UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Faculty of Manufacturing Engineering

STUDY ON SURFACE RETROGRESSION AND RE-AGING (SRRA) BEHAVIOUR OF ALUMINIUM ALLOY 7075

Nor Azrin Binti Nozmi

Master of Manufacturing Engineering
(Manufacturing System Engineering)

2013

STUDY ON SURFACE RETROGRESSION AND RE-AGING (SRRA) BEHAVIOUR OF ALUMINIUM ALLOY 7075

NOR AZRIN BINTI NOZMI

A thesis submitted

in fulfillment of the requirements for the degree of Master of Manufacturing

Engineering (Manufacturing System Engineering)

Faculty of Manufacturing Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2013

DECLARATION

I declare that this thesis entitles Study on Surface Rerogression and Re-aging (SRRA) Behavior of Aluminium Alloy 7075 is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any degree.

Signature : ....

Name : **NOR AZRIN BINTI NOZMI**

Date : ....

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Master of Manufacturing Engineering (Manufacturing System Engineering). The members of supervisory committee are as follow:

....

Prof. Madya Dr. Mohd Warikh Bin Abd. Rashid

PROF. MADYA DR. MOHD WARIKH BIN ABD. RASHID
Deputy Dean (Research & Post Graduates Studies)
Faculty of Manufacturing Engineering
Universiti Teknikal Malaysia Melaka
Hang Tuah Jaya
76100 Durian Tunggal, Melaka

DEDICATION

Special to my beloved husband, Muhammad Izwan Arief Bin Mat Nor

and my dear parents & family

ABSTRACT

Aluminum alloy 7075 is a very high strength material and have a wide range application throughout automotive, aircraft and aerospace industries, which possesses good mechanical properties with a combination of high strength, low density, ductility, and moderate toughness but often disposed to corrosion attack especially stress-corrosion cracking (SCC). The effects of the mechanical properties and stress corrosion cracking behavior were investigated to aluminium alloy 7075 when introduce to surface-retrogression and re-aging (SRRA) heat treatment and compared with T6, T73 and retrogression and re-aging (RRA) results. The method starts with carrying out solution heat treatment at 470°C for 1 hour. The samples were then quenched and followed by ageing process at 120°C from 0h to 24 hours ageing time. The 7075-T6 was obtained at this stage while 7075-T73 acquired after over-aged at 170°C for 8 hours. The samples were exposed to heat temperature at 160°C, 180°C and 200°C, at holding time 5 and 25 minutes respectively, which produced RRA treated samples. At the same temperature and holding time as RRA, the samples were then introduced to oil on the surface of each specimen to produce the SRRA samples. After all, the RRA and SRRA samples were exposed to re-aging process at 120°C for 24 hours. Evaluation was based on mechanical properties, microstructure evaluation and corrosion test. It was found that the hardness value for SRRA (from 86.7 – 70.7HRB) progressively reduced as the temperature and holding time increased if compared to T6 (88.8 HRB), but the strength of SRRA-treated (around 602.44 N/mm^2 - 658.52 N/mm^2) was similar to T6 (651.53 N/mm^2) but much higher than T73 (541.56 N/mm^2) temper. As for the microstructure evaluation of SRRA, the precipitation of η' phase inside the grains broaden and become denser as the temperature increase. The η' of SRRA became thicker as the temperature and time increased if compared to RRA. This finding suggests that the long retrogression time at higher retrogression temperature cause the hardness value decrease due to η' and η phases coarsening. It was found that the corrosion rate value of SRRA-treated without load is 0.0196 mm/yr and decrease to 0.0031 mm/yr when applied load. This result can be related to the crack occurrence at grain boundaries once the load was applied and encourage the corrosion to penetrate and grow at the grain boundaries. This is significant to the strength of the specimen where the strength of RRA and SRRA-treated is lower in this state as compared to the T6. The findings suggest that study for SRRA rotation speed can be conducted because it may be influenced to the microstructural stability since the heating of the alloy only be held at the surface and additional testing is recommended using Finite Element Method (FEM) in order to determine the behavior of SCC.

