

**RESPONSE OF 7075 AND 7050 ALUMINIUM ALLOYS TO HIGH
TEMPERATURE PRE-PRECIPITATION HEAT TREATMENT**

By

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ABSTRACT

Al-Zn-Mg-Cu (7xxx series) aluminium alloys are widely used for aircraft structures. It is difficult to obtain a combination of optimal strength and stress corrosion cracking (SCC) resistance for these alloys. It appears that SCC resistance of these alloys is related to grain boundary precipitate morphology. One technique to control the grain boundary precipitate morphology is to introduce a controlled cooling procedure referred to as High Temperature Pre-precipitation (HTPP) treatment following the solution heat treatment. There is need for a detailed study of the effect of HTPP on the properties of commercial Al-Zn-Mg-Cu alloys using different intermediate temperatures. In this thesis research, the results of ten HTPP processes applied to 7075 and 7050 commercial 7xxx series alloys are presented in terms of hardness, electrical conductivity, corrosion resistance, TEM analysis of grain boundary precipitate morphology and EDS analysis of solute concentration profile at the grain boundary. Results indicate that subsequent to HTPP processing, the 7050 alloy can be precipitation aged to a higher hardness compared to 7075; this result is associated with the modification of 7050 alloy by zirconium versus chromium in 7075 alloy. HTPP heat treatment achieves better SCC resistance compared to standard T6 temper. However, it does not appear that HTPP can achieve a combination of hardness, electrical conductivity and corrosion resistance superior to standard T6 and T7X tempers.

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CHAPTER ONE

INTRODUCTION

7xxx series aluminium alloys are high strength, heat treatable alloys containing zinc and magnesium as the main alloying elements. Similar to all heat treatable aluminium alloys, 7xxx series alloys rely on precipitation hardening to improve properties [1]. This series of alloys can be divided into Al-Zn-Mg and Al-Zn-Mg-Cu alloys [2]. Al-Zn-Mg alloys are relatively weldable and are referred to as medium strength alloys [3]. Al-Zn-Mg-Cu alloys have the highest strength compared to other aluminium alloys. 7xxx series alloys are often used for high strength applications, predominantly in compressively loaded airframe structures, as shown in Figure 1.1 [4]. Examples are upper wing panels, frames, stringers, longerons, extruded parts, etc. Other applications include mobile equipment and other highly stressed components. Properties of particular interest for structural applications of 7xxx series alloys are toughness, fatigue crack growth rate, strength, exfoliation and stress corrosion resistance [3, 5].

1.1 Historical development of Al-Zn-Mg-Cu alloys for airframe structures

Airframe structures are high strength applications. For instance, upper wing structures are loaded in compression during flight. Hence, materials selection for these parts requires a high compression strength to weight ratio. Other important properties considered for materials selection are cost, corrosion resistance and toughness [6].

Application of aluminium alloys in aircraft can be traced back to the engine crankcase in Wright brothers' plane in 1903 [7]. A study of the Wright Flyers internal combustion engine crank case showed a composition of Al-8% copper, about 1.0% iron and 0.4% silicon impurities.

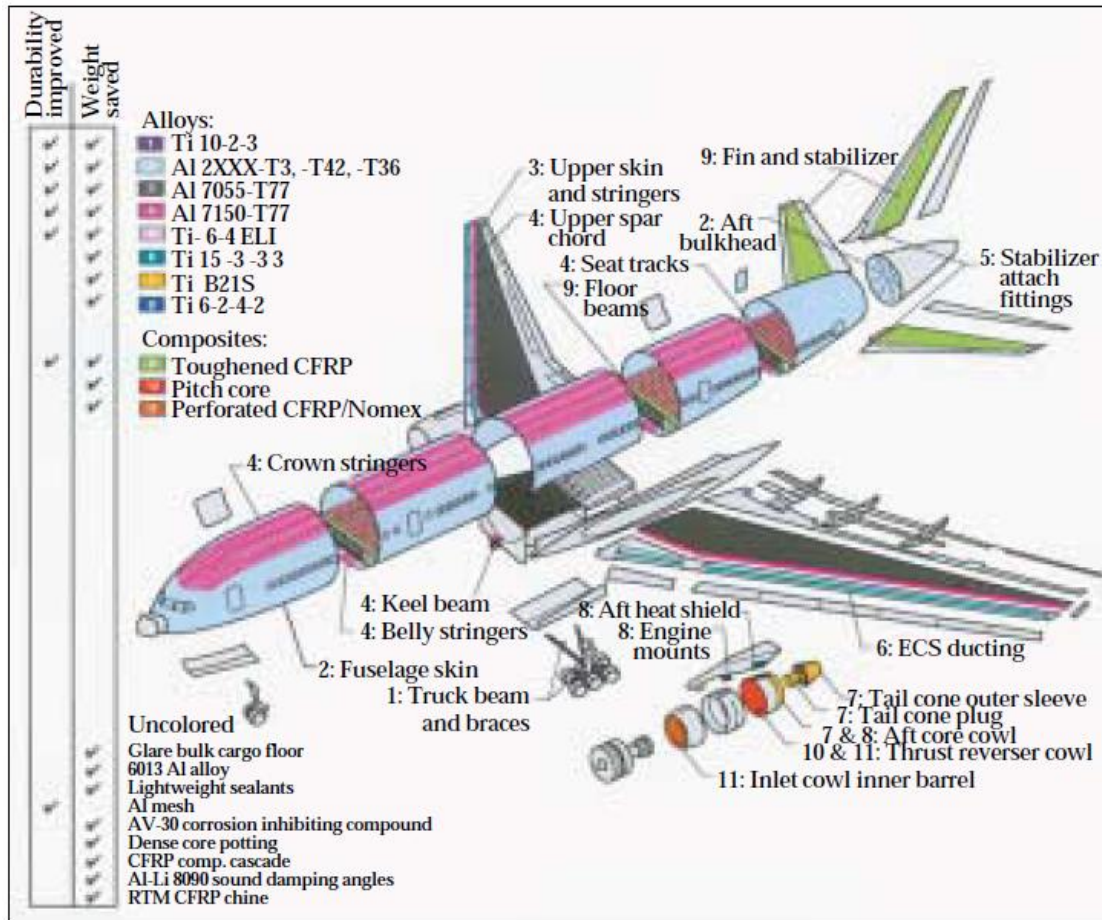


Figure 1.1: Typical applications of aluminium alloys and tempers in aircraft structures, such as the Boeing 777 illustrated [4]

The strength of the alloy, low weight requirements of the aircraft and good casting qualities are likely reasons for selection of this material [8].

The discovery of age hardening of Al-Cu alloys by Alfred Wilm in 1906 led to research on the mechanism of precipitation hardening [9]. Guinier and Preston's work provided evidence that explains the precipitation mechanism in aged Al-Cu alloy [9]. Further studies revealed that GP (Guinier-Preston) zones are formed during nucleation of strengthening precipitate phases from the solid solution. These studies started the evolution of alloy development for airframe structures [7, 9].

Earliest application of aluminium alloys in aircraft wing structures used alloys based on the Al-Cu-Mg system (2xxx series alloys), an example being the Junkers F-13 and DC-3 aircraft. These alloys had the highest yield strength at that time in the range of 275-310 MPa. The need for increased aircraft performance motivated effort to find other precipitation hardenable alloys [9]. Among the few other alloys that showed precipitation hardening, Al-Zn-Mg alloys were discovered in 1923. Investigation showed that Al-Zn-Mg alloys showed the best response to precipitation hardening compared to other aluminium alloy compositions. Figure 1.2 shows various materials used for upper wing skin of aircrafts from 1919-1994 [9]. This shows that continuous increase in demand for materials with higher strength to weight ratios has enabled the development of precipitation hardenable Al-Cu (2xxx series) and Al-Zn-Mg-Cu (7xxx series) alloys over the years [5]. The broken line in Figure 1.2 shows the transition in aircraft application in early 1940's from 2xxx to 7xxx alloys based on higher strength.

In the 1920's and 30's, studies were carried out to understand tensile strength and corrosion resistant properties of Al-Zn-Mg alloys. Part of this was the development of alloy X74S (5.2% Zn, 2.1% Mg, 1.5% Cu, 0.4% Mn) with a low Zn:Mg ratio in 1938. 7076-T6 alloy (7.5% Zn, 1.6% Mg, 0.7% Cu and 0.6% Mn) with a high Zn:Mg ratio was developed in 1940 for application as a high strength forging alloy for propeller blades. However, deformed sheet of this alloy was susceptible to stress corrosion cracking. Studies showed that addition of 0.2-0.35% Cr increased the corrosion resistance of X74S [10].

In 1943, 7075-T6 was developed based on the previous alloy compositions by adjusting the composition of Zn, Mg and Cu for improved strength and the addition of Cr. First application of 7075-T6 sheet was as skin and stringers in B-29 Fortress

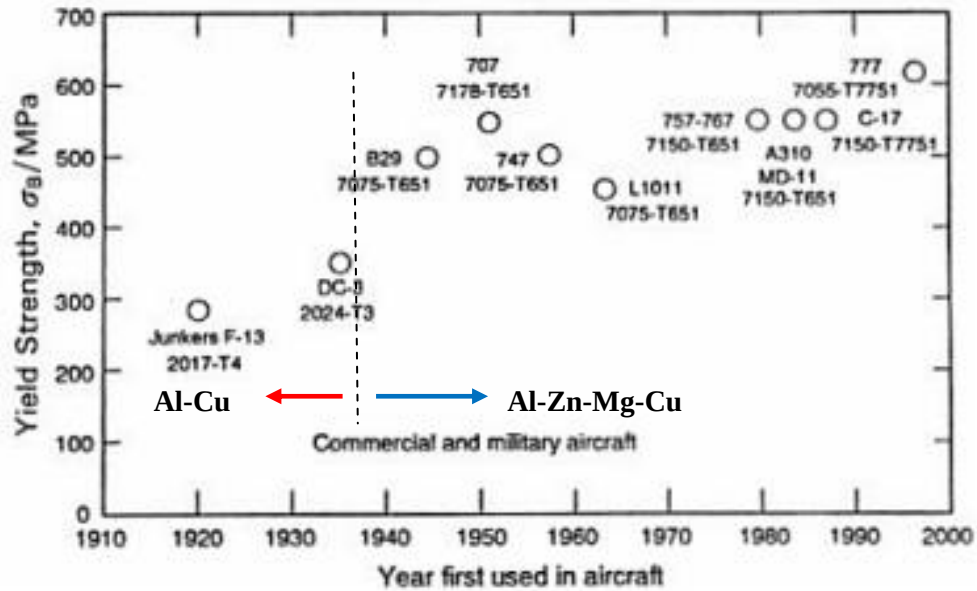


Figure 1.2: Materials used for upper wing skin from 1919-1994 [9]

bomber during World War II. Higher strength 7xxx alloy were developed for passenger and military aircraft after the war [10].

In the 1950's, forgings were used for several aircraft structures. The susceptibility of 7075-T6 to corrosion and high quench sensitivity led to development of another alloy more suitable for forgings. Hy43 with low copper content (about 0.6% Cu) was developed as a forging alloy and designed to have good heat treatment properties. Hy43 was better in terms of ductility in short-transverse section, quench sensitivity and strength. After successful accelerated stress corrosion time to failure testing was carried out, Hy43 was commercialized as alloy 7179 and used for a wide range of old and new designs. However, in the late 1950's, it was discovered that alloy 7179 exhibited high susceptibility to stress corrosion that was not predicted by the accelerated corrosion tests. This is due to the slow crack initiation in 7179, but higher crack growth rate compared to 7075 alloy [10]. The severe corrosion problems of 7179 alloy led to its withdrawal from aerospace applications [11]. Nevertheless,

airworthiness issues related to the high stress corrosion cracking susceptibility of 7179/7079 alloy continue today [12]. Tables 1.1 and 1.2 show chemical composition and properties of some of the alloys mentioned.

In the 1960's, the problem of stress corrosion cracking and requirement for thicker sections of 7075-T6 for forgings in aircraft designs led to development of 7075-T73 temper. 7075-T73 forgings were developed for improved corrosion resistance using a two-step aging heat treatment procedure. After aging to T6 temper at about 120 °C for 24 hours, T7 temper was obtained by overaging above 150 °C for 7-9 hours to increase resistance to stress corrosion cracking [1].

Although, this comes with about 10-15% loss of strength compared to 7075 and 7179-T6, 7075-T73 was accepted by aircraft designers [10]. One of the first application of 7075-T73 was in DC10. T76 temper was later applied in Lockheed L1011 [7, 11]. The increasing demand for lighter weight, higher strength structures especially for commercial aircraft led to the development of 7178-T651 alloy with higher Zn, Mg and Cu content compared to 7075. However, the increasing demand for higher fracture toughness and damage tolerance limited the use of this alloy [10]. Over the years, applications of T73 alloys have no record of stress corrosion cracking [10].

In the T6 and T73 tempers, there is an inverse relationship between strength and SCC resistance as shown in Figure 1.3 [13]. This shows that as aging time and temperature increases, the strength decreases while resistance to SCC increases. In Figure 1.3, stage I shows the initial aging step. Stage II can be related to T6 temper and it shows that higher strength can be obtained but the resistance to SCC is low. Stage III shows that prolonged aging can increase SCC resistance but there is loss of strength.

Table 1.1: Composition (wt.%) of some aluminium alloys used in aircrafts [14, 15]

Alloy	Zn	Cu	Mg	Mn	Cr	Zr	Fe	Si
2014	-	4.4	0.5	0.80	0.10	-	0.70	0.80
2024	-	4.4	1.5	0.60	0.10	-	0.50	0.50
7075	5.6	1.6	2.5	-	0.30	-	0.50	0.40
7178	6.8	2.0	2.7	-	0.30	-	0.50	0.40
7050	6.3	2.4	2.3	0.10	0.04	0.12	0.15	0.12
7079	4.3	0.6	3.3	0.20	0.20	-	0.50	0.30
7150	5.9-6.9	1.9-2.5	2.0-2.7	0.10	0.04	0.08-0.15	0.15	0.12
7055	7.6-8.4	2.0-2.6	1.8-2.3	0.05	0.04	0.08-0.25	0.15	0.10
7A52	4.4	0.13	0.13	0.24	0.13	0.12	0.25	0.20

Table 1.2: Typical mechanical properties of aluminium alloys [13, 15, 16, 17, 18, 19, 20, 21, 22]

Alloy	Year of introduction	Temper	Yield strength (MPa)	Fracture toughness (MPa√m)	SCC resistance
2024	1931	T351	269-276	Excellent	Good
		T851	386-400	Excellent	High
		T6	345	Good	
2017	1920's	T4	276		
7075	1943	T6	503		Moderate
		T73	434		Excellent
7076	1940				
7175		T6	503		
		T73	476		
7178	1951	T6	537		
		T76	503		
7179		T6			
7050		T74	510		
		T76	455		
7150		T7751	565	29.7	
7055		T7751	614	28.6	
		T651	544		
		T7451	496		
7079	1954	T6	469		

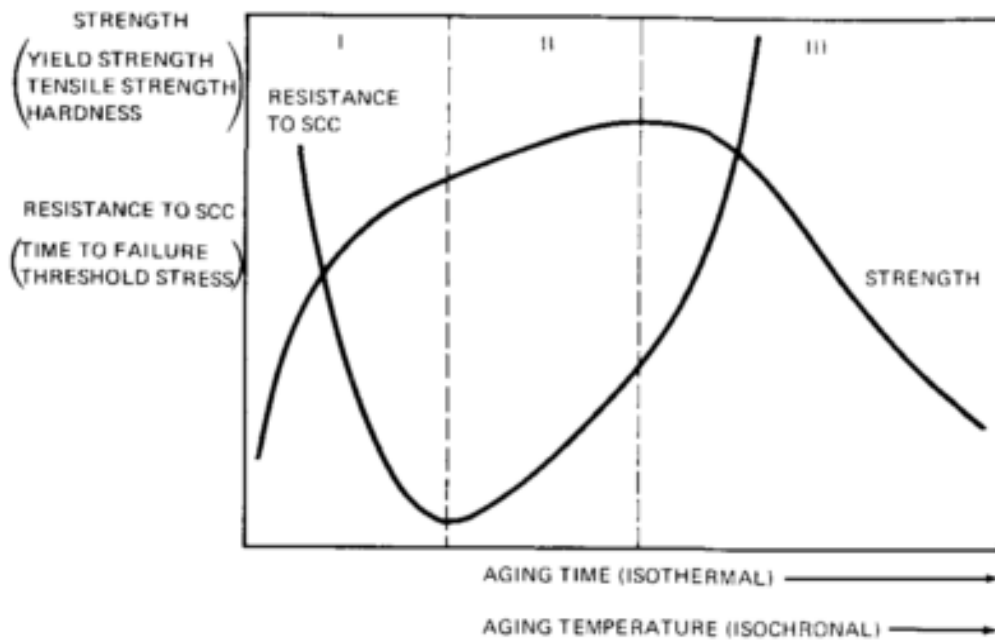


Figure 1.3: Variation of SCC resistance and strength due to aging [13]

The loss of strength in 7075-T73 temper led to compositional modification to obtain 7050 alloy with about 2.22% copper and use of Zr in place of Cr for improved strength and less quench sensitivity. Chromium containing dispersoids in 7075 alloy control grain structure and prevent recrystallization during processing but causes quench sensitivity, i.e. properties are dependent on the quenching rate. Hence, the strength of 7075 alloy decreases as the quenching rate decreases. The Zr addition in 7050 alloy reduces the quench sensitivity. Thus, 7050 alloy is more useful for thick structural sections [23].

In the 1970's, Cina developed the Retrogression and Reaging (RRA) heat treatment to address the trade-off between strength and stress corrosion resistance in the T6 and T73 tempers. The RRA heat treatment is applied to 7075-T6 and involves 'retrogression' (partial re-solutionizing) of the T6 temper at a higher temperature for a

very short period of time (for example 200 °C for 2 minutes, 240 °C for 7 seconds). During retrogression, dissolution of strengthening precipitate phase and dislocations occur, causing loss of strength as shown in Figure 1.4 [24]. This is followed by reaging using T6 aging conditions (121 °C for 24 hours) to achieve a better combination of strength and SCC resistance [14, 25]. Figure 1.4 shows the effect of retrogression time on hardness after retrogression and subsequent reaging heat treatment. It was observed that the strength reaches the lowest minimum after a short retrogression time. Increase in retrogression time causes increase in hardness but further increase in retrogression time causes the hardness to decrease. Subsequent reaging treatment increases the hardness. However, higher strength after reaging is obtained at the lowest minimum on the retrogression curve corresponding to few seconds of retrogression.

