



Faculty of Manufacturing Engineering

**CHARACTERIZATION OF OIL RETROGRESSION OF ALUMINUM
ALLOY 7075**

NG GUAN YAO

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**CHARACTERIZATION OF OIL RETROGRESSION OF ALUMINUM
ALLOY 7075**

NG GUAN YAO

**A thesis submitted
in fulfillment of the requirements for the degree of Master of Science in
Manufacturing Engineering**

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2014

DECLARATION

I declare that this thesis entitle “Characterization of Oil Retrogression of Aluminum 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 other degree.

Signature :

Name : NG GUAN YAO

Date :

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering.

Signature :

Supervisor Name : PM DR. MOHD WARIKH BIN ABD RASHID

Date :

ABSTRACT

Aluminum alloy 7075 is a useful industrial material thanks to its high strength to weight ratio. However, it is susceptible to corrosion due to numerous constituent particles with various electrochemical potentials the combination of which leads to susceptibility to corrosion. This issue was solved by using overage treatment with the penalty of 10-15% of peak strength. Previous research found that retrogression and re-aging (RRA) heat treatment improves corrosion resistance while maintaining the strength of the alloy. In this research, the retrogression process was studied to a greater extent with oil as the heating medium in relation to the standard retrogression. The tensile specimens are peak aged before retrogressed at 165°C, 185°C, and 205°C for 10 and 30 minutes in oil and standard furnace followed by re-aging at 120°C for 24 hours. Next, the specimens were put through tensile test, hardness test, Tafel DC corrosion test, and microstructure characterization by using Scanning Electron Microscope (SEM). The result of the analysis showed the mechanical properties of the alloy to decrease with increasing retrogression temperature and time for both oil and standard retrogression due to the increase in incoherent η phase volume fraction as observed under the SEM analysis. In comparison between oil and standard retrogression, oil retrogression out-performed standard retrogression at 165 °C for 10 minutes with better corrosion resistance at corrosion current density of 6.64 $\mu\text{A}/\text{cm}^2$ and tensile strength of 632.73 MPa.

ABSTRAK

Aluminium aloi 7075 adalah bahan industri yang sangat berguna atas sebab nisbah kekuatan kepada berat badan yang tinggi. Walau bagaimanapun, ia mudah terdedah kepada kakisan kerana banyak zarah jujuk yang mempunyai pelbagai potensi elektrokimia dan kombinasinya mengakibatkan kecenderungan kepada kakisan. Isu ini telah diselesaikan dengan menggunakan rawatan haba T73 dengan penalti sebanyak 10-15% daripada kekuatan puncak. Kajian sebelum ini telah menemui rawatan haba kemunduran dan penuaan semula akan meningkatkan rintangan kakisan di sampingnya masih mengekalkan kekuatan aloi. Dalam kajian ini, proses kemunduran telah dikaji dengan peringkat selanjutnya dengan menggunakan minyak sebagai medium pemanasan yang berhubung dengan kemunduran biasa. Spesimen dirawat dengan rawatan T6 dan kemudiannya dimundur pada 165 °C, 185 °C, dan 205 °C selama 10 minit dan 30 minit dalam minyak dan relau diikuti oleh penuaan semula pada 120 °C selama 24 jam. Seterusnya, spesimen dikaji dengan ujian tegangan, ujian kekerasan, ujian kakisan Tafel DC, dan pencirian mikrostruktur dengan menggunakan mikroskop imbasan elektron. Keputusan analisis telah menunjukkan sifat-sifat mekanik aloi akan berkurang dengan peningkatan suhu kemunduran dan masa bagi kedua-dua kemunduran minyak dan kemunduran biasa yang disebabkan oleh peningkatan pecahan isipadu fasa η yang tidak keruan sebagaimana yang diperhatikan di bawah analisis mikroskop imbasan elektron. Berbanding di antara kemunduran minyak dan kemunduran biasa, kemunduran minyak mengatasi prestasi kemunduran biasa pada 165 °C selama 10 minit dengan rintangan kakisan yang lebih baik pada kakisan ketumpatan arus $6.64\mu\text{A}/\text{cm}^2$ dan kekuatan tegangan 632.73 MPa.