ABSTRAK

Aloi aluminium 7075 adalah logam berkekuatan tinggi dan digunapakai secara meluas seperti bahagian pesawat dan industri aeroangkasa, serta memiliki sifat-sifat mekanikal yang baik dengan gabungan kekuatan yang tinggi, ketumpatan yang rendah, kemuluran, dan keliatan sederhana. Namun bahan ini sering terdedah kepada serangan kakisan terutama kakisan tegasan. Oleh itu, kajian berkenaan sifat-sifat mekanikal dan ciri-ciri kakisan tegasan terhadap jenis aloi aluminium 7075 apabila didedahkan kepada proses rawatan haba jenis 'surface retrogression and re-aging' (SRRA) dilakukan dengan melihat perbandingan antara T6, T73 dan RRA. Kaedah ini bermula dengan menjalankan rawatan haba pada suhu 470°C selama 1 jam. Sampel kemudiannya dilindapkejut dan diikuti dengan proses penuaan pada suhu 120°C dan direndam dari 0h sehingga 24 jam. Sampel jenis 7075-T6 diperolehi pada peringkat ini manakala sampel jenis 7075-T73 diperolehi selepas dipanaskan pada suhu 170°C selama 8 jam. Sampel kemudiannya didedah kepada suhu 160°C, 180°C dan 200°C, dan masing-masing direndam selama 5 dan 25 minit bagi menghasilkan sampel RRA. Pada suhu dan masa yang sama, sampel kemudian didedahkan kepada minyak di permukaan setiap spesimen untuk menghasilkan sampel jenis SRRA. Kemudian, kesemua sampel RRA dan SRRA didedahkan kepada proses penuaan semula pada suhu 120°C selama 24 jam. Penilaian dilakukan berdasarkan kepada sifat-sifat mekanik, ujian mikrostruktur dan sifat-sifat hakisan. Hasil ujikaji telah mendapati bahawa nilai kekerasan bagi SRRA (dari 86.7 - 70.7HRB) menurun apabila suhu dan masa rawatan haba dipanjangkan jika dibandingkan dengan T6 (88.8 HRB). Walaubagaimanapun kekuatan SRRA (sekitar 602,44 N/mm2 - 658,52 N / mm2) secara purata adalah menghampiri sampel T6 (651.53 N/mm2) dan lebih tinggi daripada sampel T73 (541.56 N/mm2). Melalui pemerhatian mikrostruktur bagi sampel SRRA, wujud pemendakan fasa η 'di dalam bijirin semakin berkembang dan menjadi lebih padat dengan peningkatan suhu. Ketebalan η' bagi SRRA semakin meningkat apabila suhu dan masa rawatan meningkat jika dibandingkan dengan RRA. Penemuan ini menunjukkan bahawa masa kemunduran yang panjang pada suhu yang lebih tinggi boleh menyebabkan nilai kekerasan berkurangan akibat daripada perubahan fasa η' kepada η. Selain itu, hasil kajian mendapati bahawa nilai kadar kakisan bagi SRRA yang dirawat tanpa beban adalah 0.0196mm/tahun dan menurun kepada 0.0031mm/tahun apabila beban digunakan. Keputusan ini boleh dikaitkan dengan kejadian retak di sempadan bijian apabila beban digunakan dan menggalakkan hakisan untuk berkembang pada sempadan bijian. Hal ini berhubungkait dengan nilai kekerasan RRA dan SRRA yang mana mempunyai nilai lebih rendah berbanding sampel T6. Oleh hal yang demikian, kajian lanjutan boleh dilakukan kepada kelajuan putaran sewaktu mengendalikan proses SRRA kerana hal ini mungkin boleh mempengaruhi kestabilan mikrostruktur memandangkan pemanasan aloi hanya akan diadakan di permukaan sahaja.

ACKNOWLEDGEMENT

I would like to thank to Allah Almighty for blessing and giving me the strength to accomplish this study. A special thanks to everyone attributes to accomplish this study, especially to my supervisor, Prof. Madya Dr. Mohd Warikh Bin Abd. Rashid, for the good advice and guidance. Also thanks to Dr. Nur Izan Syahriah Bt Hussein for the motivation, lecturers, technicians, husband, parents, family, friends and UTeM staff. Finally special honored to Universiti Teknikal Malaysia Melaka, UTeM for providing me with all the knowledge.

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

°C	–	Degree celcius
μA	–	Micro ampere
$\mu A/cm^2$	–	Micro ampere/centimetre square
ρ	–	Density
A\`	–	Final cross sectional area
A_0	–	Initial cross sectional area
Al	–	Aluminium
cm^2	–	Centimetre square
Cr	–	Chromium
CR	–	Corrosion penetration rate
Cu	–	Cuprum
E_{corr}	–	Corrosion potential
Fe	–	Ferum
g/cm^3	–	gram/centimetre
GP	–	Guinier-Preston
HCL	–	Hydrocloric acid
HF	–	Hydrofluoric acid
HNO_3	–	Nitric acid
HRB	–	Hardness Rockwell scale B

i_{corr}	–	Corrosion current density
I_{corr}	–	Total anodic current
in.	–	inch
Kgf	–	kilogram force
kN	–	kilo Newton
L`	–	Final gauge length
L_0	–	Initial gauge length
$MgZn_2$	–	Magnesium Zinc
mm	–	milimetre
mm/yr	–	millimetre/year
Mn	–	Mangenese
N/mm^2	–	Newton/millimetre square
OM	–	Optical Microscope
RRA	–	Retrogression and re-aging
SCC	–	Stress corrosion cracking
Si	–	Silicon
SRRA	–	Surface-retrogression
Ti	–	Titanium
UTM	–	Universal Tensile Machine
V	–	Voltage
XRF	–	X-Ray Fluorescence Spectroscopy
Zn	–	Zinc