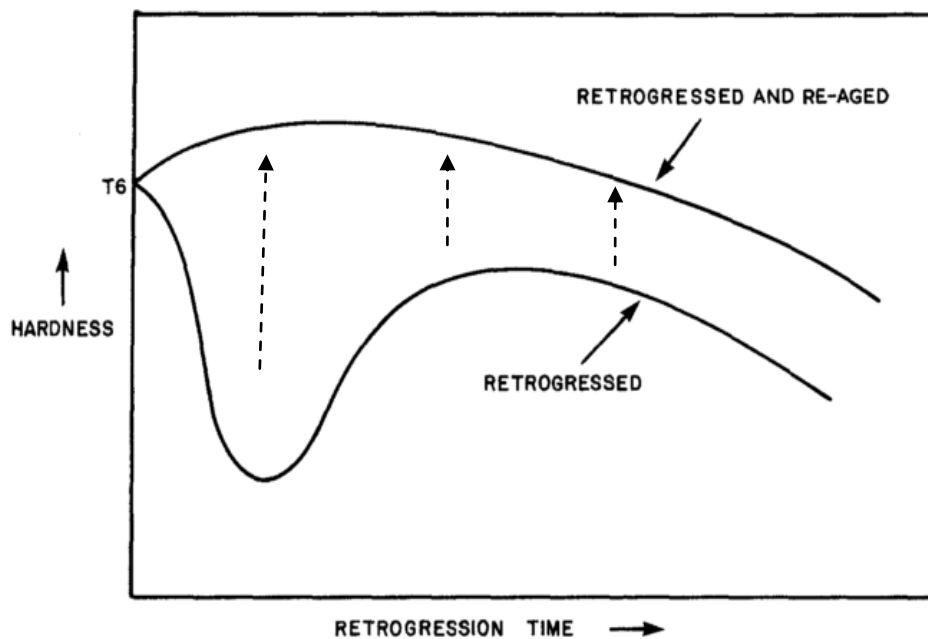


Figure 1.4: Changes in hardness during RRA heat treatment [24]. Arrows indicate change in hardness after reaging

A T77 temper designation is used when RRA is applied to 7055 alloy. 7055-T77 is used for several structural components on the Boeing 777, as shown in Figure 1.1. However, the short retrogression time required to obtain high strength in RRA treatment makes it difficult or impossible to apply RRA to thick section components [24].

1.2 Motivation and Objectives of this Thesis Research

The difficulty of applying RRA to thick sections of components during manufacturing creates the need to develop a heat treatment procedure that avoids the relatively high temperature retrogression process.

This research aims to study the effect of high temperature pre-precipitation (HTPP) heat treatment on strength and SCC resistance of 7xxx series alloys. HTPP is a recently developed heat treatment procedure which involves slow cooling from solution heat treatment temperatures to a lower, intermediate temperature before quenching, followed by applying T6 aging. The aim of this procedure is to modify the grain boundary microstructure to form coarse, discontinuously distributed precipitates and achieve higher resistance to stress corrosion cracking [26]. Similar slow cooling heat treatment procedures in other materials such as nickel superalloys or titanium aluminide intermetallics showed that this procedure can modify the grain boundary microstructure and improve targeted material properties [27]. Previous work on the effect of HTPP on experimental Al-Zn-Mg-Cu alloys shows that HTPP treatment can improve SCC resistance in experimental alloys [26, 28, 29]. However, the influence of HTPP heat treatments applied to commercial 7xxx alloys is not well understood. It is also important to understand the effect of the intermediate temperature used during the HTPP process on the properties of Al-Zn-Mg-Cu alloys. Accordingly, the goal of

this thesis research is to study the effect of HTPP heat treatments on the electrical conductivity, quench sensitivity, hardness, corrosion resistance and microstructure of commercial 7075 and 7050 alloys.

CHAPTER TWO

Al-Zn-Mg-Cu ALLOYS

2.1 Alloying elements

Pure aluminium is well known for its low density and high corrosion resistance. However, pure aluminum cannot be used for structural applications due to low strength. Alloy development involves the addition of different alloying elements to improve material properties. Alloying aluminium to high strength enables design applications where high strength to weight ratio is important [2]. Although the primary effect of alloying is increase in strength, other material properties are affected by alloying. Aluminium alloys usually contain 90-96% aluminium and about two major alloying elements to obtain a combination of properties. Major alloying elements usually have significant solubility in aluminium. Zinc, magnesium, manganese, copper and silicon are used as major alloying elements. Although, minor elements may have low solubility in aluminium, addition of minor elements improves fabrication and performance [30, 31].

Aluminium is a reactive metal but it relies on an ability to form a protective oxide layer for good corrosion resistance. Generally, corrosion resistance decreases as the alloying element content increases. Alloying elements may form solid solutions or phases that sometimes affect the continuity of the protective oxide layer or electrochemical potential of phases versus matrix [32]. Specific alloying elements in 7xxx series aluminium alloy are as follows:

- **Zinc and Magnesium**

The addition of zinc increases the solution potential of the alloy. However, binary aluminium-zinc alloys are seldom used due to high susceptibility to SCC and hot cracking in the wrought and cast alloys respectively. Magnesium and zinc are often added together to increase strength by increasing the heat treatment response of the alloy due to formation of $MgZn_2$ precipitate phase. However, there is need for control of composition, microstructure and processing (heat treatment) to obtain resistance to SCC [2, 33].

- **Copper**

The addition of up to 1% copper in the presence of zinc and magnesium increases the strength by solid solution strengthening. Increase in copper content to about 1.5% increases resistance to SCC. However, high amount of copper increases quench sensitivity and reduces weldability [2].

- **Chromium/Manganese /Zirconium**

Chromium, manganese and zirconium are used as additions in 7xxx alloys to control grain structure and prevent recrystallization. Chromium can form finely dispersed phases in these alloys due to its slow rate of diffusion [2]. However, the use of chromium is challenged by increase in quench sensitivity. Zr is being used in newly developed alloys to reduce quench sensitivity. This will be a subject of discussion later in this chapter.

- **Iron and silicon**

Iron and silicon are the commonest impurities found in aluminium alloys. Iron has a high solubility in aluminium in the molten state, but the solubility reduces in solid state. This enables iron to form intermetallic compounds with aluminium and other elements [2]. Silicon reduces the melting point, increases the strength and fluidity of

aluminium [1]. Both iron and silicon significantly affect properties of aluminium alloy. Hence, strict limits on the quantity of impurity elements are important to achieve required material properties. 7050 alloy contains lesser amount of iron and silicon compared to 7075. This compositional modification reduces the amount of constituent particles formed in 7050 alloy thereby improving the fracture toughness. Constituent particles are intermetallic compounds containing Fe and Si formed during solidification. More information on the types and effect of constituent particles will be discussed later in this chapter. The effect of this compositional modification on the microstructure of 7050 and 7075 alloys will be studied later in this thesis.

2.2 7xxx versus 2xxx series aluminium alloys for aircraft structures

Both 2xxx and 7xxx series are heat treatable alloys widely applied as airframe structures. 2xxx series (Al-Cu) alloys have been widely applied since the discovery of age hardening by Alfred Wilm which led to development of Duralumin (Al-3.5%Cu-0.5%Mg-0.5%Mn) [7, 9]. Modifications in chemical composition led to development of 2014 alloy with higher silicon content for higher strength and 2024 alloy with higher magnesium and lower silicon content for better response to aging at room temperature. Typical tensile strength of 2014 alloy is 485 MPa. 7xxx alloys have higher strength in the range of 470-600 MPa [31]. This explains the replacement of 2xxx with 7xxx alloys for upper wing structures shown in Figure 1.2.

A critical property required for upper wings is high compressive strength. This is necessary since the upper wing is under compression loading during flight [6]. 7xxx alloys have higher compressive strength compared to 2xxx alloys. However, Figure 1.1 shows that 2xxx series alloys are still applied as structural materials in aircrafts

especially for high fracture toughness applications. An example is the lower wing which is under tension loading during flight.

An important property that differentiates 2xxx and 7xxx alloys is fracture toughness. Recently, reduction in the level of iron, silicon and copper has achieved an increase in fracture toughness of 2xxx alloys. Also, high damage tolerance and high resistance to fatigue crack propagation enables 2xxx alloys to be applied as aircraft fuselage skin and lower wing skins on commercial aircrafts. Figure 2.1 shows the fracture toughness-strength requirement of both alloys [34]. At lower strength compared to 7xxx series alloys, higher fracture toughness can be obtained in 2xxx series alloys [34].

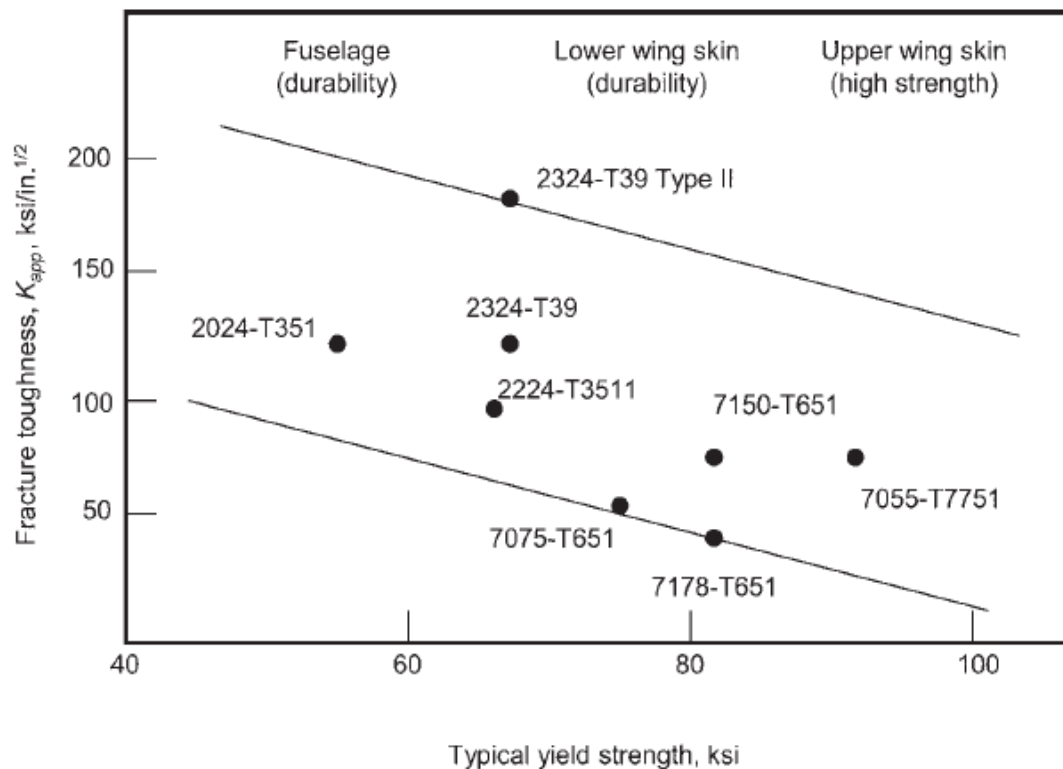


Figure 2.1: Fracture toughness versus yield strength relationship for 2xxx and 7xxx series alloys [34]

2.3 Precipitation hardening in 7xxx alloys

Precipitation hardening is the process of developing fine, second phase particles called precipitates in an alloy such that higher strength is achieved. Figure 2.2 shows the phase diagram of precipitation hardenable alloys [35]. For precipitation hardening to occur, it is important that the solid solubility limit of the solute in the alloy system is decreasing as temperature decreases as shown by the broken line in Figure 2.2.

2xxx, 6xxx and 7xxx series alloys (with Cu, Mg-Si and Zn-Mg as main alloying element respectively) are precipitation hardenable and are also known as heat treatable alloys [33]. To achieve precipitation hardening in 7xxx alloys, the following heat treatment steps (shown in Figure 2.2) must be carried out.

- Solution heat treatment
- Quenching
- Aging

Solution heat treatment (SHT) is the process of dissolving second phase particles in an alloy to form a single phase. SHT involves heating to a temperature in the single phase region as shown in Figure 2.2. Figure 2.3 summarizes the heat treatment procedure for precipitation hardening in 7xxx alloys. The alloy is kept at the SHT temperature (T_o) for some time to develop a single phase at equilibrium. Thermal processes occurring during SHT also allows formation of vacancies in the single phase. The choice of SHT temperature is determined by the alloy composition. High SHT temperature is used to achieve high strength after aging. However, in alloys containing eutectic phases, SHT temperature higher than eutectic temperature can cause incipient melting of eutectic phases.

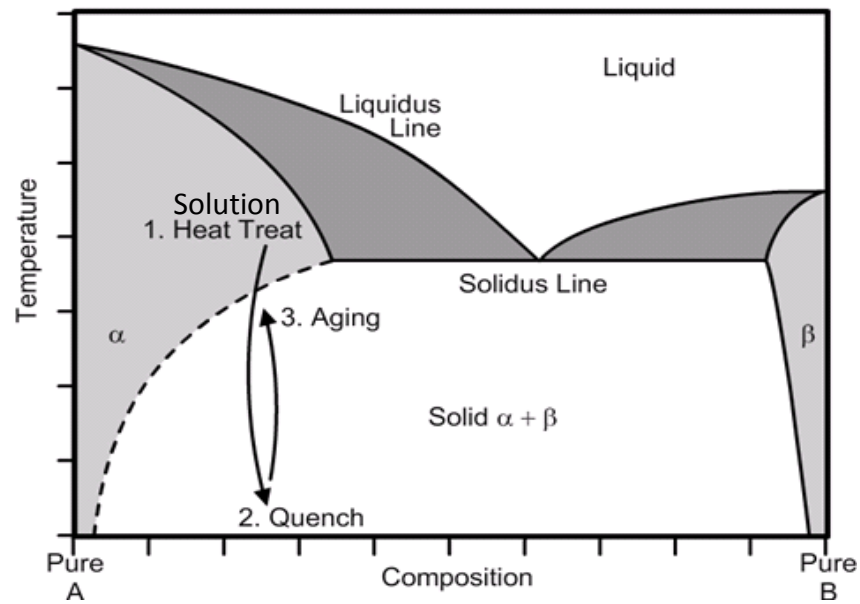


Figure 2.2: Phase diagram of a typical precipitation hardenable alloy showing heat treatment processes for precipitation hardening. Broken line indicates decrease in solubility of solute as temperature decreases [35]

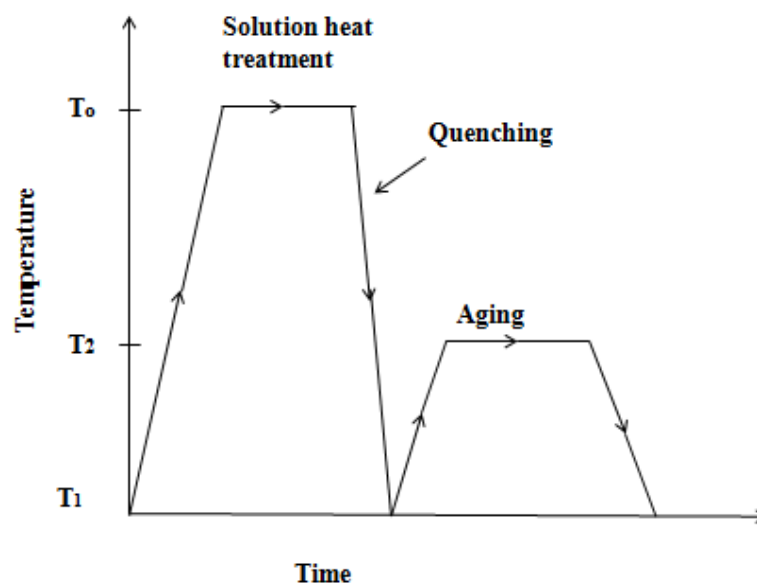


Figure 2.3: Heat treatment process for precipitation hardening in 7xxx alloys

This will have a negative effect on the final mechanical properties of the alloy. Depending on the alloy composition, potential improvement in the properties of aluminium alloy can be obtained by appropriate design of SHT process. For instance, non-isothermal SHT between the solvus temperature and the eutectic temperature starting from low SHT temperature allows homogenization of the eutectic phase to occur and subsequent increase in the temperature will maximize the solute concentration [27].

Quenching is the process of rapidly cooling the alloy from the SHT (T_0 to T_1 as shown in Figure 2.3) to form a supersaturated solid solution (SSS) of solute and highly concentrated vacancies. SSS is important for the subsequent nucleation and growth of precipitates during aging. Optimum strength is often associated with fast quenching rate [33]. However, fast quenching rate may induce stress and cause distortion in intricate parts. Slow quenching allows migration of solute and vacancies to grain boundary regions which may cause a reduction in mechanical properties after aging. Hence, the choice of quenching medium and temperature are important to determine the quenching rate. Water is a common medium for quenching aluminium alloys. It is important to avoid quench delays to achieve optimum strength properties [1].

Aging is the controlled decomposition of the SSS to form second phase particles called precipitates. The supersaturated solid solution formed during quenching is unstable and this provides a driving force for precipitation to occur through diffusion [27, 33]. Aging can occur naturally at room temperature or artificially at temperature below the equilibrium solvus (T_2 in Figure 2.3). During aging, precipitates are formed with an interface with the matrix which may be coherent (perfect match at the interface between precipitate and the matrix), semi-coherent or incoherent. Fine precipitates formed at the initial stage of precipitation hardening are coherent or semi-

coherent with the matrix. However, prolonged aging causes loss of coherency of the precipitates with the matrix. This is called overaging and it is usually associated with loss of strength.

2.4 Introduction to physical metallurgy of 7xxx alloys

7xxx series alloys have good solid solution range that allows formation of supersaturated solid solution during quenching from SHT temperature [1]. 7xxx series alloys are highly unstable after quenching from SHT such that natural aging occurs even at room temperature. Artificial aging (aging above room temperature) is commonly used in practical applications. During aging, precipitate phases are formed due to precipitation reaction. In 7xxx alloys, the mechanism of homogeneous precipitation can be summarized as follows:

Supersaturated Solid Solution (SSS) $\rightarrow$ Guinier-Preston (GP) zones $\rightarrow$ non-equilibrium (metastable) phase (η') $\rightarrow$ equilibrium precipitate (η) [26].

