (LREEs) as Ce.

Several studies have been conducted to determine the effect of Pr addition as individual alloy element to Al alloy such as Al-Cu, Al metal matrix composites (Al_2O_3-SiO_2) and Ce-Pr combination on Al-7Si-0.7Mg alloy and Al-8.52Zn-2.41Mg-2.1Cu-0.16Zr. In general, results showed that the addition of Pr as individual elements improved the microstructure and mechanical properties. Addition of a combination of Ce-Pr resulted in, not just microstructure and mechanical properties but also enhanced the exfoliation corrosion (EXCO) and IGC resistance.

In short, the REEs are critically important as alloying elements in modifying Al-alloys to enhance its microstructure, mechanical properties, and corrosion resistance. The optimal addition of REEs varies between different elements and alloys, such that the selection of the optimal quantity of the addition depends on the type of element added as well as the alloy type (a type of the modified alloy). In addition, previous studies have shown that the addition of REEs as a mixture achieves more benefits than individual REE additions. However, the researches were mainly emphasised on investigating the properties by individual element addition. Therefore, this research was aimed at improving the microstructure, mechanical properties, and corrosion behaviour of AA5083 by adding rare earth elements in combined form. The research investigated the addition of a combination of Ce-Pr in the amount of 0.5wt%

each. In addition, the investigation was also conducted on the addition of individual element Ce and Pr in the amount ranging between 0.1wt% and 1wt%.

1.2 Problem statement

Aluminium-magnesium-manganese (Al-Mg-Mn) alloys in particular AA5083 alloy has good corrosion resistance where its applications are best used in the marine environment and industrial atmospheres. However, its medium mechanical strength (Huang *et al.*, 2017) and its susceptibility to localised corrosion attack in a corrosive medium (NaCl), which are both caused by the presence of Mg-rich and Fe-rich phases (Aballe *et al.*, 2001; Czechowski, 2005; Yasakau *et al.*, 2007) has limits it uses in many applications. This phenomenon becomes more pronounce when the alloy is subjected to high-speed conditions (Gehring Jr, 1979; Gehring Jr and Peterson, 1981). Another problem is its susceptibility to IGC and IGSCC when exposed to temperatures between 50°C and 200°C (Jones *et al.*, 2001; Oguocha *et al.*, 2008; Searles *et al.*, 2001) resulting from precipitation of the β-phase (Mg_2Al_3) along the grain boundary, which acts as an anodic site relative to the AA5083 alloy matrix.

Many elements have been used to improve the mechanical properties of aluminium alloys. However, research has shown that besides other elements such as Zr, Sc, and Sr, REEs have been added to aluminium alloy to improve its microstructure, mechanical properties, and corrosion resistance. These include Nd, Er, Ce, La, and Pr. Ce has been widely researched in altering the microstructure and mechanical properties of Al-Mg alloys. However, the investigations were limited to microstructure and mechanical properties studies only. For marine and industrial environment applications particularly corrosive environment, where AA5083 alloy is much suited, very limited works have been carried out to investigate the effect of REEs addition. Nd, Sc, and Ce as an individual element added in the range of 0.1wt% to 0.5wt% to AA5083 alloy proved to refines the grain size and thus enhance the mechanical properties of the alloy even though REEs added to other Al alloys in the amount of up to 1wt% were found to produce a better effect. It indicated that the capability of the REEs either being individually added or in combination to enhancing the microstructure, mechanical properties and corrosion resistance on AA5083 has not been fully explored. Therefore, further investigation is needed to explore the possibility of adding REEs on the microstructure, mechanical properties, and corrosion behaviour of AA5083 alloy.

1.3 Research objectives

The overall objectives of this research were to evaluate the influence of certain REE (Ce, Pr, and Ce+Pr) additions on the microstructure, mechanical properties, and corrosion behaviour of newly developed AA5083 alloys. Specific objectives are as follows:

1. To determine the effect of different amounts of Ce, Pr, and a mixture of Pr and Ce additions on the microstructure and phase/compound of the modified AA5083 alloy.
2. To investigate the mechanical properties of the modified AA5083 alloy using tensile and Charpy tests
3. To investigate the influence of different amounts of Ce, Pr, and a mixture of Pr and Ce additions on the corrosion behaviour of the modified AA5083 alloys.