TABLE OF CONTENT

CHAPTER 1

INTRODUCTION

1.1 Background of The Project

Aluminum is a well known material with its lightweight properties and resistant to corrosion that capable to be strengthened through alloying, heat treatment and/or cold working depending upon composition and strengthened principal. Aluminum and its alloy generally have good electrical and thermal conductivities, and can be melted, cast, formed or machined as much as other metals can perform. This is due to its special properties which has low melting point and density as well as ductile. These are the characteristics of aluminum which grant extreme versatility includes in transportation equipment, petroleum industries, food processing equipment, and building structural.

Aluminum alloy 7075 is a very high strength material used for highly stressed structural parts. This type of aluminum has a wide range application throughout automotive, aircraft and aerospace industries, which possesses good mechanical properties with a combination of high strength, low density, ductility, and moderate toughness but disposed to corrosion attack especially stress-corrosion cracking (SCC). This was agreed by Li et. al. (2007) where the 7xxx series of aluminum alloys including 7075 aluminum alloy are sensitive to localized corrosion such as intergranular corrosion and stress corrosion cracking which required enhanced corrosion resistance application.

When improved corrosion resistance is required, 7xxx series aluminum alloy is often exposed to several types of heat treatment such as over-aging treatments which are T73, T76 and T74. In contrast, this will affected to the decreasing of the strength of the alloy. As for 7075 aluminum alloy, it is often used T6 and T73 heat treatment in turn for improving corrosion resistance. The T6 temper acquired high strength, but low toughness and poor stress-corrosion cracking resistance while T73 temper enhanced the stress-corrosion resistance over T6 temper with a decrease in strength. Somehow, an alternative to improve both strength (equivalent to T6) and high stress corrosion resistance properties (equivalent to T73) in a single temper is via performing the retrogression and re-aging (RRA) heat treatment on parts in the T6 condition (Military Handbook, 2003). This process has been proven by Peeler *et. al*. (2003) where the result shows that RRA in the T6 temper definitely enhance the stress corrosion resistance while maintaining the strength at the level or slightly below the T6 state.

During T6 condition, a reduction in hardness is occurred due to dissolve of the Guinier-Preston (GP) zones and enhance in solid solution. The remaining ŋ' grows on the aluminium grains in a short period, but it begins to fall again as ŋ' coarsen and start to transform to ŋ. Therefore, the RRA process provides an alloy with a strength slightly below the single T6 temper with resistance to corrosion cracking almost equivalent to T73 material.

Previous works have mostly focused on the effect of RRA treatment on the stress corrosion cracking resistance, microstructure as well as mechanical property changes for 7XXX series alloys as verified by Murat *et. al*. (2004), Xu *et. al*. (2011), Li *et. al*. (2010) and Yan *et. al*. (2010). However, there is no published data concerning the effect of surface-retrogression treatment of surface corrosion cracking resistance and properties behavior of this alloy. Thus, in this project, experiment is conducted in order to study the effects of the mechanical properties and stress corrosion cracking behavior of aluminium alloy 7075 when introduce to surface-retrogression and re-aging heat treatment.

1.2 Problem Statement

Alloy aluminum 7075 most widely used as structural material in aircraft and aerospace industries due to its combination of high strength with moderate toughness and resist to corrosion cracking compared to many other aluminum alloys. However, according to Li *et. al*. (2007) the authors claimed that 7075 aluminium alloys are sensitive to localized corrosion, especially stress corrosion cracking (SCC). The in-service stress corrosion cracking failures can be caused by stresses produced from a wide variety of sources.

The corrosion resistance of this alloy can only be modified by heat treatment. Therefore, in order to increase the corrosion resistance, T73 heat treatment was developed but this treatment will reduce the strength of the material after over-aging treatment. As for T6 treatment, it's a vice versa of T73 which possess high strength, but poor in corrosion resistance. Viana *et. al*. (1999) claim that the retrogression temperature is influence the microstructural stability after re-aging heat treatment. The greater the retrogression temperature, the more stable is the microstructures obtained after re-aging. Thus, the resistance to corrosion cracking can be obtained almost the same result with T73 but slightly decrease their strength.