An increase in the concentration of vacancies in the supersaturated solid solution enhances homogenous precipitation by formation of vacancy clusters and diffusion of solute to vacancy clusters to form GP zones. As aging continues, diffusion of solutes facilitates the growth of GP zones to intermediate precipitates. These are spherical, metastable (η') phases. Increase in internal strain induced by the intermediate precipitates causes partial loss of coherency with the matrix. As aging continues, equilibrium precipitates, η or T phase that are incoherent with the matrix are formed [1].

The general precipitation mechanism of 7xxx alloys showing decomposition of GP zones to intermediate (η') phase and later equilibrium precipitate phase (η) is given

above. Precipitation sequence that follows such decomposition from GP zones to η is common in alloys with high Zn:Mg ratio like 7075 and 7050 alloys. The composition of intermediate precipitate (η') is not so clear. Some studies claim that the composition of intermediate precipitate is same as equilibrium precipitate, other researchers have shown that the composition of intermediate precipitate is closer to GP zone with Zn:Mg ratio 1:1 [1, 36, 37]. Equilibrium precipitate is hexagonal, MgZn_2 with Zn:Mg ratio 2:1. In alloys with low Zn:Mg ratio, the precipitation mechanism involves formation of GP zones and decomposition to intermediate T' and later equilibrium T phase likely composed of cubic, $\text{Mg}_{32}(\text{Al}, \text{Zn})_{49}$ [1, 37]. In 7075 and 7050 alloys, optimum strength is obtained from GP zones and intermediate precipitates. However, continuous aging causes precipitates to grow to equilibrium precipitates and this causes loss of strength.

Precipitation hardening also modifies the grain boundary precipitate structures. Since 7075 and 7050 alloys are susceptible to intergranular corrosion like SCC, heat treatments are designed to develop precipitate structure that will prevent a continuous path for corrosion at the grain boundary. As discussed earlier, a coarse, discontinuous distribution of precipitates has been related to higher corrosion resistance of T7 and T77 (RRA) tempers. The effect of HTPP on the grain boundary precipitate morphology and corrosion resistance will be analyzed later in this study.

Figure 2.4 shows the simplified Al-MgZn₂ equilibrium diagram for 7xxx series alloys [27]. The composition of Zn and Mg content in 7075 alloy is represented by the broken line. The presence of copper and impurities in commercial 7075 alloys changes the composition of the equilibrium phase and this will alter the temperatures given in Figure 2.4. For instance, the given solvus temperature is 420 °C for 7075, but this might be in the range of 420-455 °C in commercial alloys [33].

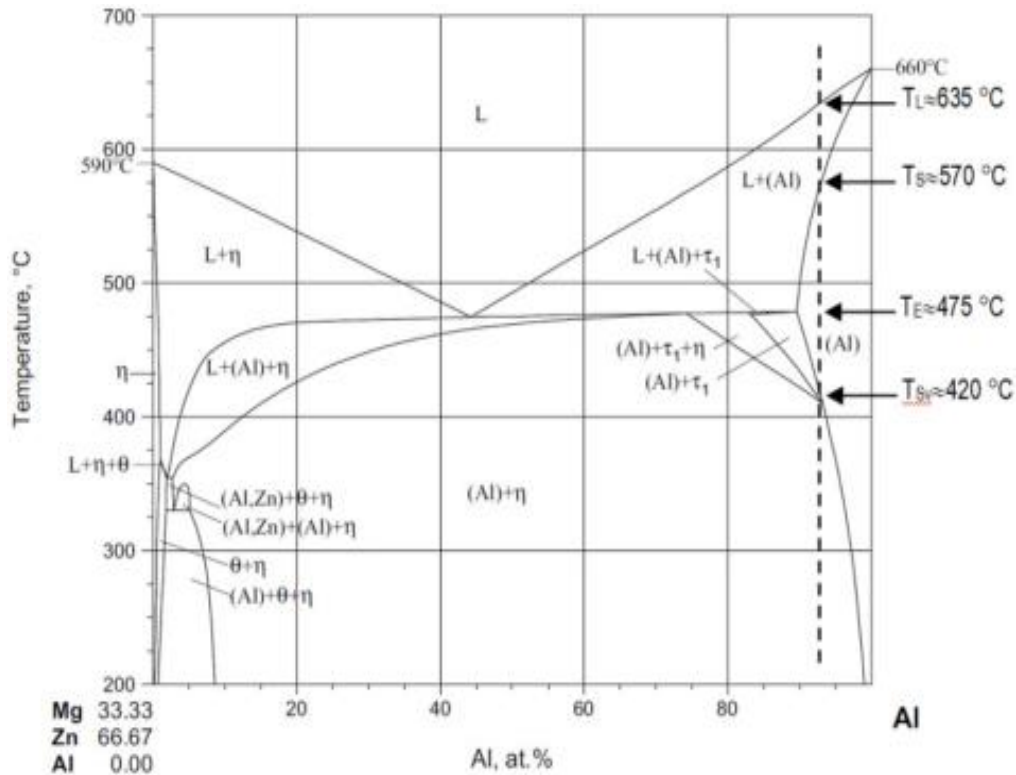


Figure 2.4: The Al-MgZn₂ equilibrium diagram for Al-Zn-Mg alloys. Zn and Mg content in 7075 alloy corresponds to broken line [27]

2.5 Mechanism of precipitation hardening in 7xxx alloys

In 7xxx alloys, strength is increased due to the interaction between dislocation and second phase particles formed during precipitation. Dislocation-precipitate interaction is determined by size, spacing and degree of coherency between the precipitate and the matrix. Depending on the alloy, strength is determined by the ability to resist dislocation motion through the following [38, 39]:

Strain fields formed by coherent GP zones: The presence of misfit between the precipitate and the matrix generates strain fields. This increases the internal stress and higher stress will be required for dislocation motion thereby increasing strength.

Shearing of deformable precipitates: Interaction between small precipitates and dislocations is often by shearing. This occurs due to deformability of small precipitates that allows dislocations to cut through the precipitates. The stress required to initiate dislocation motion is called critically resolved shear stress (CRSS). At the initial stage of precipitate growth, the CRSS is low. This enables dislocations to shear through precipitates. This form of interaction is also known as chemical hardening. During chemical hardening, changes occur along the slip plane due to formation of an additional interface. Strength is increased in that more work will be required by the applied stress to achieve these changes.

Bypass of coarse, non-deformable precipitates: Dislocations bypass coarse particles due to increase in the particle spacing and loss of coherency at the precipitate-matrix interface. As the precipitates grow, resistance to shearing from dislocations increases. Strengthening is obtained due to work hardening by the debris dislocation left after looping of particles by dislocation. Figure 2.5 shows that particle size affects the CRSS in deformable and non-deformable particles as precipitate particles grow [1]. As shown in the Figure, the critical particle size corresponds to high critically resolved shear stress. At the critical particle size, high strength can be obtained due to difficulty in achieving the high CRSS.

2.6 Important microstructural features

Some microstructural features affect properties of 7xxx series alloys. Dispersoids, intermetallic compounds, constituent particles, grain boundary and matrix precipitates among others are important features that affect fracture toughness, strength and corrosion resistance of 7xxx alloys.

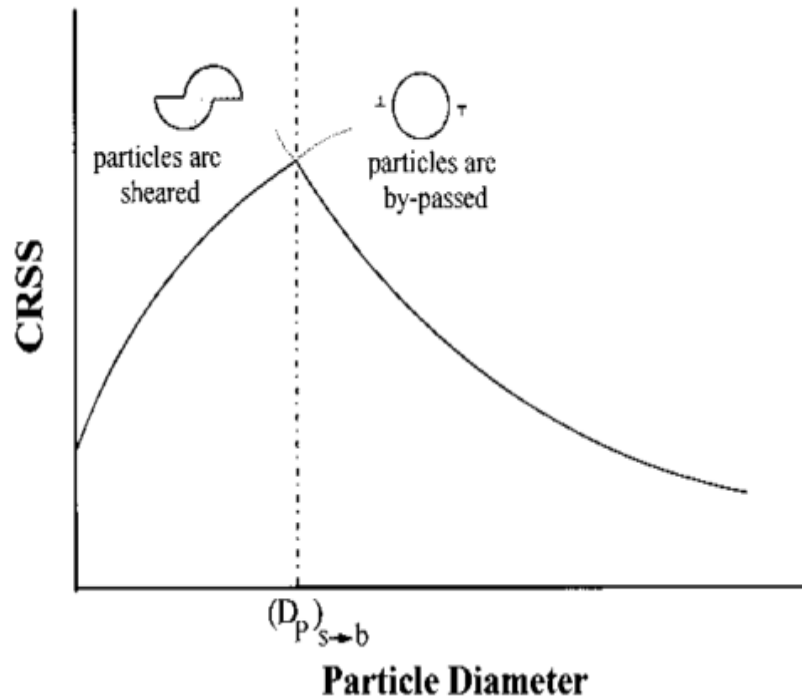


Figure 2.5: Effect of particle size and CRSS on dislocation-precipitate interaction [1]

- **Matrix and grain boundary precipitate**

The composition, structure and distribution of precipitates in the matrix and grain boundary region affect the properties of 7xxx alloys. Among other microstructural factors, matrix precipitates affect strength while the grain boundary precipitates affect resistance to intergranular corrosion [29, 40]. During aging, coherent precipitates are finely distributed in the matrix to achieve high strength by restricting movement of dislocations. Continuous aging to the overaged condition increases precipitate size in the matrix and grain boundary region, causing loss of strength and coherency. Figure 2.6 shows the fine and coarse precipitate morphology in T6 and T7 matrix respectively. While high concentration of fine precipitates observed in T6 increases the strength, coarsening of the precipitates in T7 temper causes loss of strength [29].

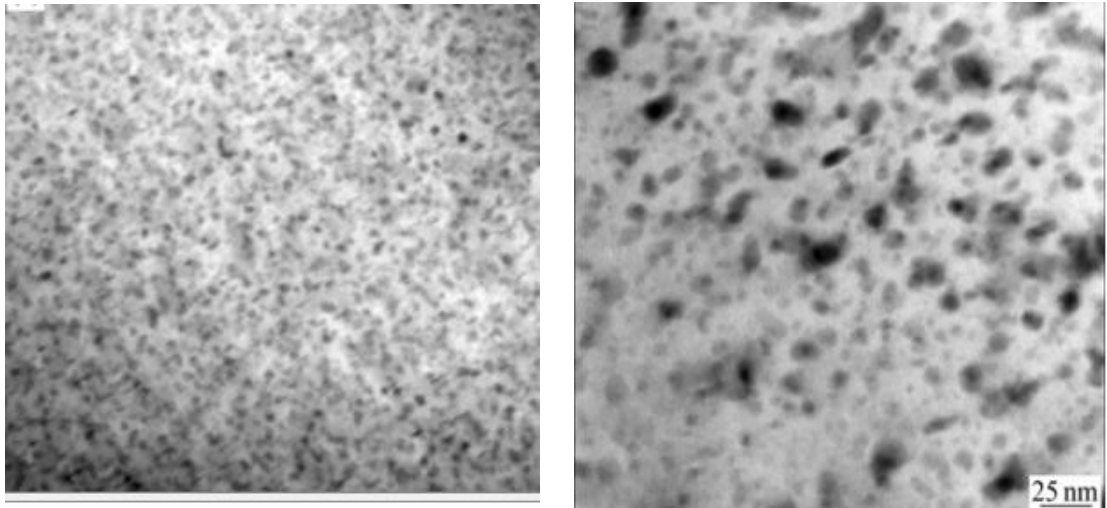


Figure 2.6: Distribution of precipitates within the matrix in (a) T6 and (b) T7 temper respectively [29]

Figure 2.7 shows grain boundary precipitate morphology in T6 and T7 temper respectively. A continuous distribution of small precipitates is observed in T6 while T7 temper shows discontinuous distribution of coarse precipitates. The coarsening of precipitates in the grain boundary during overaging improves the resistance to intergranular corrosion by eliminating a continuous path for corrosion to occur [40]. Studies of other heat treatment procedure that have improved the resistance to SCC like retrogression and reaging shows that coarse precipitates are discontinuously distributed along the grain boundary (similar to T7 temper shown in Figure 2.7) [29].

- **Constituent particles**

Constituent particles are intermetallic compounds formed during solidification through eutectic reactions. Constituent particles may contain Fe or Si with other alloying elements and sometimes aluminium. Constituent particles containing Fe are insoluble while those without Fe may be soluble or slightly soluble based on the solute content in the solid solution [1]. Constituent particles larger than 1 μm in

diameter are undesirable in aluminium alloys. Due to incoherency with the matrix, constituent particles are not effective strengthening particles. Constituent particles can favour crack growth, reduce fatigue resistance and fracture toughness. In 7075 alloy, $\text{Al}_7\text{Cu}_2\text{Fe}$, SiO_2 , $\text{Al}_{12}(\text{Fe}, \text{Mn})_3\text{Si}$, $\text{Al}_{23}\text{CuFe}_4$, $\text{Al}_6(\text{Fe}, \text{Mn})$, Mg_2Si , are the common constituent particles. The compositional modification through the use of lower amounts of iron and silicon in 7050 alloy achieves decrease in the size and number of constituent particles in 7050 alloy. $\text{Al}_7\text{Cu}_2\text{Fe}$, Mg_2Si and Al_2CuMg are common in 7050 alloy [1].

- **Dispersoids**

Dispersoids are small particles that are formed by aluminium and transition metal alloying elements such as Zr, Cr and Mn during hot rolling. Due to small size and fine distribution, dispersoids are useful in preventing recrystallization by pinning the movement of grains. The ability of dispersoids to effectively prevent recrystallization requires small size, small spacing and coherent or semi-coherent interface with the matrix. However, dispersoids can nucleate microvoids and this is undesirable. In 7075 alloy, $\text{Al}_{12}\text{Mg}_2\text{Cr}$ is a common dispersoid as shown in Figure 2.8 [1]. Al_3Zr is common in 7050 and other high strength aluminium alloys [1]. The type of dispersoids formed in an alloy can affect the quench sensitivity.

2.6.1 Quench sensitivity

Quench sensitivity is a phenomenon in heat treatable alloys whereby the properties of the alloy are dependent on the quench rate. An important factor that contributes to quench sensitivity is formation of dispersoids. Figure 2.9 shows the effect of quenching rate on strength of some aluminium alloys [1]. As the quenching rate is decreased, there is decrease in strength of both alloys.

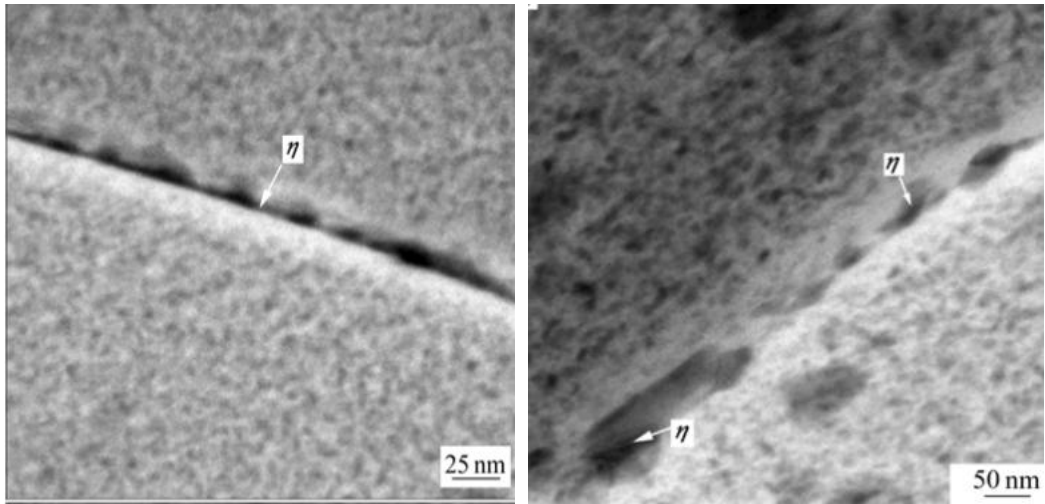


Figure 2.7: Continuous and discontinuous distribution of precipitates along grain boundary in T6 and T7 temper respectively [29]

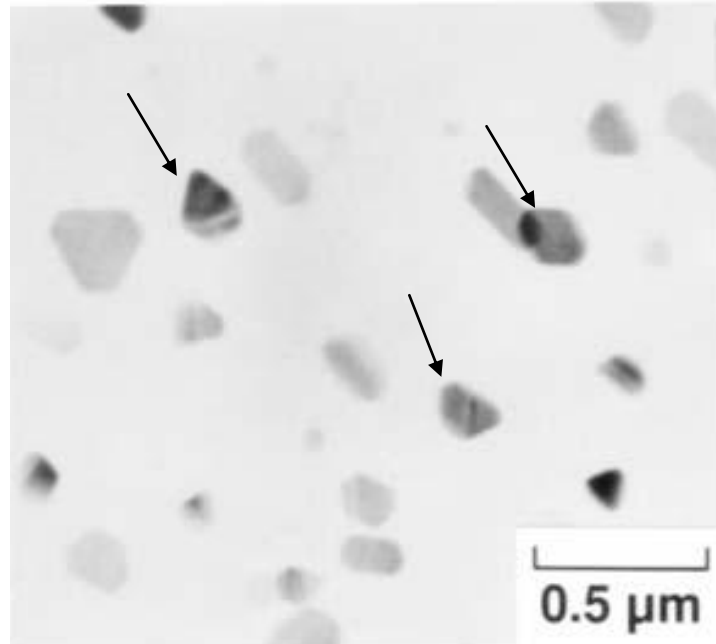


Figure 2.8: Dispersoid particles of $\text{Al}_{12}\text{Mg}_2\text{Cr}$ (indicated by the arrows) in alloy 7075 [1]

However, 7075 alloy exhibits more loss of strength compared to 7050 alloy. 7075 and 7050 alloys show that the type of dispersoid affects quench sensitivity. As shown in Figure 2.9, 7050 is less quench sensitive compared to 7075 alloy [1]. In 7075 alloy, Cr is used to control grain size and prevent recrystallization. However, Cr forms incoherent dispersoids that allows heterogeneous nucleation of precipitates to occur during quenching thereby causing reduction in strength called quench sensitivity. The loss of strength is due to reduction in the concentration of solute atoms available for the formation of strengthening phases through homogeneous precipitation. In 7050 alloys, the use of Zr allows formation of dispersoids at semi-coherent interface with the matrix and this prevents heterogeneous precipitation at the interface.