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LIST OF ABBREVIATIONS

Al	-	Aluminum
ALCOA	-	American Aluminum Industry Company
Al ₂ O ₃	-	Aluminum Oxide
at.	-	Atomic
Cr	-	Chromium
Cu	-	Copper
DC	-	Direct Current
E _{corr}	-	Corrosion Potential
EXCO	-	Exfoliation Corrosion
F	-	Basic temper designation (As fabricated)
GP zone	-	Guinier-Preston zone
H	-	Basic temper designation (Strain hardened)
H ₂ O ₂	-	Hydrogen Peroxide
IACS	-	International Annealed copper Standard
I _{corr}	-	Corrosion Current Density
IGC	-	Inter-Granular Corrosion
Mg	-	Magnesium
NaCl	-	Sodium Chloride
O	-	Basic temper designation (Annealed)
o	-	Oil (retrogression medium)
PFZ	-	Precipitates Free Zone
RRA	-	Retrogression and Re-Aging
SCC	-	Stress Corrosion Cracking
SEM	-	Scanning Electron Microscope
SHT	-	Solution Heat Treatment
Si	-	Silicon
SSSS	-	Super-Saturated Solid Solution
TEM	-	Transmission Electron Microscopy

UTS	-	Ultimate Tensile Strength
v_{η}	-	Volume Fraction of η phase
v_{GP}	-	Volume Fraction of GP zone
$v_{\eta'}$	-	Volume Fraction η' phase
W	-	Basic temper designation (Thermally Treated)
wt.	-	Weight
XRD	-	X-ray Diffraction
Zn	-	Zinc

LIST OF PUBLICATIONS

NG, G.Y., Warikh, A.R., Zolkepli, B., Nadiah, N., & Leng, S.C., 2012. Effect of Retrogression Medium to the Mechanical Properties of Aluminum Alloy 7075. *Journal of Applied Mechanics and Materials*, 165, pp.6-11.

CHAPTER 1

INTRODUCTION

1.1 Background

Aluminum is a light weight and soft metal in the pure state. With the addition of alloying element such as titanium, copper, zinc, carbon, or chromium, its properties will change and produce an aluminum alloy in various series from 1xxx to 9xxx. Among these alloys, the 7xxx series alloy, in which the major addition is zinc (Zn) possesses good mechanical properties. Aluminum alloy 7075 with the addition of zinc, copper, and magnesium has the top mechanical strength in the 7xxx series alloys.

In the aerospace and automotive industries, aluminum alloy 7075 is widely used due to its high strength to weight ratio (Sun *et al.*, 2009). Its benefits include weight reduction while maintaining the strength of the alloy. Moreover, the strength of the alloy can be improved by precipitation hardening from heat treatment (George and Scott, 2003).

Aluminum alloy contains elements such as zinc, magnesium, and copper that exceeds the equilibrium of solid solubility. These elements are soluble to solid solution when it is heating up to just below the eutectic temperature for a period in which equilibrium is achieved. This process is generally known as solution heat treatment (SHT). When the elements are solute into the solid solution, it leaves behind the vacant lattice site, called “vacancies”. To retain it, rapid quenching must be used because the material’s temperature has to be dropped rapidly to room temperature. The result will be a supersaturated solid solution at room temperature. This phase is called natural aging, it is non equilibrium at room temperature and will continuously reverse the effect to achieve

equilibrium in room temperature. The soluble elements will precipitate out of the supersaturated solid solution to form the transition and equilibrium phases. Evidence shows that the yield strength of a solution heat treated aluminum alloy 7075 sample has increased from 150 MPa to 465 MPa after 25 years at room temperature (Hatch, 1984).

Natural aging is a very slow process, and therefore, artificial aging was introduced to the aluminum alloy to accelerate the precipitation hardening process. This is done by exposing the material to the temperature range of precipitation heat treatment for a sufficient period to promote growth of precipitates into a specific size or volume. This process is generally called T6, which controls precipitation of fine particles to develop the mechanical properties of the alloy. However, this heat treated material is highly susceptible to stress corrosion cracking (SCC) and corrosion (Zupanc and Grum, 2010).

To prevent SCC, a two-step aging process was introduced to aluminum alloys called T73, which is able to enhance the SCC resistance of the material. The heating process was started with under aging and followed by over aging. However, with this treatment, there is a sacrifice of 10% to 15% of strength due to coarse η phase precipitation growth in the microstructure (Qu and Yang, 2000).

From previous research (Cina, 1974), it has been reported that retrogression and re-aging (RRA) heat treatment is able to improve the resistance to SCC and minimize the loss of strength of the aged alloy. This heat treatment is an intermediate heat treatment that alters the properties of the aged alloy (T6), so that it will maintain the high strength of the T6 temper, but also will gain the SCC resistance of the T73 temper.

The focus of this research is on the retrogression step by using oil and air as the heating medium. Oil retrogression means that the material is retrogressed in hot oil, and, retrogression in air refers to the standard retrogression process in a furnace. In comparison, oil has a better heating rate than air, which can promote faster growth rate for the

precipitation. The resulting samples are compared in mechanical properties, SCC susceptibility, and microstructure alteration.