1.4 Scope of the research

The scope of the research is designed to satisfy the research objectives as follows:

1. Fabricate three types of modified AA5083 alloy via the in-situ casting technique using an induction furnace in a controlled atmosphere. The three modified AA5083 alloys are:
    i. AA5083 + xCe alloys {x = 0.1, 0.3, 0.5, 0.7 and 1.0wt%}
    ii. AA5083 + yPr alloys {y = 0.1, 0.3, 0.5, 0.7 and 1.0wt%}

    iii. AA5083 + Mm alloys (Mm = xCe + yPr), where (x:y) = {(0.1:0.1), (0.1:0.3), (0.1:0.5), (0.3:0.1), (0.3:0.3), (0.3:0.5), (0.5:0.1), (0.3:0.5) and (0.5:0.5)}

2. Perform non-destructive testing (NDT: visual inspection, VT; liquid penetrant testing, LT; and ultrasonic testing, UT) for cast product (modified AA5083 alloy) quality assessment.
3. Perform the homogenisation process at 450°C for 24hr by using an electric furnace in a controlled atmosphere.
4. Perform microstructure characterisation on the modified AA5083 alloys using an optical microscope, a variable pressure scanning electron microscope (VPSEM) with energy disperse x-ray spectroscopy (EDS), and x-ray diffraction analysis (XRD) as well as image processing techniques using Image J Software.
5. Perform mechanical tests for the modified AA5083 alloys, including tensile tests at room temperature and Charpy impact tests at both room temperature and subzero temperature, according to ASM standard procedures.
6. Perform corrosion tests for the modified AA5083 alloys, including IGC susceptibility by mass loss after exposure to nitric acid test (NAMLT), exfoliation and pitting corrosion susceptibility (ASSET-Test), and accelerated electrochemical corrosion testing through potentiodynamic polarization (PDP) and Tafel extrapolation (TE) techniques. The erosion test is done by solid particle impingement at normal incidence, full immersion corrosion test, and full immersion corrosion test under high-speed conditions (corrosion-erosion).

1.5 Significance of the research

This study is an effort to provide significant information about the microstructure, mechanical properties, and corrosion behaviour of the AA5083 alloy and the modified AA5083 alloys with the REEs (Ce and Pr) in the form of individual or combined additions. The findings of this study will benefit the industries that use the AA5083 alloy in their applications, such as the shipbuilding, automotive, pressure vessel, armour plate, and aerospace industries. Positive results are expected from the implementation of the microalloying technique via Pr, Ce, and Pr + Ce additions. The outcome will extend the scope of application of the modified AA5083 alloys and will enhance the importance of using REE additives as alloying elements in the development of the AA5083 alloy. Future researchers are encouraged to utilise the microalloying technique using REE additions.

1.6 Thesis organisation

This thesis is organized into five chapters. The **first chapter** is an introduction to the study. It aims to provide a brief overview of the research, including the research background, problem statement, research objectives, scope of the research, and significance of the research. The **second chapter** is a comprehensive literature review related to the study topic (microstructure, mechanical properties, and corrosion behaviour of AA5083 alloys modified with REEs). The **third chapter** describes the research methodology for this study and presents a detailed description of the experimental procedures. In the **fourth chapter,** the results and discussion for the experimental work are presented in five phases according to the sequence in the third chapter. The **fifth chapter** provides conclusions based on the results and discussions. Recommendations for further study based on current research gaps that were recognized during this study will be highlighted in the fifth chapter.

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APPENDIX O

Publications:

1) A. Ali Al-Bakoosh, Jamaliah Idris (2018) ‘Impact of rare earth elements ce and pr addition on grain refinement of AA5083 alloy’, Journal of Chemical Technology and Metallurgy, 53(5), 916-923.

2) A. Ali Al-Bakoosh and Jamaliah Idris (2018) ‘Effect of cerium and praseodymium additions on impact toughness of AA5083 alloy at room temperature and sub-zero-temperature ’, *Contemporary Engineering Sciences,* 11 (52), 2551-2562.

3) Jamaliah Idris, A.Al-Bakoosh (2014) ‘Application of non-destructive testing techniques for the assessment of casting of AA5083 alloy’, *Journal of Advanced Research in Applied Mechanics*, 3(1), 25-34.

4) I. J, A. A. Al bakoosh and S.T. Olohunde (2020) ‘Effect of rare earth elements (C and Pr) addition on the eroded AA5083 alloy by a solid spherical particle impingement ’, International Journal of Advanced Science and Technology, 29 (02), 1763-1772.

5) S. Olohunde, Abd. M, Idris Jamaliah, A. Ali Al-Bakoosh (2019) ‘Corrosion resistance of aluminium–silicon hypereutectic alloy from scrap metal’, Journal of Bio- and Tribo-Corrosion 5(41), 1- 8.