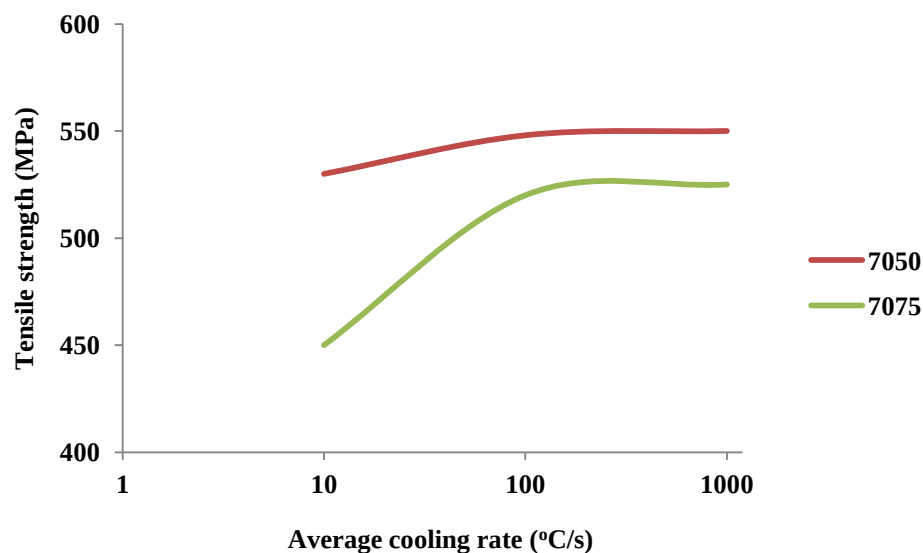


Figure 2.9: Effect of quenching cooling rate on strength of aluminium alloys [adapted from 1]

2.6.2 Electrical conductivity

Electrical conductivity (EC) is used to easily assess the stress corrosion cracking susceptibility of 7xxx series alloys. EC measurement provides an easy, fast method of distinguishing between tempers likely to be prone to SCC and those likely to be resistant. It is believed that there is a direct relationship between SCC resistance and electrical conductivity. This means that as aging increases, EC increases and the SCC resistance increases [41]. High electrical conductivity is obtained in high SCC resistant T7 while low EC is obtained in T6 temper. The minimum acceptable level of EC for T73 treated 7075 alloy is 38% IACS (International Annealed Copper Standard) while T6 is 31-33% IACS [42]. Heat treatments are often designed to obtain higher EC to increase resistance to SCC [43].

Electrical conductivity is a bulk property. The observed changes in EC of different tempers can be due to the amount of impurity element and solutes in the solid solution. A high amount of impurity elements in solution causes an increase in the resistance to flow of electrical current thereby reducing the EC value. During quenching, the high concentration of solutes in the supersaturated solid solution increases resistivity and decreases EC. EC can also be related to hardness and strength. Strength decreases due to loss of strengthening phases to the solid solution after quenching from SHT. During aging, formation of precipitates increases strength but decreases the amount of solutes in the matrix. Intermediate precipitates and GP zones are associated with coherent strain and this induces scattering effect (deviation from straight trajectory by one or more paths) on electrons thereby decreasing EC. Continuous aging causes transformation of coherent precipitates to incoherent

precipitates (as in T7 temper) causing loss of strength. This transformation reduces strain on the matrix to achieve increase in EC [41, 44, 45, 46].

Tsai et al. suggested that the correlation between EC and SCC resistance holds in the near peak aging (T4) and overaged condition (T7) in a particular alloy [44]. For instance, the EC of 7475-T6 is higher than 7075-T6 (as expected in view of the higher impurity elements in 7075). However, results of SCC test showed that the average time to failure of 7075-T6 is longer than 7475-T6. Also, EC of as-quenched 7475 is less than 7475-T6 as expected, but the average time to failure to SCC is longer in as-quenched state than the T6 [44]. This shows that even though the relationship between EC and SCC resistance has been established for T6 and T7 temper, there is need to understand the SCC response of EC values between these two extremes. The effect of HTPP heat treatment on EC of 7075 and 7050 alloys as it relates to SCC resistance will be discussed later in this thesis research.

CHAPTER THREE

LOCALISED CORROSION IN Al-Zn-Mg-Cu ALLOYS

Localized corrosion is a form of accelerated attack by a corrosive environment at discrete locations on a component while the other parts of the component corrode at a much slower rate. Localized corrosion may be due to breakdown of protective oxide film or the effect of localized condition of the environment [47]. Among different forms of localized corrosion, Al-Zn-Mg-Cu alloys are highly susceptible to intergranular corrosion, exfoliation corrosion and stress corrosion cracking. Localised corrosion affects mechanical properties and structural integrity of Al-Zn-Mg-Cu alloys.

3.1 Intergranular Corrosion

Intergranular corrosion is a form of localized corrosion in which attack is localized to grain boundaries and regions closely adjacent to grain boundaries while the grains remain unattacked [48]. The selective attack on the grain boundary region is due to segregation/depletion of impurity elements or precipitation of phases at the grain boundary. Segregation or precipitation often causes localized decrease in corrosion resistance in the alloy [48]. Intergranular corrosion penetrates into the material until the self-limiting depth is reached. The limiting depth of penetration is determined by the ease of transportation of corroding species and oxygen through the corrosion path along the grain boundary. At the limiting depth, intergranular corrosion spreads over the surface laterally. Intergranular corrosion is highly deleterious because it is difficult to detect without the help of a microscope. Also, the sharp crack tips serve as stress raisers making the component more prone to fatigue failure [32, 49].

3.2 Exfoliation Corrosion

Exfoliation corrosion is a form of intergranular corrosion also referred to as layered or stratified corrosion. Exfoliation corrosion involves localized attack and build-up of insoluble corrosion products along the grain boundary. Figure 3.1 shows an example of exfoliation corrosion [50]. The expansion of insoluble corrosion products exerts pressure on the grains causing swelling, leafing or lifting effect as shown by the arrows in Figure 3.1. Exfoliation corrosion often starts from the sheared edge of the material and progresses laterally into the material [32, 49, 51].

Exfoliation corrosion is highly deleterious because the corrosion rate is linear. This is due to continuous splitting action of the grains exposing a fresh, film free surface. Also, the strength of the material is lost due to the splitting off of uncorroded material [32, 49].

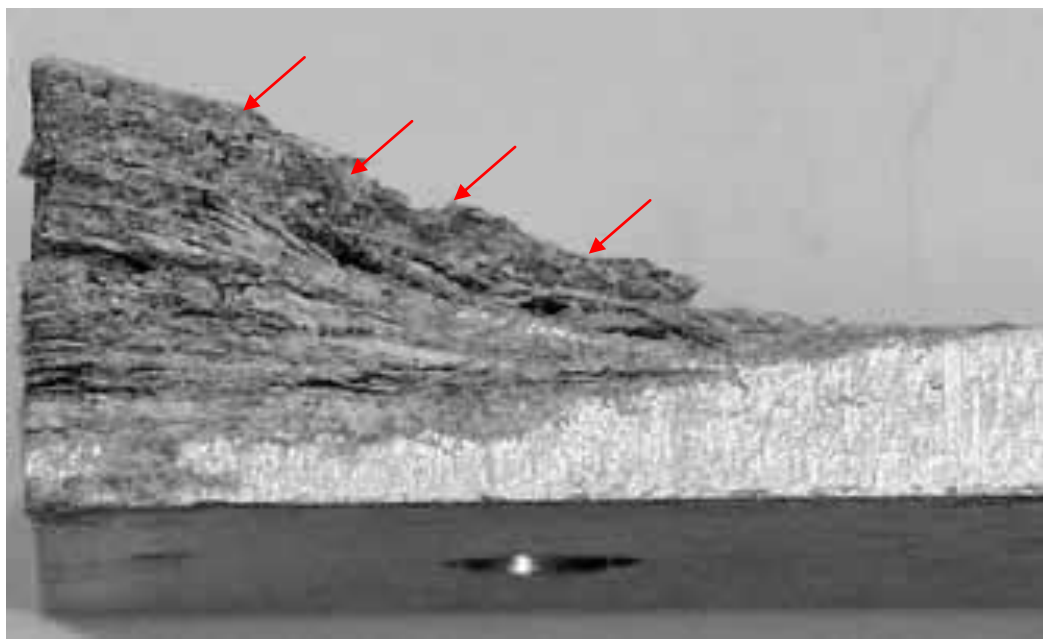


Figure 3.1: Exfoliation corrosion causing delamination shown by the red arrows [50]

Exposure to salt, acidic deposits and high temperature accelerates the delamination of a susceptible material. Al-Zn-Mg-Cu alloys are highly susceptible to exfoliation corrosion especially in the peak age (T6) condition. The mechanism involves electrochemical reaction between grain boundary precipitate (anode) and the matrix (cathode) accompanied by expansion of insoluble corrosion products [32, 49].

3.3 Stress Corrosion Cracking

Stress corrosion cracking (SCC) is a form of intergranular corrosion that involves the conjoint interaction of tensile stress, corrosive environment (water, atmospheric moisture or aqueous solutions) and a susceptible material [32]. SCC often causes brittle failure of a susceptible material. SCC is also a form of environmentally assisted cracking because external influence of the environment is required. The stress required for SCC to occur may be externally applied (due to use of the component in service) or residual (initiated during manufacturing) at stress levels lower than the yield stress [32]. As indicated by the arrows, Figure 3.2 shows the presence of stress corrosion cracks in a component. SCC is highly deleterious because it is difficult to detect by casual inspection, it causes slight metal loss but higher loss of mechanical strength. Also, the cracks produced can initiate fast fracture of component as shown in Figure 3.2 [52]. SCC in aluminium alloys is characterized by the presence of static tensile stress. Most intergranular cracks are propagated perpendicular to tensile stress [32, 49]. Fracture mechanics is usually used to evaluate SCC susceptibility. This involves the use of pre-cracked specimens to relate the plane strain stress intensity at the crack tip to crack growth velocity [53].

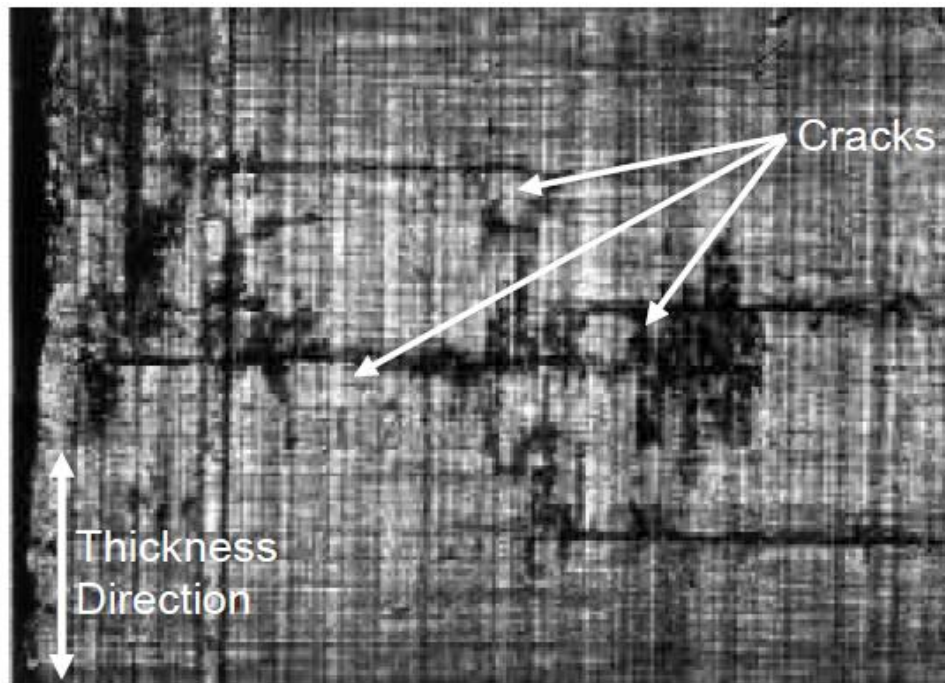


Figure 3.2: Stress corrosion cracking (shown by arrows) in a 7075-T6 wing lock [52]

3.3.1 Mechanism of Stress Corrosion Cracking

The actual mechanism of SCC is not fully understood. However, two commonly accepted theories are stress assisted dissolution and stress sorption cracking mechanism [33].

Stress assisted dissolution considers that heat treatment develops certain microstructural features (e.g. grain boundary precipitates, precipitate free zone) with different electrochemical potentials. In a corrosive environment, certain features may be anodic with respect to others. This generates a galvanic cell, causing dissolution of the anodic part leading to crack initiation by formation of pits [33]. Pits serve as stress raisers such that application of tensile stress causes plastic deformation in the crack tip region due to high stress concentration. The cold work effect of plastic deformation in this region increases the local energy level making the material more susceptible to

corrosion. Film rupture mechanism proposes that protective film is initiated after anodic dissolution. However, further application of tensile stress causes rupture of the passive film, allowing more metal dissolution in the environment and continuous corrosion of the material [54].

In Al-Zn-Mg-Cu alloys, precipitation of $MgZn_2$ occurs preferentially at the grain boundary. This creates the precipitate free zone that is depleted of precipitates adjacent to the grain boundary. The difference in electrochemical potentials sets up a galvanic corrosion cell between the PFZ and the solute rich matrix causing anodic dissolution [32, 49]. However, experimental results showing that condensation will not occur at the tip of a propagating crack at relatively low humidity, suggest that anodic dissolution might not be responsible for such cracking [55].

The stress sorption cracking mechanism proposes that SCC occurs due to adsorption of damaging species into the material causing weakening of atomic bonds. The presence of the damaging species in the interstitial spacing of the grain boundary reduces the surface energy and weakens the atomic bonds. Hydrogen is a peculiar damaging specie causing hydrogen damage or hydrogen induced cracking (HIC) [33]. HIC is common in 7xxx aluminium alloys and it is due to incomplete reduction of hydrogen ion. This causes high concentration of hydrogen atoms. The diffusion of the atomic hydrogen into the grain boundary of the material causes breakdown of atomic bonds. Also, the atomic hydrogen could migrate to preferred locations like voids and combine to produce hydrogen gas. This reaction generates high pressure that causes rupture of the material [33, 54].

3.4 Effect of slip character on stress corrosion cracking in Al-Zn-Mg-Cu alloys

Aging heat treatment carried out after quenching develops microstructural features that influence the SCC behavior of a material. An important effect of aging is the change in slip character of dislocations in the material. Slip character measures the extent of dispersion of dislocations during plastic deformation and it is an important factor used to explain susceptibility of 7xxx alloys to SCC [14].

Within the underaged to peak age condition, the precipitates are small and deformable. This causes long dislocation lines and planar slip character. Figure 3.3 shows planar slip character of dislocations in a nickel based superalloys [56]. In planar slip, a high concentration of dislocations form a planar array due to presence of shearable, semi-coherent or coherent phases like GP zones and intermediate precipitate (η') structures as in T6 temper of Al-Zn-Mg-Cu alloys. Dislocations interact with the coherent precipitates causing shearing of the precipitates and reducing resistance to dislocation motion. Planar slip bands formed due to this interaction impinges on the grain boundary causing shear stress concentration. Therefore, in planar slip, cracks with sharp tips are formed, increasing the susceptibility to SCC [40, 53, 57].

Figure 3.4 shows the wavy slip character of dislocations in a nickel based superalloys [56]. Overaging heat treatment causes precipitate particles to grow and lose coherency with the matrix. Due to prolonged aging, there is decrease in the concentration of dislocations. Dislocation-particle interaction occurs through bypassing the non-deformable precipitates. This generates wavy slip character that is uniformly distributed. Lesser concentration of dislocations with wavy slip character causes blunting of the crack tip thereby increasing SCC resistance [40].

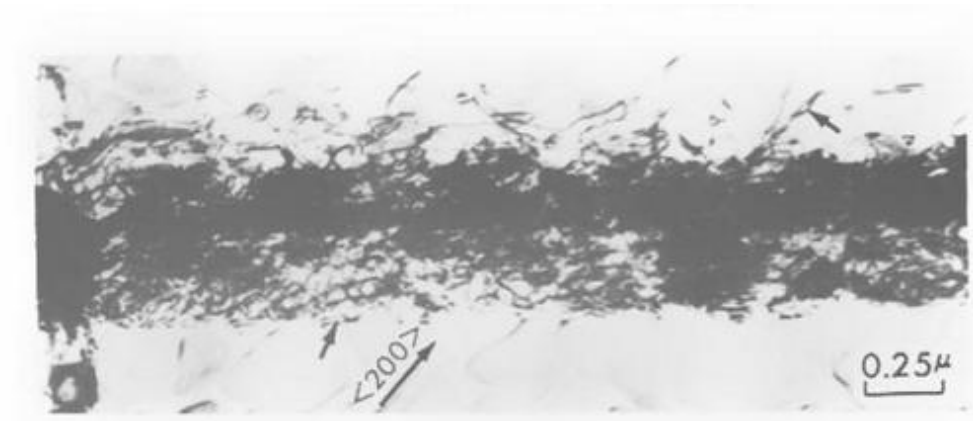


Figure 3.3: TEM micrograph showing planar dislocation slip in a nickel based superalloy [56]

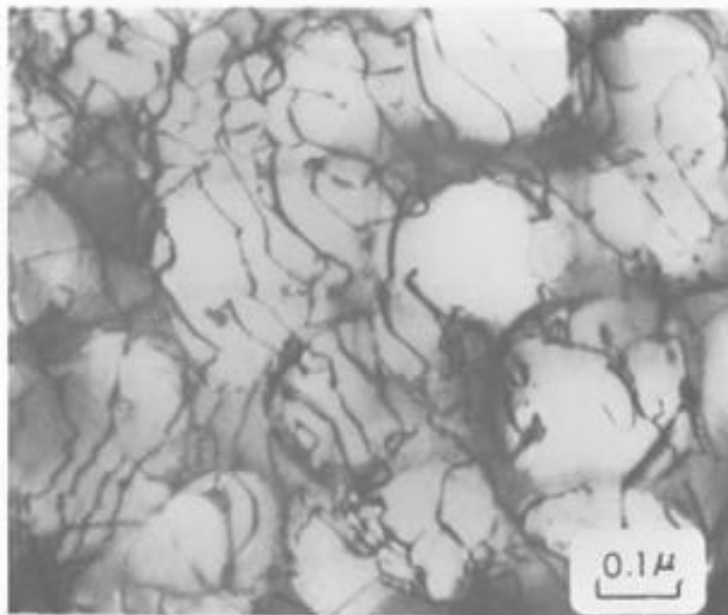


Figure 3.4: TEM micrograph showing dislocations in wavy slip in a nickel based superalloy [56]

3.5 Influence of grain boundary precipitate structure on SCC

The structure of the grain boundary precipitate is largely affected by aging treatment. As aging progresses, there is transition from continuous distribution of precipitates (formed during the peak age) to discontinuous distribution in the overaged condition. In Al-Zn-Mg-Cu alloys, overaging decreases the crack growth rate as the grain boundary precipitates becomes coarse and discontinuous [58]. Grain boundary precipitate morphology has been widely used to explain the theory that SCC occurs through anodic dissolution of precipitates in Al-Cu and Al-Zn-Mg-Cu alloys [40].