1.2 Problem Statement

Aluminum alloy 7075 is seldom used for marine applications, due to its susceptibility to general corrosion. It is widely used in aviation applications, due to its high strength over weight ratio. Peak strength of this material is achieved through artificial aging, or so called T6 temper. However, this treatment results in low corrosion resistance. In contrast, T73 is a two-step heat treatment that is introduced to this material to improve the corrosion resistance. However, this treatment reduced the strength of the material by 10% to 15% (Qu and Yang, 2000).

Retrogression and re-aging (RRA), an additional two step heat treatment process for the T6 temper, was first introduced by Cina (1974). This treatment is able to increase corrosion resistance and yet retain the mechanical strength of the alloy. (Xiao *et al.*, 2011), (Reda *et al.*, 2008).

For example, Reda *et al.*, (2008) vary retrogression temperature and time to 160 °C for 250 minutes, 180 °C for 34 minutes, and 200 °C for 8 minutes, with three different pre-aging temperatures (100 °C, 120 °C, and 140 °C). From their research, they found that retrogression of aluminum alloy 7075 at 160 °C for 250 minutes with 120 °C pre-aging temperature exhibited the lowest corrosion rate (0.021 mm/year), and highest strength (672.89 MPa) sample. Other than temperature and time, the heating rate plays an important role in the phase transformation and precipitation growth. Li *et al.*, (2010) claim that a high retrogression heating rate at 190 °C will yield lower mechanical properties, due to the promotion of η phase precipitation and the re-dissolving of a large number of Guinier-Preston (GP) zone and η' phase, which are smaller than critical size. However, their study

does not cover lower retrogression temperature ranges such as 165 °C, or using a different heating medium. In this research, the effect of heating rate will be analyzed by comparing the standard retrogression method to retrogression in oil immersion.

1.3 Objectives

- i. To compare and evaluate the oil retrogression with the conventional retrogression method.
- ii. To evaluate the mechanical properties of aluminum alloy 7075 when treated with different retrogression parameters.
- iii. To investigate the corrosion properties of aluminum alloy 7075 when treated with different retrogression parameters in 3.5 weight % (wt%) sodium chloride solution.

1.4 Scope

This research covers heat treatment process, mechanical testing, corrosion testing, and microstructure evaluation. The sample used is in the round tension shape, in accordance with ASTM E08, which was pre-fabricated by the supplier. No fabrication process was covered in the scope of this research. The heat treatment process includes the SHT, quenching in ice water, artificial aging, under aging, over aging, standard retrogression, oil retrogression, and re-aging. Mechanical testing covers the tensile test and hardness test. Microstructure evaluation is conducted by using an optical microscope and a SEM. Sample preparation is accomplished by using grinding and two-step polishing, followed by an etching process by using Keller's etching reagent. The corrosion test only covers the Tafel DC corrosion test in 3.5 wt% sodium chloride solution by using the Gamry Euro Cell kit.

1.5 Contribution

This dissertation contributes to the area of heat treatment of aluminum alloy 7075. Specifically, it introduces a comparison of oil retrogression to standard retrogression processes to achieve a higher phase transformation through greater heat transfer from the heating medium to the alloy. Oil retrogression results in better performances of mechanical properties, and corrosion resistance at lower retrogression temperature ranges as compared to standard retrogression. A paper was submitted and accepted in the “Journal of Applied Mechanic and Materials, Vol. 165 (2012)” with the title of “Effect of Retrogression Medium to the Mechanical Properties of Aluminum Alloy 7075”.

CHAPTER 2

LITERATURE REVIEW

2.1 Aluminum

Pure aluminum (more than 99.99 wt%) was first obtained in 1920 by using Hoopes electrolytic process (Hatch, 1984). The density of aluminum is only 2.7 g/cm^3 , which is lighter than most other metals such as steel (7.83 g/cm^3), copper (8.93 g/cm^3), or brass (8.53 g/cm^3) (ASM, 1990). Typically, it has excellent corrosion resistance, non-ferromagnetic properties, and electrical and thermal conductivity, which makes it very important in the industrial application (ASM, 1990).

2.2 Aluminum Alloys

Aluminum alloys are composed of aluminum and of one or more other elements such as silicon, zinc, magnesium, beryllium, germanium, copper, and manganese. Different elements can be segregated according to their characteristic properties. For example, zinc and copper are special alloying elements for high strength properties. Zinc, magnesium, silicon and iron are primary alloying elements for strength, ductility, and toughness. Titanium, boron, zirconium, chromium, nickel, bismuth, and lead are additional elements for special properties [(Storen and Oslo, 1994) and (George and Scott, 2003)].

Aluminum alloys are divided into two categories, which are wrought and casting composition, based on the primary mechanism of property development. Each alloy was classified by nomenclatures and divided into families for simplification by the Aluminum Association system, which is the most recognized system in the United State of America

(USA). For wrought alloy, the four digits numerical designation system used is shown in Table 2.1. For casting composition, a three digits numerical designation system followed by a decimal value was used as shown in Table 2.2.