During applications on industrial heat treatments, it is difficult to retain the temper process if the products are coming from many different sizes and thickness. The retrogression process in RRA temper is relatively short, depends on the applied temperature. The time of re-aging temper (177°C for 5-6 hours) will define the temper variations as well as the mechanical properties and SCC performance as stated by Oliveira *et. al*. (2004). Therefore, it can be predicted that heating temperature is a key factor to microstructure evolution during retrogression.

Many different heat treatment cycles with various parameters were conducted to aluminum alloy in order to look at the effects on microstructures behaviors, corrosion resistance behavior, and mechanical and physical properties of the aluminum alloy. Therefore, this study is to analyze the effect of SCC behavior on aluminum alloy 7075 which possess T6, T73, RRA and surface-retrogression (SRRA) heat treatment cycle. The SRRA is introduced in order to evaluate the surface hardness of the aluminum alloy 7075 after exposed to retrogression treatment.

1.3 Objective

The overall objective of this project is to report the susceptible strength and stress corrosion cracking behavior mainly on surface-retrogression and re-aging (SRRA) heat treatment. The objectives of this project are:

i. To evaluate the effect of physical and mechanical properties of aluminum alloy 7075 after exposed to different heat treatment.

ii. To analyze the influence of surface-retrogression and re-aging (SRRA) on tensile properties, corrosion behavior, and microstructure morphology of aluminium alloy 7075.

iii. To evaluate the stress corrosion cracking behavior of aluminium alloy 7075 after exposing to SRRA heat treatment.

1.4 Scope

This study is mainly focused on the improvement of mechanical properties and corrosion resistance after surface hardening of alloy aluminium 7075 via surface-retrogression and re-aging method. The scope of this project can be categories into three main parts which are:

i. Material preparation

  a. Carry out T6 and T73 temper process through a solution heat treatment procedure accompany with aging and quenching on aluminium alloy 7075.
  b. Carry out RRA heat treatment in the T6 condition of aluminium alloy 7075 incorporate with surface-retrogression treatment.
  c. Quenching process will be using water/ice water medium.

ii. Microstructure and material properties testing

  a. Carry out mechanical testing to each T6, T73, RRA-treated and SRRA-treated samples include microstructures morphology towards evaluating the evolution of the precipitation alloying element.
  b. Both mechanical properties and microstructure morphology of T6, T73 and RRA-treated samples will be used as an indicator for SRRA upon interpretation of the changing behavioral after heat treated process.
  c. Material specimen for mechanical properties will be prepared according to ASTM standard and Optical Microscope (OM) is used to analyze the precipitate alloying element in the grain boundaries of the specimen.

iii. Corrosion behavior evaluation

  a. The Tafel extrapolation technique will be used to evaluate the corrosion potential rate among all samples.
  b. The stress corrosion cracking behavior will carry out by exposing the specimens to corrosive environment while subjected to tensile load to determine the corrosion cracking behavior.

1.5 Significance of Study

Aluminium alloy 7075 is a high strength alloys which available in a wide range of product form especially in aeronautical industry or aircraft structural parts. The study of SCC behaviour after surface-retrogression may be used for aeronautical industry as a point of references to improve the research and development (RnD) of their products, by determining the relevant parameter of heat treatment processes. The improvement of the properties not only beneficial for the products only but may influence the cost of production and maintenance which highly impact to the use of this alloy. Moreover, the application of aluminium alloy 7075 will be further extending to other industrial sector in the future due to uniqueness behaviour which is high mechanical properties and high stress corrosion cracking resistance.

CHAPTER 2

LITERATURE REVIEW

2.1 Aluminium Alloy 7xxx Series

7xxx series aluminium alloys are widely used as structural material in the aerospace industry which provide high strength and stiffness but poor stress corrosion resistance (Rajan *et. al.* (1982). Aluminium alloy 7075 is a high strength alloy and available in several types of tempers including T6, T73 and T76 type in order to improve the properties and corrosion resistance behavior (Military Handbook, 2003). It has been used for a long time before in aerospace industry mainly for critical parts due to its high strength and light weight properties.

Although other 7XXX alloys have been improving their properties, alloy 7075 remains to be the point of reference due to its good balance of properties which required for aerospace applications. Recently, they used in aluminium alloy has been extended and applied not only for mechanical industries but often used in transport applications, including marine, automotive and aviation. The typical composition in percent by weight of aluminium alloy according to Aerospace Aluminium Alloy (UAC, 2010) can be roughly described as in Table 2.1.