The actual role of grain boundary precipitates in anodic dissolution mechanism of SCC is not clear. Decrease in crack growth rate as the size of grain boundary precipitates increases has been used to explain the decrease in susceptibility of Al-Zn-Mg-Cu alloy to SCC. One explanation for this is that grain boundary act as internal sacrificial anode during SCC [40]. This means that in Al-Zn-Mg-Cu alloys, SCC susceptibility is due to sacrificial dissolution of grain boundary precipitates. In highly susceptible conditions like T6 temper, small volume of continuous grain boundary precipitates dissolves sacrificially, thereby increasing cracking along the grain boundary region. Heat treatment to T7 and RRA temper causes the precipitates to grow to larger volume such that anodic dissolution does not increase SCC [59]. Higher SCC resistance has also been related to the ability of large grain boundary precipitates to act as hydrogen discharge sites thereby reducing SCC through HIC [60, 61]. However, other studies have reported that SCC resistance is improved by increase in the amount of copper in grain boundary precipitates [62].

3.6 Influence of magnesium and copper content

Segregation of magnesium has been used to explain hydrogen embrittlement in Al-Zn-Mg-Cu alloys. Song et al. showed that the amount of magnesium segregated at the grain boundary decreases as the aging time increases in 7050 alloy aged at 145 °C [63]. The decrease in magnesium solute segregated at the grain boundary can be related to increase in the precipitation of MgZn_2 precipitate as aging time increases. Segregation of magnesium solute at the grain boundary accelerates hydrogen embrittlement by increasing diffusion and absorption of hydrogen. The reaction of magnesium and hydrogen causes reduction in fracture toughness thereby increasing grain boundary embrittlement [63]. It has been shown that magnesium is important in promoting environmentally active mechanical behavior in 7xxx series alloys [64]. This is supported by results showing that:

- magnesium oxide is preferentially developed at intersections of grain boundary on external surface.
- magnesium segregation is observed at the grain boundary in as-quenched state [65].

Grain boundary segregation studied by Hepples et al. show that Mg segregated at the grain boundary in 7150-T651 is higher compared to matrix but, grain boundary segregation of Mg increased as aging increased in T7 and T77 conditions [62]. Higher SCC resistance in T7 and RRA compared to T651 temper shows that Mg segregation in the grain might not be the main factor responsible for SCC in Al-Zn-Mg-Cu alloys.

Compositional modifications in the development of Al-Zn-Mg-Cu alloys involve increasing copper content to improve corrosion resistance as observed in 7079 to 7075 and 7050 alloy [15]. Research work on the effect of copper on SCC shows that

increasing the copper content of alloys decreases crack growth velocity in Al-Zn-Mg-Cu alloys [66]. The presence of copper modifies the difference between the electrochemical potential at the matrix and the grain boundary region thereby reducing SCC through an anodic dissolution mechanism [67]. Also, a high amount of copper slows down the kinetics of hydrogen evolution [68]. Copper also segregates at the grain boundary causing toughening of the grain boundary region [64]. It has been reported that copper content in grain boundary precipitate of 7075 alloy in the overaged condition is higher compared to peak aged condition. This shows that changes in the chemistry of grain boundary to increase copper content can improve SCC resistance [62].

3.7 Crack initiation versus propagation and damage tolerance design requirement

The relationship between stress corrosion crack growth velocity (da/dt) and stress intensity at the crack tip (K) is shown in the stages involved in SCC. Figure 3.5 shows the V-K (crack growth velocity versus stress intensity) plot established for materials that are susceptible to SCC [13]. This plot is used to evaluate SCC behavior of susceptible materials. As shown in the Figure, the upper limit given as stress intensity factor (K_{IC}) is a design factor of the material [40]. K_{IC} corresponds to the plane strain crack tip stress intensity that causes unstable fast fracture [40, 69].

Three distinct regions are shown in Figure 3.5 [69]. Region I on the V-K plot shows the first stage of SCC at low stress levels. In this region, crack growth rate is dependent on stress intensity. K_{ISCC} is referred to as threshold stress level. At stresses below the K_{ISCC} , unstable crack growth does not occur. In region I, as the stress intensity increases from K_{ISCC} , the crack growth rate increases.

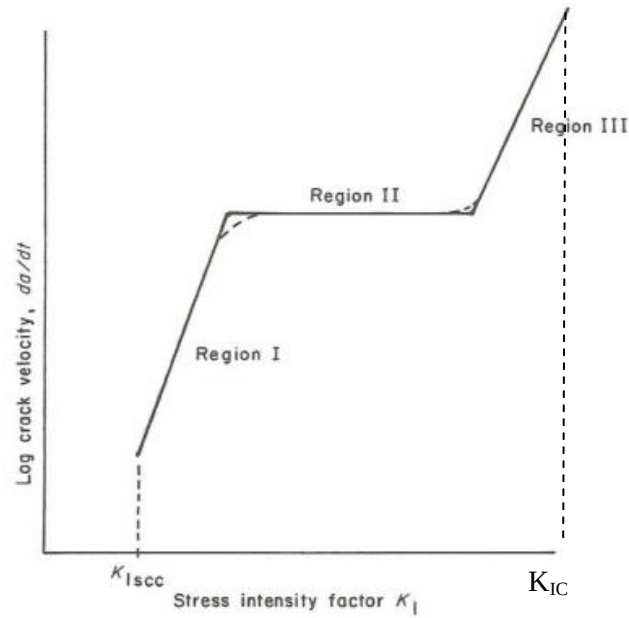


Figure 3.5: Stages of SCC showing K_{IC} and K_{ISCC} [13]

Region II shows the second stage of SCC and the crack growth rate is independent of stress intensity. As stress intensity increases, the velocity of crack growth remains constant. This shows the influence of other factors like environment and composition of materials on crack growth rate [69]. In region III, the crack growth rate is dependent on stress intensity. As the stress intensity approaches K_{IC} , the crack growth rate also increases causing fracture of the component. Thus, the stages in crack growth can be divided into crack initiation (stage I), propagation (stage II) and fracture (stage III) based on Figure 3.5. K_{ISCC} (in region I) and plateau velocity (in region II) are often used to characterize susceptibility or resistance of alloys to SCC. Higher K_{ISCC} and lower plateau velocity are common in SCC resistant tempers like T7 and RRA compared to T6 temper [24].

Although SCC is as a result of several factors, infinite life design can be used to reduce catastrophic failure due to SCC. This requires that the sum of in-service,

residual and fabrication stresses must be below the threshold stress level. However, at such low stress intensity, the component will be thick and heavy. Conversely, application of damage tolerance in design enables the structure to accommodate cracks or flaws within stage I and II such that inspection and repair process would detect the flaw before it can cause catastrophic failure [70, 71]. Although structures with thin sections can be designed with this approach, there is higher risk of missing cracks during inspection causing unexpected fracture of the component.

CHAPTER FOUR

EFFECT OF HEAT TREATMENT ON SCC RESISTANCE OF Al-Zn-Mg-Cu ALLOYS

The main heat treatments that have achieved better resistance to SCC in Al-Zn-Mg-Cu alloys are overaging (T7) and RRA (T77) heat treatments.

4.1 Overaging

Overaging involves prolonged aging of the alloy beyond maximum hardness from T6 to T7 temper. T6 temper is obtained by SHT at about 465 °C for 1-2 hours followed by quenching and aging at 121 °C for 24 hours. T7 is obtained by overaging alloy in T6 temper at about 177 °C for 7-8 hours [1]. Overaging heat treatment achieves increase in SCC resistance.

A number of factors have been used to explain the improvement in SCC resistance through overaging heat treatment. It has been reported that overaging causes increase in size and discontinuity in the distribution of precipitates along the grain boundary as shown in Figure 2.7 [29]. Discontinuous distribution of large precipitates reduces anodic dissolution and prevents a continuous path for corrosion along the grain boundaries [29]. Also, prolonged aging causes transformation of semi-coherent η' phase to incoherent η phase thereby changing slip character of dislocations at the grain boundaries from planar to wavy slip. In wavy slip character, homogenous distribution of dislocations causes blunting of crack tip and increases SCC resistance [40]. Large grain boundary precipitates formed during overaging can trap atomic hydrogen to form bubbles. These bubbles cause blunting of the crack tip thereby reducing susceptibility to SCC through hydrogen embrittlement [24, 33]. However, the ≈ 10 -15% loss of strength in T7 is very significant in view of high strength:weight

requirement for structural parts in aerospace applications. This was the main motivation for the development of RRA heat treatment.

4.2 Retrogression and reaging

The retrogression and reaging heat treatment was developed by Cina based on the principle that SCC is due to the high dislocation density formed during quenching from SHT [14]. RRA involves carrying out peak aging heat treatment to achieve T6 temper followed by retrogression treatment at a temperature higher than age hardening temperature but below the SHT temperature. As shown in Figure 4.1, 7075 alloys are usually retrogressed at 200-260 °C for about 5 minutes. The retrogression step is carried out for a short time to prevent overaging. This is followed by reaging step (at T6 aging condition) to allow precipitation of the strengthening phase to reoccur [14].

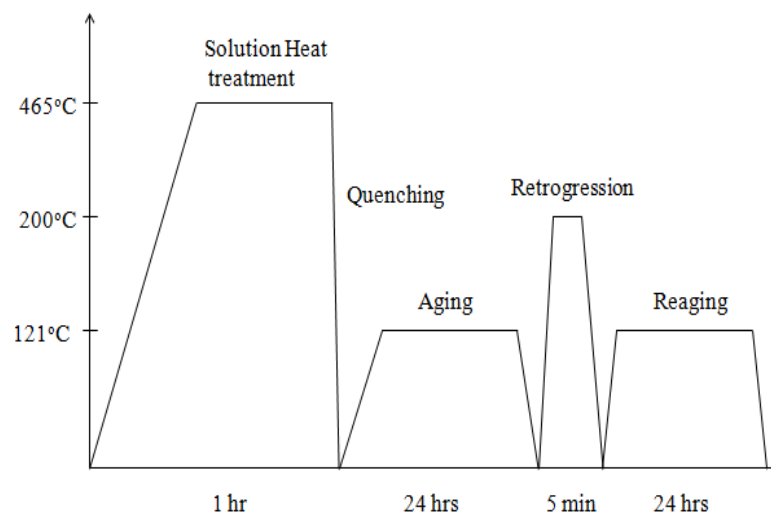


Figure 4.1: Schematic showing retrogression and reaging heat treatment procedure

It is believed that the short retrogression step dissolves dislocation network responsible for SCC [14]. Various works have shown that RRA can achieve high SCC resistance and high strength and as obtained in T6 and T7 respectively [24, 43, 72]. Figure 4.2 shows the effect of RRA treatment on SCC resistance of 7075 alloy [24]. RRA samples treated to retrogression heat treatment at 220 °C for 1 and 5 minutes respectively followed by reaging showed better SCC resistance compared to standard T6 temper. Also, high SCC resistance was observed in the sample subjected to only retrogression heat treatment without reaging. This is shown by the higher K_{ISCC} and lower stage II plateau crack growth rate in T7 and RRA samples compared to T6 temper. Further work done by Cina reaffirmed that RRA improves SCC resistance through dissolution of dislocation density during the retrogression step [14, 73]. It is believed that the high temperature involved in retrogression step is just enough to cause dissolution of quenched-in dislocations without causing overaging.

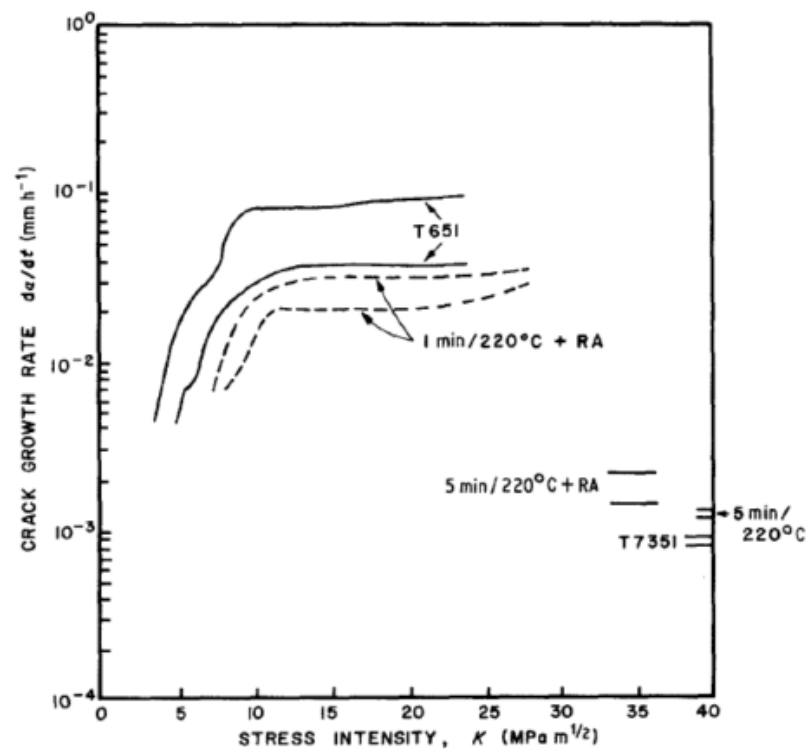


Figure 4.2: Effect of heat treatment on SCC resistance in 7075 alloy heat treated to T6, T73 and RRA retrogressed at 220°C for 1 and 5 minutes respectively [24]

Dislocations can transport hydrogen atoms through the grain boundaries. Also, high dislocation density provides additional stress field at the grain boundary that can cause crack initiation. Hence, Cina's work showed that decrease in dislocation density after RRA treatment can be related to increase in SCC resistance [73].

Increase in SCC resistance through RRA has also been related to large, discontinuously distributed precipitates at the grain boundary. Figure 4.3 shows discontinuous distribution of precipitates along the grain boundary of 7075 alloy after RRA treatment [24, 29, 40]. Since SCC occurs at the grain boundary, discontinuous distribution of precipitates improves SCC resistance by preventing a continuous path for corrosion to occur. As explained in the previous chapter, the size of grain boundary precipitate is also important in preventing SCC. RRA produces coarse precipitates at the grain boundary region thereby preventing SCC by anodic dissolution of grain boundary precipitates [29].

However, application of RRA to thick sections of materials is difficult due to the short retrogression time involved. Although Figure 4.2 shows that longer retrogression time can achieve higher SCC resistance, Figure 1.4 shows that longer retrogression time causes overaging and loss of strength. In order to achieve a high SCC resistance and high strength, it is important to use a very short time for the retrogression step. However, short retrogression time makes it difficult for the heat treatment to achieve uniformity in the distribution of properties in thick sections. This limits the application of RRA heat treatment to thin section of materials. 7075 and 7050 alloys used as structural materials in aircrafts are usually in thick sections.

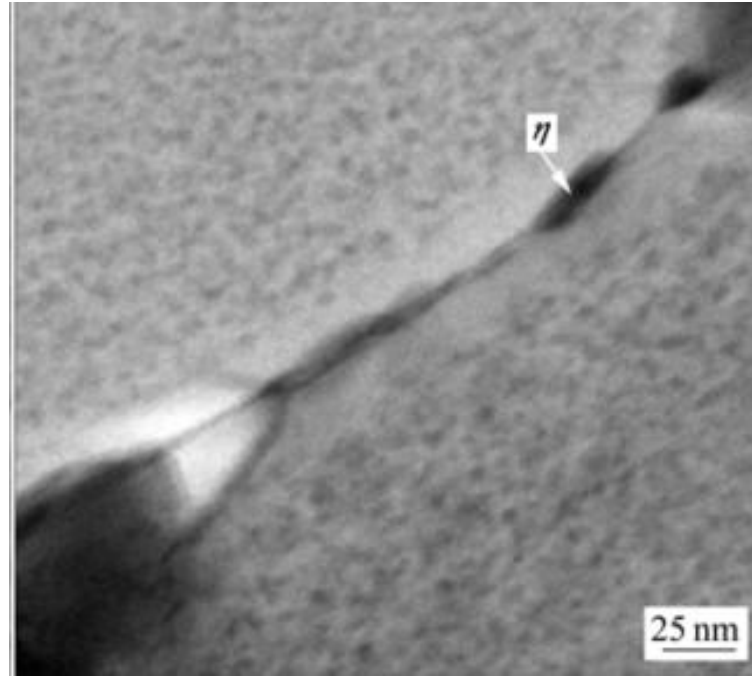


Figure 4.3: Discontinuous grain boundary precipitates in 7075 RRA [29]

Hence, there is need to develop a heat treatment that will achieve a good combination of strength and SCC resistance without using high temperature involved in retrogression process.

4.3 High Temperature Pre-precipitation heat treatment

High temperature pre-precipitation (HTPP) heat treatment has been suggested as a potential heat treatment procedure that could increase resistance of 7xxx series alloys to SCC [26, 28]. Figure 4.4 shows the steps involved in HTPP process. HTPP involves solution heat treatment at a temperature in the single phase region followed by controlled cooling to a temperature in the two phase region and quenching. This is followed by aging as shown in Figure 4.4. It is believed that due to the controlled cooling through the solvus, preferential heterogeneous precipitation will occur at the grain boundary region.