Table 2.1: Wrought aluminum alloy series (ASM, 1990)

Series	Description
1xxx	Controlled unalloyed (pure) compositions
2xxx	Alloys in which copper is the principal alloying element, though other elements, notably magnesium, may be specified
3xxx	Alloys in which manganese is the principal alloying element
4xxx	Alloys in which silicon is the principal alloying element
5xxx	Alloys in which magnesium is the principal alloying element
6xxx	Alloys in which magnesium and silicon are principal alloying elements
7xxx	Alloys in which zinc is the principal alloying element, but other elements such as copper, magnesium, chromium, and zirconium may be specified
8xxx	Alloys including tin and some lithium compositions characterizing miscellaneous compositions
9xxx	Reserved for future use

Table 2.2: Casting aluminum alloy series (ASM, 1990)

Series	Description
1xx.x	Controlled unalloyed (pure) compositions, especially for rotor manufacture
2xx.x	Alloys in which copper is the principal alloying element, but other alloying elements may be specified
3xx.x	Alloys in which silicon is the principal alloying element, but other alloying elements such as copper and magnesium are specified
4xx.x	Alloys in which silicon is the principal alloying element
5xx.x	Alloys in which magnesium is the principal alloying element
6xx.x	Unused
7xx.x	Alloys in which zinc is the principal alloying element, but other alloying elements such as copper
8xx.x	Alloys in which tin is the principal alloying element
9xx.x	Unused

2.2.1 Aluminum-Zinc

The pioneering aluminum alloys to be developed for commercial use were aluminum-zinc binary alloys, despite having been since replaced majorly by aluminum-silicon and aluminum-copper alloys. The primary use of aluminum-zinc alloys is to prevent electrolytic corrosion. There is a possibility that these alloys could have other commercial applications owing to their super plasticity, exhibited near the aluminum-zinc eutectic. Copper and magnesium are used with zinc in production of wrought aluminum product (George and Scott, 2003).

Combining zinc with aluminum at 380 °C results in a eutectic type system. In the eutectic reaction, a liquid with 94.9 wt% zinc reacts forming an aluminum solid solution with 82.8 wt% zinc and a zinc solid solution with 1.1 wt% aluminum (George and Scott, 2003).

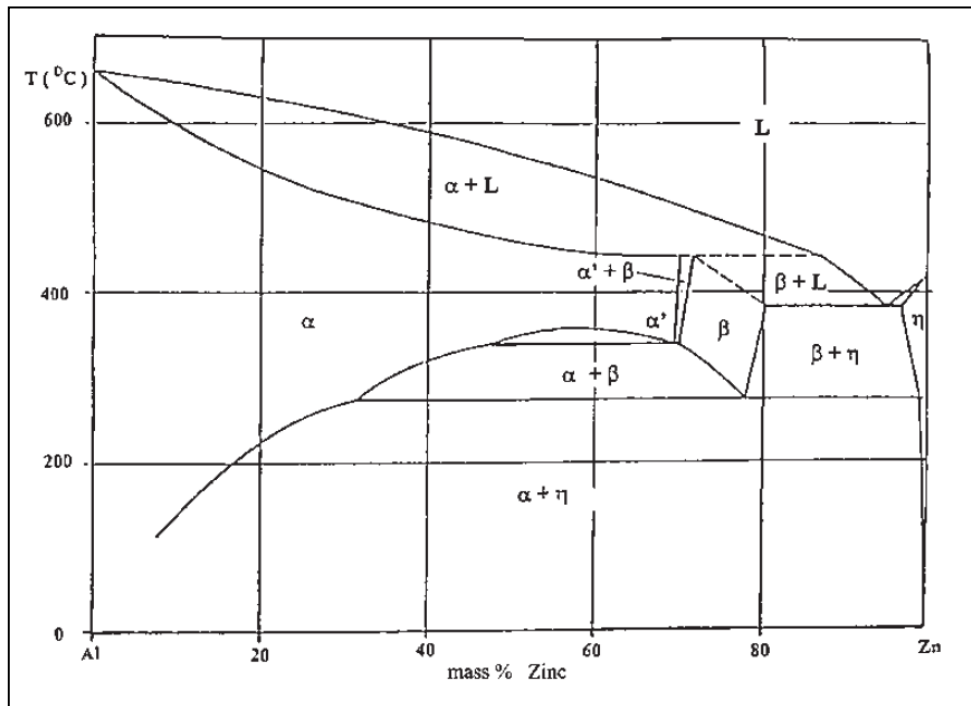


Figure 2.1: Aluminum-zinc phase diagram (Zhu, 2003)