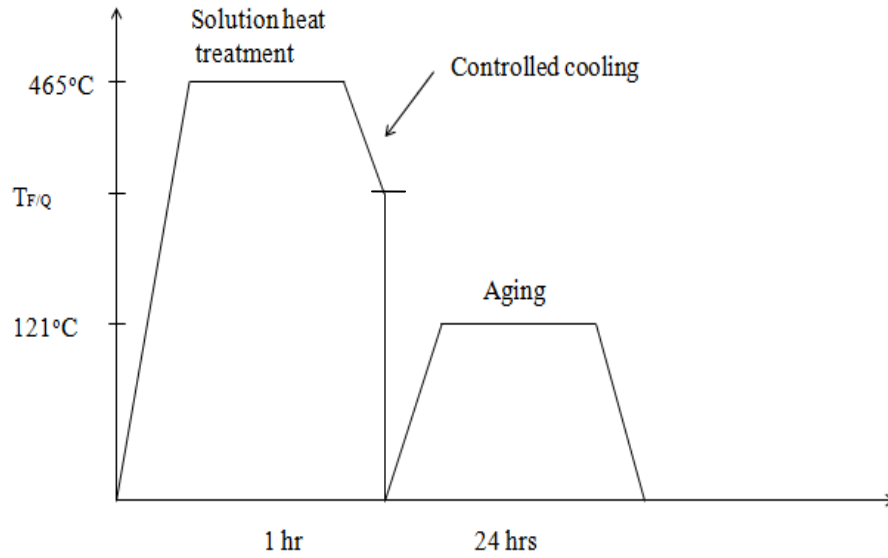


Figure 4.4: High temperature pre-precipitation heat treatment procedure showing (intermediate) $T_{F/Q}$ temperature

These precipitates serve as nucleation sites for more precipitation to occur during subsequent artificial aging at the normal T6 conditions [29]. Previous research by Huang et al. studied the effect of HTPP heat treatment on experimental 7A52 alloy (composition is included in Table 1.1) [26]. Stepped SHT was carried out at 465 °C for 30 minutes, heated to 470 °C for 30 minutes and 475 °C for 1 hour. This was followed by controlled cooling at 30 °C/h to 400 °C. This temperature was held for 30 minutes followed by quenching and aging at 100 °C for 10 hours and 150 °C for 24 hours. The results showed that HTPP can achieve a combination of increase in strength and resistance to SCC without significant loss of strength (just about 1.8% loss of strength compared to sample without pre-precipitation) in experimental 7A52 alloy. TEM observation of 7A52 alloy after HTPP showed the presence of finely distributed coherent, metastable precipitates within the grains and a discontinuous distribution of incoherent, equilibrium precipitates at the grain boundary region [11].

In another study, Huang et al. carried out HTPP treatment on 7055 alloy using stepped SHT at 450 °C for 1 h, 470 °C for 1 h and 480 °C for 3 h [28]. This was followed by controlled cooling of different samples at 30 °C/h to 465 °C and 455 °C respectively. The samples were held at this temperature for thirty minutes before quenching. Aging was carried out at 130 °C for 24 h. The results showed that HTPP can achieve discontinuous distribution of precipitates at the grain boundary, decrease dislocation density and improve SCC resistance without significant loss of strength.

Further study of 7075 alloy by Li et al. showed that HTPP achieved better SCC resistance, but loss of strength in the alloy [29]. Figure 4.5 shows TEM micrograph of 7075 matrix after HTPP. Figure 4.5 (a) shows that the loss of strength after HTPP can be explained by decrease in the density of fine, coherent, non-equilibrium precipitates in the matrix due to loss of solute element to grain boundary region during heterogeneous precipitation. Hence there is lesser amount of solute available to form the strengthening precipitates in the matrix [29]. The loss of strength can also be due to formation of coarse precipitates in the matrix as shown in Figure 4.5 (b). Figure 4.6 shows the precipitate morphology at the grain boundary region. Discontinuous distribution of equilibrium precipitates observed at the grain boundary region was related to heterogeneous precipitation due to controlled cooling below the solvus [29].

In view of this, it is important to analyze the effect of HTPP heat treatment on commercial 7xxx alloys in terms of strength and resistance to SCC. This thesis research will focus on applying HTPP heat treatment to commercial 7050 and 7075 aluminium alloys. Particularly, the effect of the intermediate temperature to which the sample is cooled before quenching will be studied and related to strength (measured in terms of hardness) and electrical conductivity while corrosion resistance will be determined by exfoliation corrosion and double beam SCC tests.

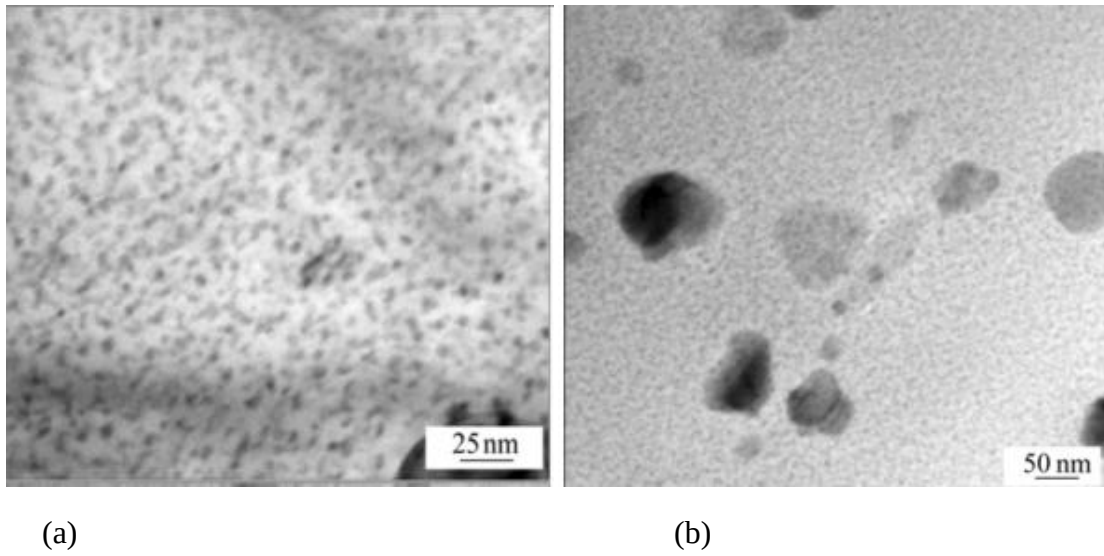


Figure 4.5: Micrographs of (a) fine and (b) coarse precipitates in the grains of HTPP temper [29]

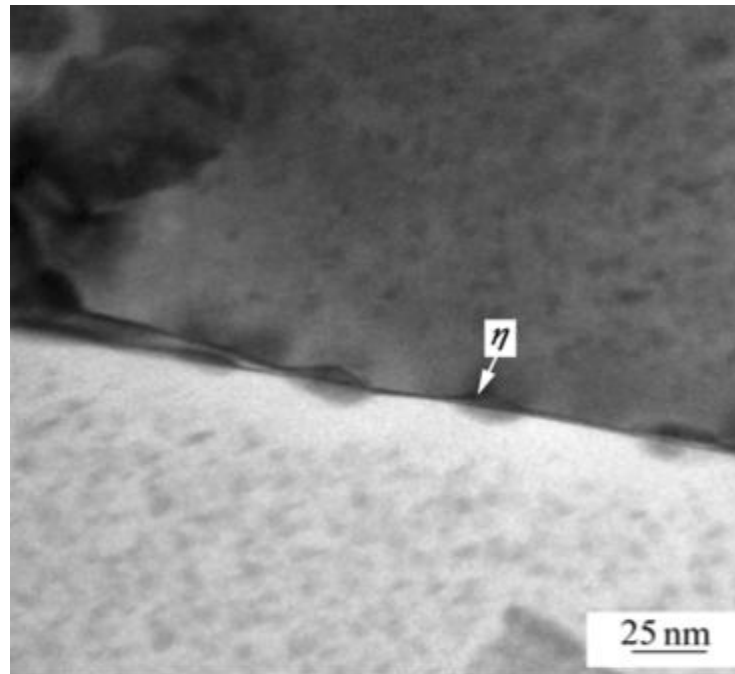
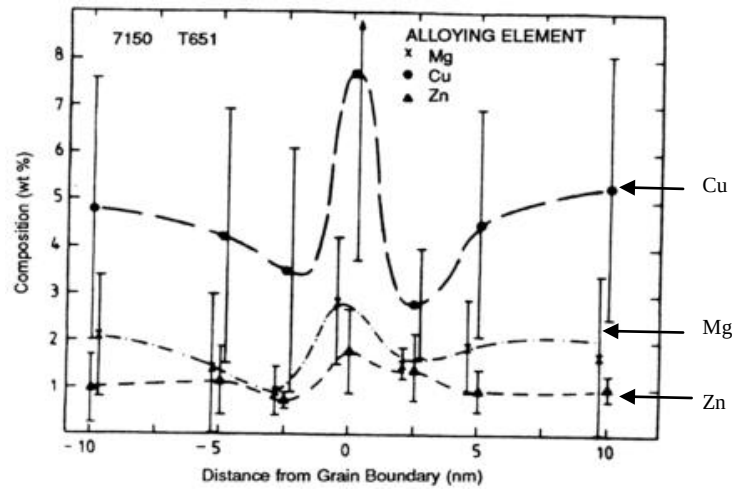


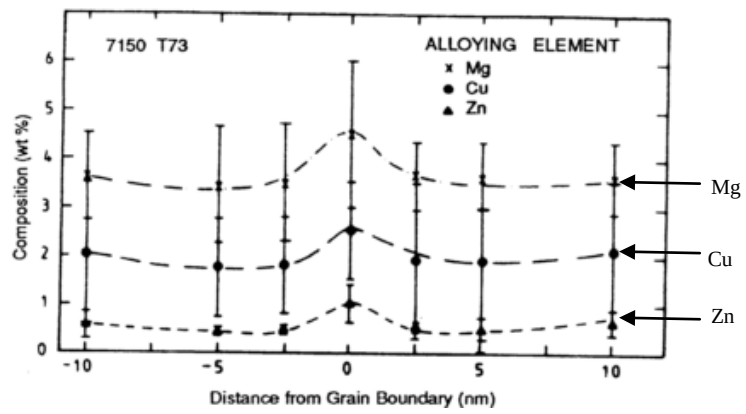
Figure 4.6: Discontinuous precipitates at the grain boundary region of HTPP temper [29]

4.4 Effect of heat treatment on grain boundary segregation

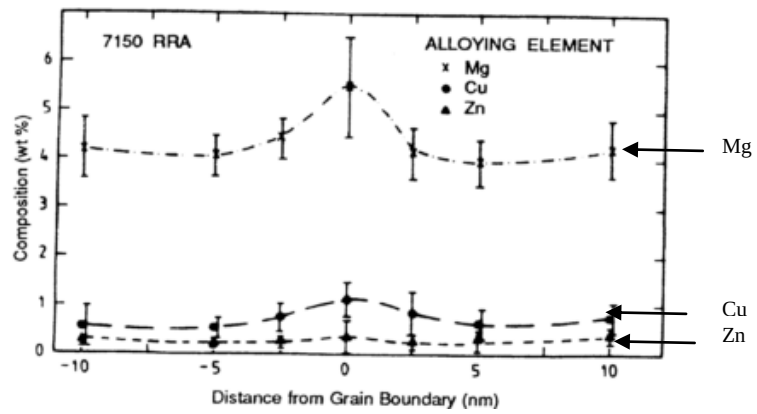
Heat treatment affects solute segregation along the grain boundaries. Hepples et al analyzed the effect of T651, T73 and RRA heat treatment conditions on grain boundary segregation in 7150 alloy [62]. Figure 4.7 shows that the copper solute concentration in T651 is high as shown in Figure 4.7 (a). Figure 4.7 (b) and (c) shows that there is decrease in copper solute concentration at the grain boundary in T73 and RRA conditions. This corresponds to a higher copper content of grain boundary precipitates in T73 and RRA compared to T651 temper. Similar trend was observed for Zn. Low Mg level was observed in T651 but, this increased in T73 and RRA conditions [62]. Huang et al. showed that HTPP can increase the copper solute content of grain boundary precipitates [26].



(a)



(b)



(c)

Figure 4.7: Concentration profile of Zn, Mg and Cu solute segregated across the grain boundary of (a) T651 (b) T73 (c) RRA tempers of 7150 alloy [62]

CHAPTER FIVE

EXPERIMENTAL PROCEDURE

The effect of HTPP on properties of 7075 and 7050 commercial aluminium alloys was determined by carrying out HTPP treatment on the as-received tempers of both alloys (T651 and T74 respectively). Samples of both materials were subjected to HTPP treatment and standard heat treatments of peak aging (T6) and overaging (T7) temper. The effect of the heat treatment on hardness and electrical conductivity were tested before and after aging. Exfoliation corrosion tests were carried out on selected heat treated samples. Double beam stress corrosion tests were used to determine SCC resistance of selected heat treated samples. Transmission electron microscopy was used to study microstructural features responsible for these properties.

5.1 Experimental materials

The materials used in this research are rolled plate of 7075-T651 and 7050-T74 of thickness 1 in and 1.5 in respectively. The actual composition (given by suppliers) and nominal compositions of the alloys are listed in Table 5.1. The longitudinal (L) direction is parallel to the rolling direction of the plate, transverse (T) direction is perpendicular to the rolling direction while short (S) direction is parallel to the thickness of the plate.

5.2 Heat treatment procedure

The heat treatments applied are standard T6, T7 tempers and ten HTPP treatments. The heat treatment procedure is listed in Table 5.2. Heat treatments were conducted in a box furnace shown in Figure 5.1. To achieve the desired sample temperature, a

thermocouple located immediately adjacent to the sample was used as an input to the furnace temperature control. The furnace was heated to 465 °C for about two hours.

Table 5.1: Nominal and actual composition of 7075 and 7050 alloys used*

Alloy	Composition (wt %)										
	Al	Zn	Mg	Cu	Fe	Si	Mn	Ti	Cr	Zr	Others
7075 Nom.	Bal.	5.1- 6.1	2.1- 2.9	1.2- 2.0	0.5	0.4	0.3	0.2	0.2- 0.3	0.01	0.5
7075 Act.	Bal.	5.6	2.5	1.5	0.2	0.09	0.03	0.03	0.20	0.01	0.03
7050 Nom.	Bal.	5.7- 6.7	1.9- 2.6	2.0- 2.6	0.2	0.12	0.1	0.06	0.04	0.08- 0.12	0.5
7050 Act.	Bal.	6.0	2.2	2.22	0.1	0.04	0.003	0.02	0.004	0.11	0.03

Table 5.2: Analysis of heat treatment procedure

MATERIAL TEMPER	HEAT TREATMENT PROCEDURE[§]
T651	As-received (7075 alloy)
T74	As-received (7050 alloy)
T6	SHT 465 °C/1.5 h + WQ + 121 °C/24 h
T7	SHT 465 °C/1.5 h + WQ + 121 °C/24h + 177 °C /7 h
T_{F/Q} = 455 °C	SHT 465 °C/1 h + cool to 455 °C + WQ + 121 °C/24 h
T_{F/Q} = 445 °C	SHT 465 °C/1 h + cool to 445 °C + WQ + 121 °C/24 h
T_{F/Q} = 435 °C	SHT 465 °C/1 h + cool to 435 °C + WQ + 121 °C/24 h
T_{F/Q} = 425 °C	SHT 465 °C/1 h + cool to 425 °C + WQ + 121 °C/24 h
T_{F/Q} = 415 °C	SHT 465 °C/1 h + cool to 415 °C + WQ + 121 °C/24 h
T_{F/Q} = 405 °C	SHT 465 °C/1 h + cool to 405 °C + WQ + 121 °C/24 h
T_{F/Q} = 395 °C	SHT 465 °C/1 h + cool to 395 °C + WQ + 121 °C/24 h
T_{F/Q} = 385 °C	SHT 465 °C/1 h + cool to 385 °C + WQ + 121 °C/24 h
T_{F/Q} = 375 °C	SHT 465 °C/1 h + cool to 375 °C + WQ + 121 °C/24 h
T_{F/Q} = 370 °C	SHT 465 °C/1 h + cool to 370 °C + WQ + 121 °C/24 h

[§] SHT-solution heat treatment, WQ-water quench

The samples were then placed in the furnace for SHT. SHT was carried out at 465 °C and a period of about ten minutes was allowed before starting the solution heat treatment (SHT) time to allow the sample to reach the solutionizing temperature. For standard tempers like T6 and T7, SHT was carried out for one and half hours. This was followed by quenching in cold water at 0 to 2 °C. The time lapse before quenching did not exceed 15 seconds as specified in the standard [33]. This was followed by aging at 121 °C for 24 hours using a silicone oil bath shown in Figure 5.2 to obtain T6 temper. T7 temper was obtained by further aging of T6 samples at 177 °C for 7 hours.



Figure 5.1: Box furnace used for HTPP treatment



Figure 5.2: Fisher Scientific silicon oil bath used for aging treatment

The procedure for HTPP treatment is schematically represented in Figure 5.3. HTPP samples were first subjected to SHT at 465 °C for one hour followed by slow cooling at 30 °C/h to an intermediate temperature from 455 to 370 °C at 10 °C intervals. The intermediate temperature to which the sample was cooled before quenching is referred to as $T_{F/Q}$ (intermediate temperature between SHT in the furnace and quenching). As shown in Figure 2.4, the choice of $T_{F/Q}$ temperature is based on the solvus temperature of the alloy. For 7075 alloy, solvus temperature is approximately at 420 °C. It is believed that appreciable precipitation would occur just below the solvus temperature. Hence, cooling to temperatures above and below solvus will show the effect of pre-precipitation on the properties of the alloys. Previous work show that cooling rate of 30 °C/h has been useful in studying the effect of HTPP on Al-Zn-Mg-Cu alloys [26, 27, 28, 33].

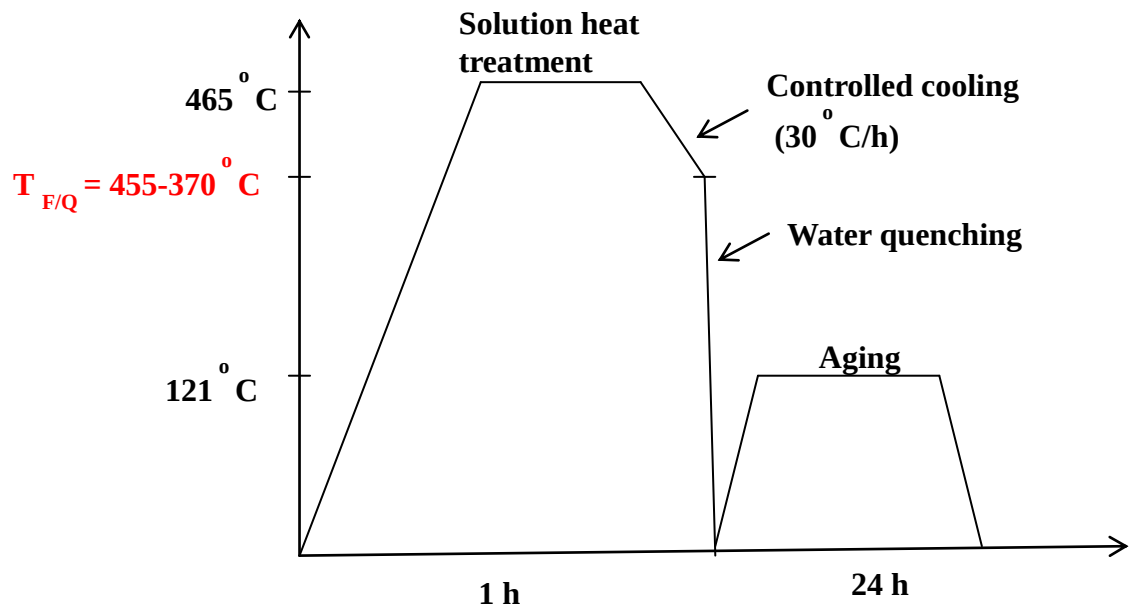


Figure 5.3: Heat treatment procedure for HTPP

5.3 Electrical conductivity testing

Following heat treatment, the samples were prepared by polishing to 1200 fine grit on SiC paper. Electrical conductivity testing was carried out using digital electrical conductivity tester (EE0021 eddy current device) shown in Figure 5.4. For each measurement, the meter was first calibrated using standard copper and aluminium samples of known EC. Percent error in EC measurements is 1.5% of readings. The probe of the tester was placed on the polished surface to obtain values of electrical conductivities in the transverse-short (TS) direction. An average of three different measurements was recorded.

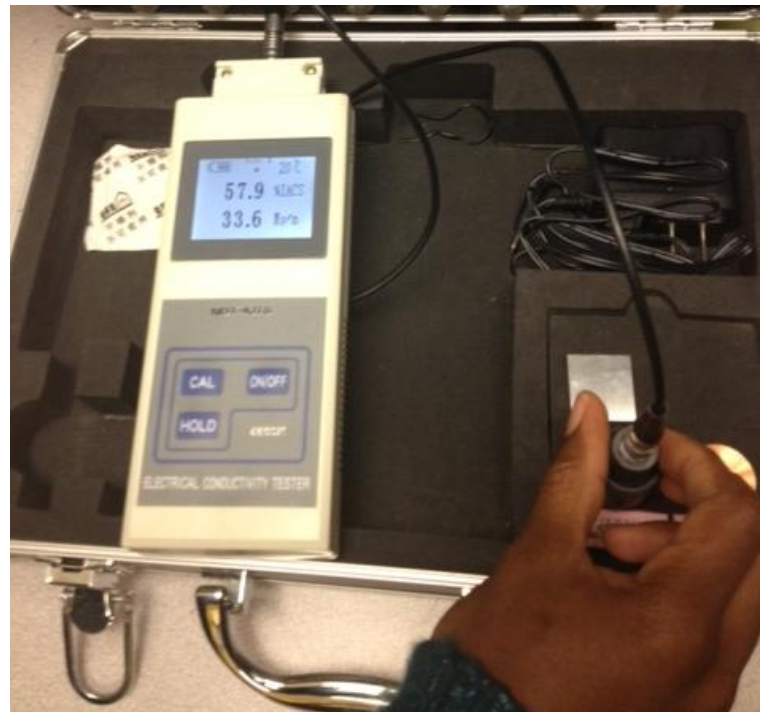


Figure 5.4: EE0021 digital electrical conductivity tester

5.4 Hardness testing

Hardness testing was conducted on the same samples used for EC testing using the Versitron Rockwell tester shown in Figure 5.5. The measurements were taken on Rockwell scale B recommended for aluminium alloys. Using a standard sample, the percent error in the measurements is 1.2% of readings. An average of ten different measurements were taken and recorded in different orientations of long-short (LS), transverse-long (TL) and transverse-short (TS) respectively.



Figure 5.5: Versitron Rockwell hardness tester

5.5 Corrosion tests

5.5.1 Exfoliation corrosion testing

Test specimens of 50×100×5 mm (in LS, TL and TS direction respectively) were cut from the as-received plates. Exfoliation tests were carried out on samples subjected to selected heat treatments, including T6, T7 and HTPP treated samples of $T_{F/Q} = 455, 425, 405$ and $370\text{ }^{\circ}\text{C}$. Figure 5.6 shows front and back view of a sample prepared for exfoliation corrosion test. Following heat treatment, samples were prepared by grinding using SiC paper followed by polishing the surface (TL direction) to 6 micron and 1 micron using diamond solution. The samples were rinsed with water and dried in air. The other surfaces apart from the one being observed were coated with paraffin wax. Exfoliation corrosion test was conducted according to ASTM G34 standard [74]. The test solution had a composition of 4.0 M of NaCl, 0.5 M of KNO_3 and 0.1 M of HNO_3 . The solution was prepared by dissolving 234 g of NaCl, 50 g of KNO_3 and 6.3 mL of conc.(70 wt%) HNO_3 in 1litre of distilled water. The solution was maintained at room temperature [74]. The samples were immersed in the test solution in a container. The test surface (transverse-long direction) was upward in horizontal direction to prevent loss of exfoliated material. The specimens were exposed for 48 hours followed by visual inspection. The exfoliation corrosion susceptibility was rated based on Table 5.3. This rating system is a modified version of the ASTM standard to better reflect the range of results obtained from this experiment.

Table 5.3: Modified exfoliation corrosion rating

CORROSION RESULT	RATING
Severe Exco	8
Moderate Exco	7
Mild Exco	6
Light Exco	5
Severe General	4
Moderate Pitting	3
Light Pitting	2
Areas Unattacked	1

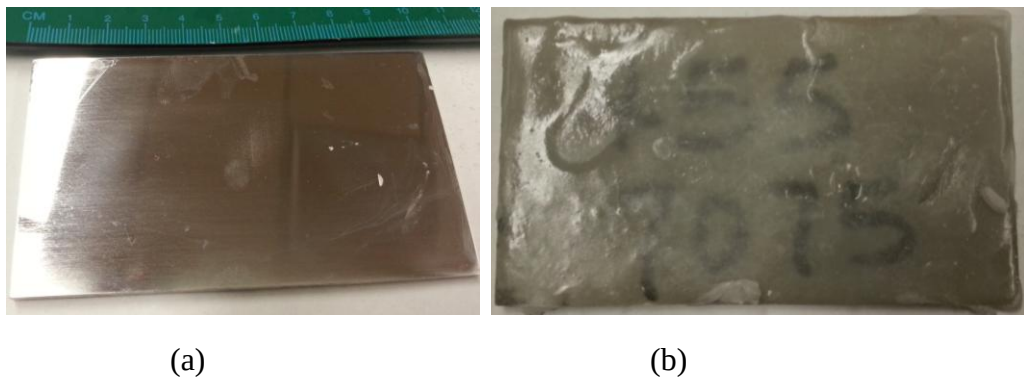


Figure 5.6: Polished plate prepared for exfoliation corrosion test showing (a) polished surface (b) unexposed parts coated with paraffin

5.5.2 Stress corrosion test (Double beam test)

Stress corrosion testing was carried out using the pre-cracked double beam (DB) method based on ASTM G168 [75]. Blanks of about 25.4×25.4×127 mm (drawing is included in appendix A) were cut from plates of 7075 and 7050 alloy respectively. Modification of ASTM standard is in the use of straight notch at 50° angle for easy machining. Heat treatment was conducted on these samples based on the procedure given for selected tempers including standard temper T6, T7 and HTPP treated samples with $T_{F/Q}$ = 455, 425, 405 and 370 °C for both 7075 and 7050 alloys. T74 (as-received) condition was also tested for the 7050 alloy. Three samples were tested for each of the conditions of 7050 alloy. For 7075 alloy, two samples were tested for each HTPP condition and one sample was used for T6 and T7 temper respectively. Final machining was carried out after heat treatment to get a smooth surface and to create a notch in the sample. The samples were machined to notch angle 50° to create a sharp tip such that the direction of crack growth was in SL (short-long) direction. After machining, the SL surface was ground using 1200 SiC paper to remove scratches. This was followed by cleaning using ultrasonic bath to remove grease and dirt.

The DB samples were tested in constant displacement condition by using steel bolts as shown in Figure 5.7. The steel bolts were mechanically loaded to create a pre-crack. The length of initial crack (a_0) was measured from the load line to the root of the crack. Load line crack opening displacement (V_0) was measured from the displacement in the crack opening at the load line. A corrosion solution of 3.62% NaCl was added to the samples in three drops, twice a day. The temperature during the test period is provided in appendix B. Measurements of crack length were taken as average of crack length on both sides of the samples at intervals by using a travelling

microscope (Model TVM-02 made by Scientific equipment and services) shown in Figure 5.8. The greatest possible error is 0.05. To get an accurate crack length reading, build-up of corrosion products were removed using a soft brush [75]. The time of taking measurements were recorded in hours. The starting stress intensity K_I as a function of crack length was determined using the relationship between stress intensity obtained from equation 1 and crack length as shown in Figure 5.9. This relationship shows the effect of the crack growth on stress intensity. The initiation of crack causes stress concentration at the crack tip. As the crack length increases, stress intensity decreases [76]:

$$K_I = \frac{vEh\sqrt{[3h(a+0.6h)^2+h^3]}}{4[(a+0.6h)^3+ah^2]} \quad (1)$$

Where:

K_I = Starting stress intensity as a function of crack length (MPa $\sqrt{m}$)

v = Crack opening displacement at load line (m)

a = Average crack length (m)

h = Specimen half height (m)

E = Young's modulus (MPa)

K_f = Final stress intensity

a_f = Final crack length

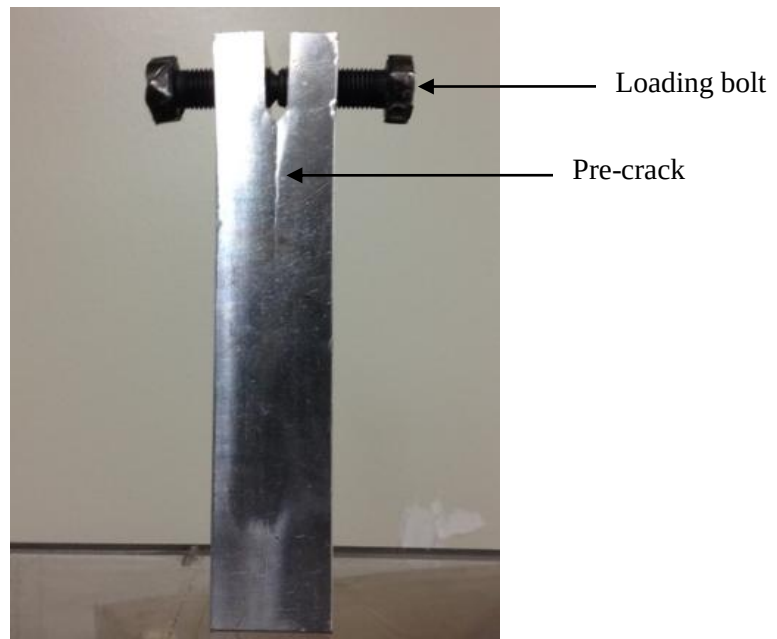


Figure 5.7: A loaded double beam sample



Figure 5.8: Travelling microscope

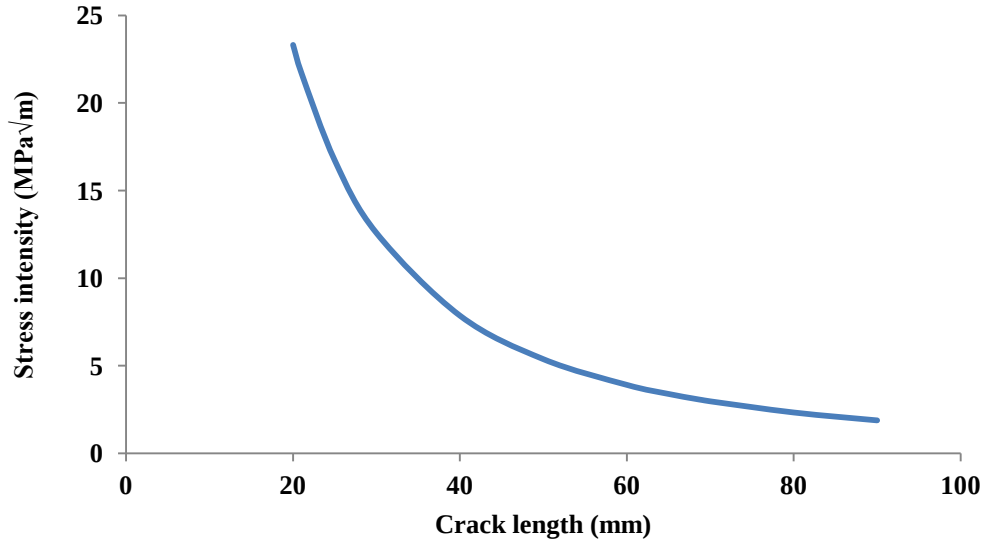


Figure 5.9: Stress intensity vs crack length as shown in equation 1

5.6 Microstructural Analysis

Optical microscopy was used to analyse the as-received materials and the exfoliation corrosion path. Samples for these analyses were prepared by:

- Cutting small blanks of about 5 mm thick from the material using Electrical Discharge Machining.
- Mounting in Phenolic resin using hydraulic mounting press.
- Grinding on SiC paper to 1200 grit size.
- Polishing with 6 and later 1 μm diamond suspension.
- Cleaning with soapy water soaked in cotton wool and rinsing in water.
- Cleaning using Ultrasonic bath for about 15 minutes followed by rinsing with water, rinsing with alcohol and drying with air.
- Samples were etched in Keller's reagent (190 ml H_2O , 5 ml HNO_3 , 3 ml HCl , 2 ml HF) by swabbing for 20 seconds.

Metallographic preparation for Transmission Electron Microscope observation involved cutting of thin foil from heat treated coupons and grinding on both sides to obtain a smooth surface on samples of about 100 μm thick. Figure 5.10 shows stages of sample preparation for TEM analysis. Samples of 3 mm diameter were punched from these samples and dimpled (to reduce the thickness at the center) to depth of about 50-60 μm (as shown in Figure 5.10 (a)). This was followed by electropolishing using an electrolyte of 25% HNO_3 and 75% methanol at 20 V, 1 A current. The temperature of the electrolyte was cooled to -40 $^{\circ}\text{C}$ using liquid nitrogen. Figure 5.10 (b) shows the hole in an electropolished sample. Two grain boundaries were selected to analyze the segregation of solute elements across grain boundary regions. Using DigitalMicrograph software, a line scan was drawn across the grain boundary and Energy-Dispersive X-Ray Spectroscopy (EDS) analysis of various points across the grain boundary was obtained using a beam diameter of 1nm for EDS analysis in Scanning Transmission Electron Microscope (STEM) mode. The same procedure was repeated on another grain boundary and compositions obtained were recorded for each point.

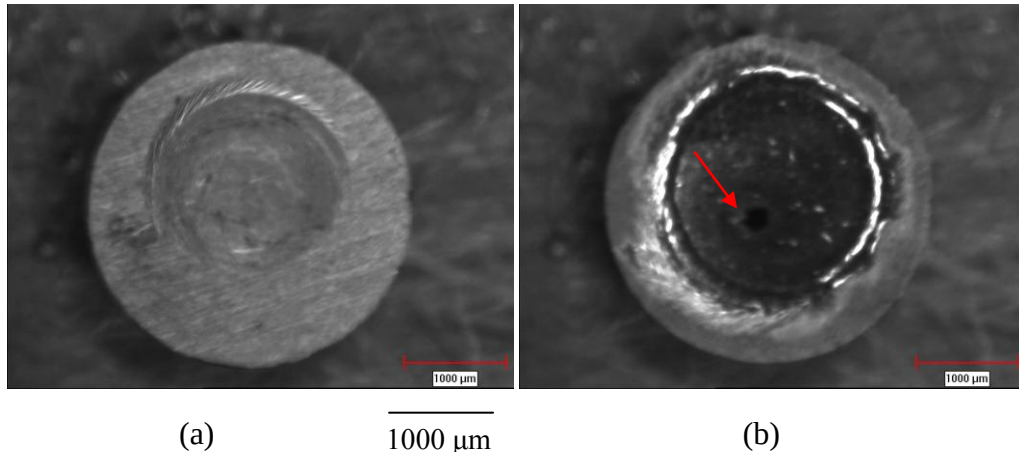


Figure 5.10: Stages of preparation of 3 mm TEM sample showing (a) dimpled surface (b) polished surface (arrow shows the hole)

CHAPTER SIX

EXPERIMENTAL RESULTS

6.1 Electrical conductivity results

Tables 6.1 and 6.2 show the electrical conductivity (EC) values measured before and after aging of 7075 and 7050 alloy respectively. EC of as-received material and standard T6 and T7 tempers are also listed in Table 6.2. Figure 6.1 compares the EC of 7075 and 7050 alloy (listed in Tables 6.1 and 6.2) in the unaged and aged condition for all the HTPP and standard T6 and T7 tempers.

As expected, a big change in EC is observed from T6 to T7 temper. It appears that aging has little effect on EC of HTPP treated samples. This indicates that EC of HTPP treated samples is determined prior to aging. However, there is gradual increase in the EC as $T_{F/Q}$ temperature decreases. In fact, 7050 alloy heat treated to HTPP at $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ has the same EC as the T7 temper. The results for both alloys shows a sharp increase in EC for HTPP treated alloys at $T_{F/Q} = 445$ and $415\text{ }^{\circ}\text{C}$ in both the aged and unaged conditions. The cause of these changes requires more investigation.

Table 6.1: Electrical conductivity results of 7075 alloy before and after aging

HEAT TREATMENT (7075 ALLOY)	ELECTRICAL CONDUCTIVITY RESULT (%IACS)	
	Before aging	After aging
T651 (as-received)	32.2	33.1
T6	32.2	32.5
T7	32.2	39.3
T _{F/Q} = 455 °C	32.7	32.6
T _{F/Q} = 445 °C	32.5	32.7
T _{F/Q} = 435 °C	32.9	33.1
T _{F/Q} = 425 °C	34.1	34.0
T _{F/Q} = 415 °C	34.1	34.3
T _{F/Q} = 405 °C	35.8	36.3
T _{F/Q} = 395 °C	35.8	36.8
T _{F/Q} = 385 °C	36.5	37.0
T _{F/Q} = 375 °C	37.3	37.5
T _{F/Q} = 370 °C	37.9	38.3

Table 6.2: Electrical conductivity results of 7050 alloy before and after aging

HEAT TREATMENT (7050 ALLOY)	ELECTRICAL CONDUCTIVITY RESULT (%IACS)	
	Before aging	After aging
T74 (as-received)	32.4	42.1
T6	32.4	32.6
T7	32.4	40.2
T_{F/Q} = 455 °C	32.5	32.7
T_{F/Q} = 445 °C	32.4	33.0
T_{F/Q} = 435 °C	34.8	34.4
T_{F/Q} = 425 °C	35.6	35.1
T_{F/Q} = 415 °C	35.6	35.3
T_{F/Q} = 405 °C	37.0	37.2
T_{F/Q} = 395 °C	37.0	37.8
T_{F/Q} = 385 °C	37.5	38.3
T_{F/Q} = 375 °C	38.4	39.3
T_{F/Q} = 370 °C	38.7	40.1

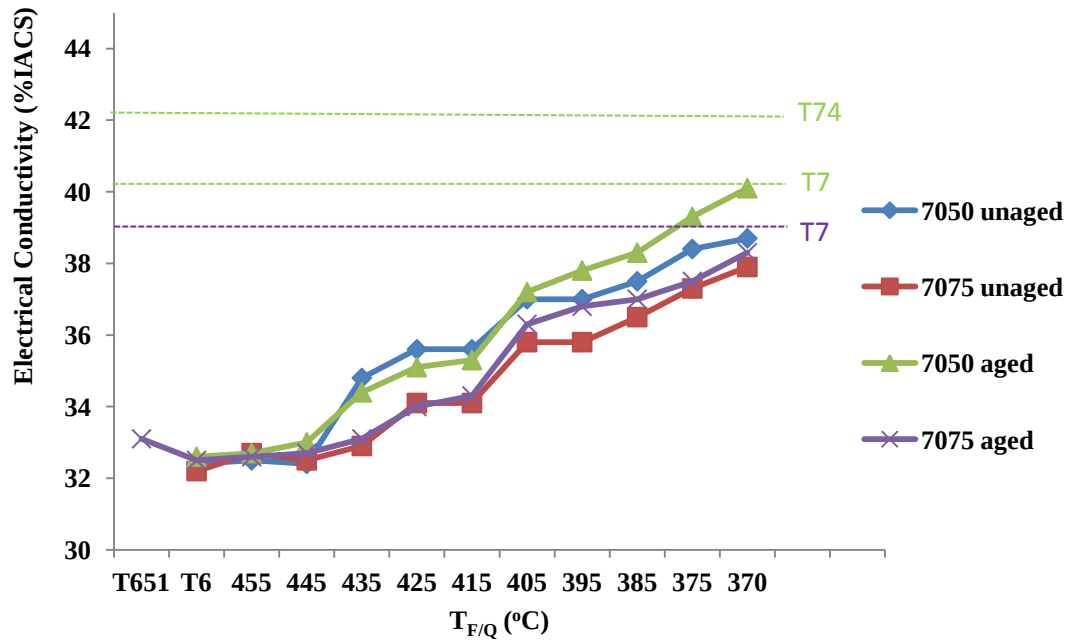


Figure 6.1: Effect of heat treatment on electrical conductivity of 7075 and 7050 alloys. Green broken lines indicate as-received T74 and standard T7 temper of 7050 alloy. Purple line indicates standard T7 temper of 7075 alloy

6.2 Hardness results

Tables 6.3-6.6 list the hardness measurements of 7075 and 7050 alloys before and after aging. In tables 6.4 and 6.6, hardness of the as-received material and standard tempers T6 and T7 are also listed. Figure 6.2 and 6.3 shows the effect of HTPP heat treatment on hardness of 7075 and 7050 alloy respectively.

It is evident from these results that there is loss of strength in the alloys before aging was applied. Compared to T6 temper, about 7% loss of hardness in T7 temper can be observed in both alloys after aging. This result is comparable to 10-15% loss of yield strength in T7 temper reported in the literature [10]. The results show that subsequent

aging of HTPP treated samples increases the hardness in both alloys. However, hardness decreases as the $T_{F/Q}$ temperature decreases.

Table 6.3: Hardness result (HRB) of 7075 alloy before aging

Heat Treatment	LT	LS	TS
T6	36.0	36.0	36.0
$T_{F/Q} = 455^{\circ}\text{C}$	45.1	47.0	47.5
$T_{F/Q} = 445^{\circ}\text{C}$	44.8	45.3	49.1
$T_{F/Q} = 435^{\circ}\text{C}$	34.7	39.2	37.0
$T_{F/Q} = 425^{\circ}\text{C}$	33.6	36.2	36.5
$T_{F/Q} = 415^{\circ}\text{C}$	32.8	35.6	35.0
$T_{F/Q} = 405^{\circ}\text{C}$	21.4	25.5	24.2
$T_{F/Q} = 395^{\circ}\text{C}$	21.2	23.8	23.2
$T_{F/Q} = 385^{\circ}\text{C}$	18.0	21.3	23.8
$T_{F/Q} = 375^{\circ}\text{C}$	14.0	14.0	14.3
$T_{F/Q} = 370^{\circ}\text{C}$	14.0	14.6	16.7

Table 6.4: Hardness result (HRB) of 7075 alloy after aging

Heat Treatment	LT	LS	TS
T651 (as-received)	89.2	89.5	89.3
T7	83.6	82.4	81.1
T6	90.0	89.0	90.0
T_{F/Q} = 455 °C	90.7	91.0	90.7
T_{F/Q} = 445 °C	91.3	91.2	90.2
T_{F/Q} = 435 °C	90.6	90.7	91.0
T_{F/Q} = 425 °C	87.2	89.1	88.8
T_{F/Q} = 415 °C	88.0	88.0	88.3
T_{F/Q} = 405 °C	81.0	85.1	83.8
T_{F/Q} = 395 °C	73.8	74.0	75.0
T_{F/Q} = 385 °C	69.2	72.5	73.6
T_{F/Q} = 375 °C	64.8	67.6	66.4
T_{F/Q} = 370 °C	63.0	62.7	65.0

Table 6.5: Hardness result (HRB) of 7050 alloy before aging

Heat Treatment	LT	LS	TS
T6	32.0	34.5	32.0
T_{F/Q} = 455 °C	40.5	45.0	47.1
T_{F/Q} = 445 °C	39.3	40.7	42.7
T_{F/Q} = 435 °C	22.9	26.2	25.0
T_{F/Q} = 425 °C	20.6	23.5	21.8
T_{F/Q} = 415 °C	23.0	23.9	22.0
T_{F/Q} = 405 °C	17.2	16.5	16.0
T_{F/Q} = 395 °C	14.0	14.0	14.0
T_{F/Q} = 385 °C	14.0	14.0	14.0
T_{F/Q} = 375 °C	14.0	14.0	14.0
T_{F/Q} = 370 °C	14.0	14.0	14.0

Table 6.6: Hardness result (HRB) of 7050 alloy after aging

Heat treatment	LT	LS	TS
T74 (as-received)	84.5	83.8	85.4
T7	84.0	86.0	85.0
T6	90.0	90.0	90.0
T_{F/Q} = 455 °C	91.0	91.0	91.8
T_{F/Q} = 445 °C	90.6	91.5	91.3
T_{F/Q} = 435 °C	90.1	90.4	90.0
T_{F/Q} = 425 °C	89.0	89.4	89.0
T_{F/Q} = 415 °C	89.1	89.6	89.1
T_{F/Q} = 405 °C	87.2	88.3	87.5
T_{F/Q} = 395 °C	85.1	86.0	86.0
T_{F/Q} = 385 °C	84.0	83.0	84.4
T_{F/Q} = 375 °C	81.8	84.0	83.6
T_{F/Q} = 370 °C	67.9	75.7	74.0

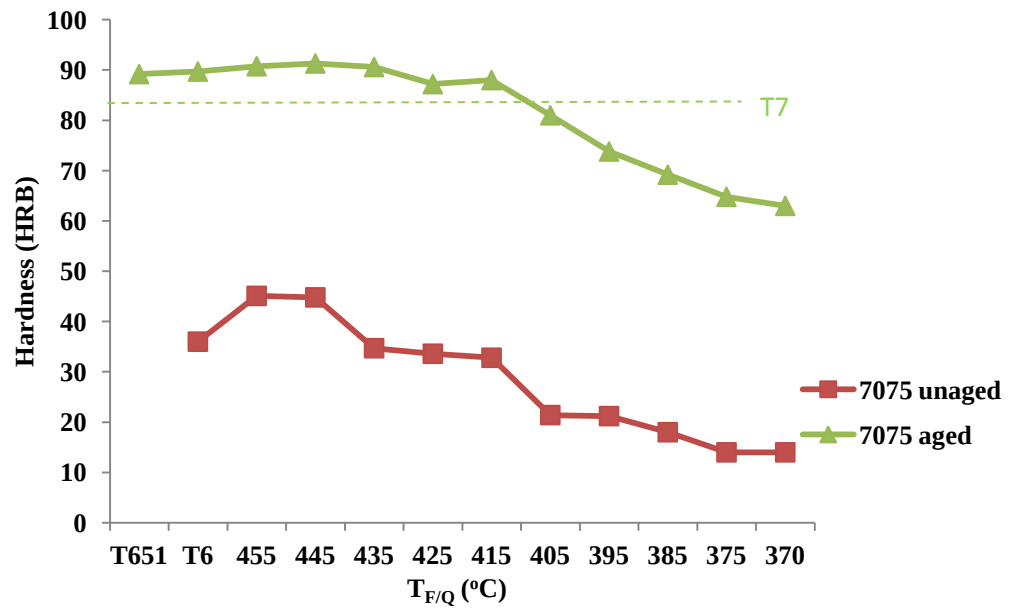


Figure 6.2: Effect of HTPP heat treatment on hardness of 7075 alloy. The green broken line shows hardness obtained for T7 temper

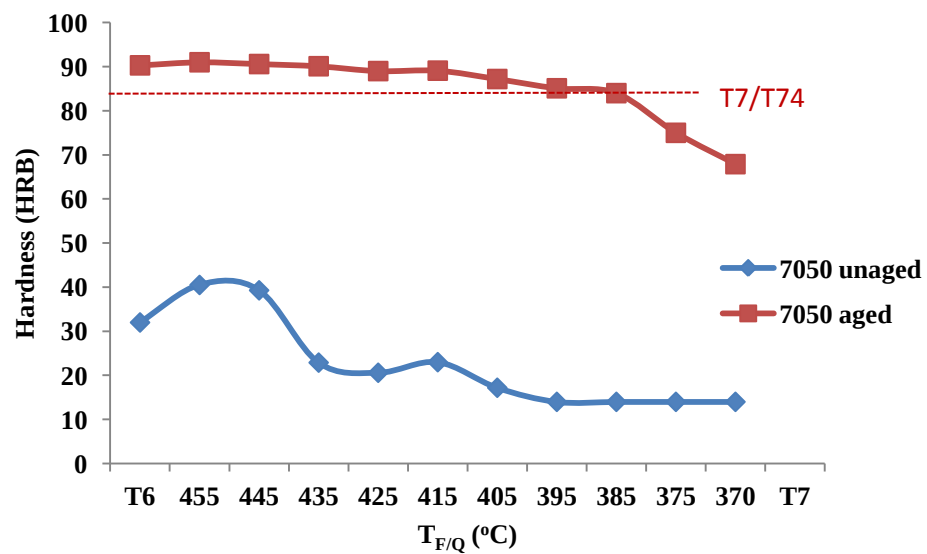


Figure 6.3: Effect of HTPP heat treatment on hardness of 7050 alloy. The red broken line shows hardness obtained for T74 and T7 temper

6.3 Corrosion test results

6.3.1 Exfoliation corrosion result

The samples used for exfoliation corrosion tests are shown in Figures 6.4 and 6.5 for 7075 and 7050 alloy respectively. The results presented in these Figures show one sample out of the two samples tested for each condition. The extent of exfoliation corrosion was rated based on the surface appearance of samples after exposure to exfoliation corrosion solution for 48 hours as shown in Table 6.7.

It is evident that both alloys are susceptible to exfoliation corrosion in almost all the conditions. From Figure 6.4 and 6.5 (a) and (b), T6 and $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ samples showed severe exfoliation and loss of material across the entire surface. Both alloys showed light exfoliation in $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ conditions. From Figure 6.4 and 6.5 (d) and (e), it appears that samples treated to $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ were more susceptible to exfoliation corrosion than $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ conditions. The cause of this variation in EXCO resistance despite lower $T_{F/Q}$ should be further investigated. From Figure 6.4 and 6.5 (e), $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ samples showed general pitting across the surface of the samples. Figure 6.4 and 6.5 (f) shows that T7 temper is highly resistant to exfoliation corrosion. These results show that, samples treated to HTPP at $T_{F/Q} = 425$ and $370\text{ }^{\circ}\text{C}$ showed better resistant to exfoliation corrosion compared to T6 and other HTPP conditions. Although, none of the HTPP treated samples achieved as good corrosion resistance as T7 temper, samples treated to $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ showed comparable exfoliation corrosion resistance to the T7 temper. The $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ samples showed pitting corrosion while few pits were observed in T7 temper. The presence of pits has been related to increase in EXCO resistance in RRA treated 7150 alloy [62].

Figure 6.6 shows the optical micrograph of an exfoliated sample. It is evident from the image that exfoliation corrosion is an intergranular corrosion that occurs preferentially at the grain boundaries. The disintegration of grains observed in the image also shows the leafing effect of corrosion products in exfoliation corrosion. The pitting corrosion observed in T7 samples and HTPP samples treated to $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ of both alloys show that there is a direct relationship between exfoliation corrosion and SCC.

Table 6.7: Exfoliation corrosion (EXCO) rating of heat treated samples

Temper	7050 alloy Electrical Conductivity (%IACS)	7075 alloy Electrical Conductivity (%IACS)	7050 EXCO rating	7075 EXCO rating
T6	32.6	32.5	8	8
$T_{F/Q} = 455\text{ }^{\circ}\text{C}$	32.7	32.6	7	8
$T_{F/Q} = 425\text{ }^{\circ}\text{C}$	35.1	34.0	5	5
$T_{F/Q} = 405\text{ }^{\circ}\text{C}$	37.2	36.3	6	7
$T_{F/Q} = 370\text{ }^{\circ}\text{C}$	40.1	38.3	4	4
T7	40.2	39.3	1	1



Figure 6.4 (a): EXCO sample of 7075 alloy in T6 temper showing severe exfoliation and disintegration of the sample surface



Figure 6.4 (b): EXCO sample of HTPP treated 7075 alloy at $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ showing severe exfoliation and disintegration of sample surface



Figure 6.4 (c): EXCO sample of HTPP treated 7075 alloy at $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ showing light exfoliation on sample surface



Figure 6.4 (d): EXCO sample of HTPP treated 7075 alloy at $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ showing moderate exfoliation on sample surface



10 cm

Figure 6.4 (e): EXCO sample of HTPP treated 7075 alloy at $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ showing severe general pitting on sample surface



10 cm

Figure 6.4 (f): EXCO sample of 7075 alloy in T7 temper showing some pitting and unattacked areas on the sample surface

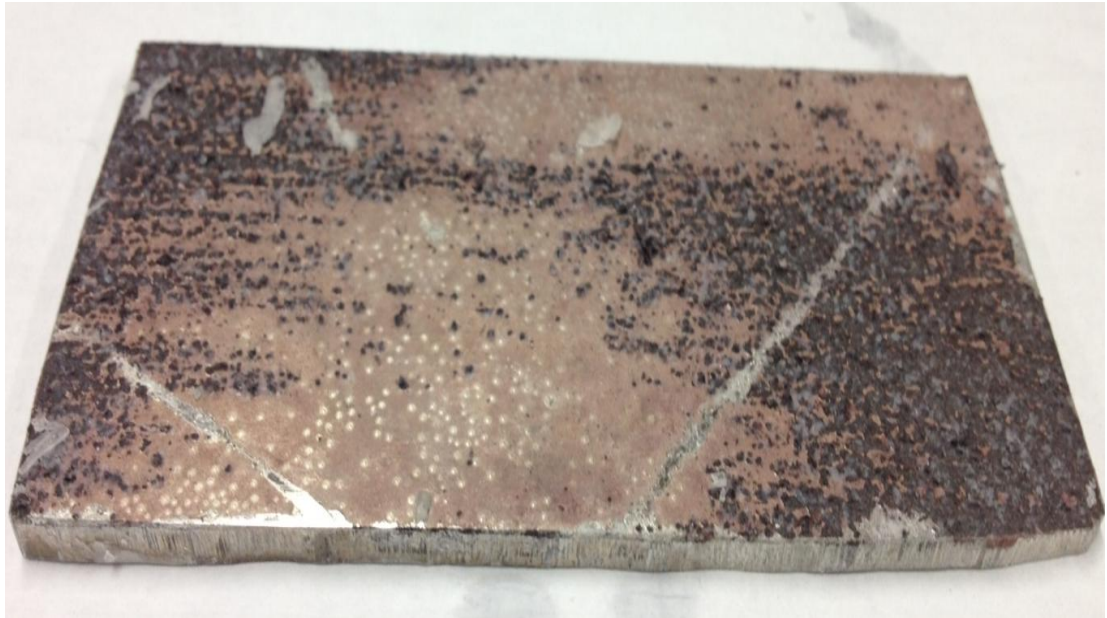
Figure 6.4: EXCO behaviour of 7075 samples in (a) T6 (b) $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ (c) $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ (d) $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ (e) $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ (f) T7 temper



Figure 6.5 (a): EXCO sample of 7050 alloy in T6 temper showing severe exfoliation of sample surface



Figure 6.5 (b): EXCO sample of HTPP treated 7050 alloy at $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ showing severe exfoliation corrosion on sample surface



10 cm

Figure 6.5 (c): EXCO sample of HTPP treated 7050 alloy at $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ showing light exfoliation of sample surface



10 cm

Figure 6.5 (d): EXCO sample of HTPP treated 7050 alloy at $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ showing mild exfoliation of sample surface



10 cm

Figure 6.5 (e): EXCO sample of HTPP treated 7050 alloy at $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ showing some pits and areas unattacked on sample surface



10 cm

Figure 6.5 (f): EXCO sample of 7050 alloy in T7 temper showing some pits and areas unattacked on sample surface

Figure 6.5: EXCO samples of 7050 alloy in (a) T6 (b) $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ (c) $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ (d) $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ (e) $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ (f) T7 temper

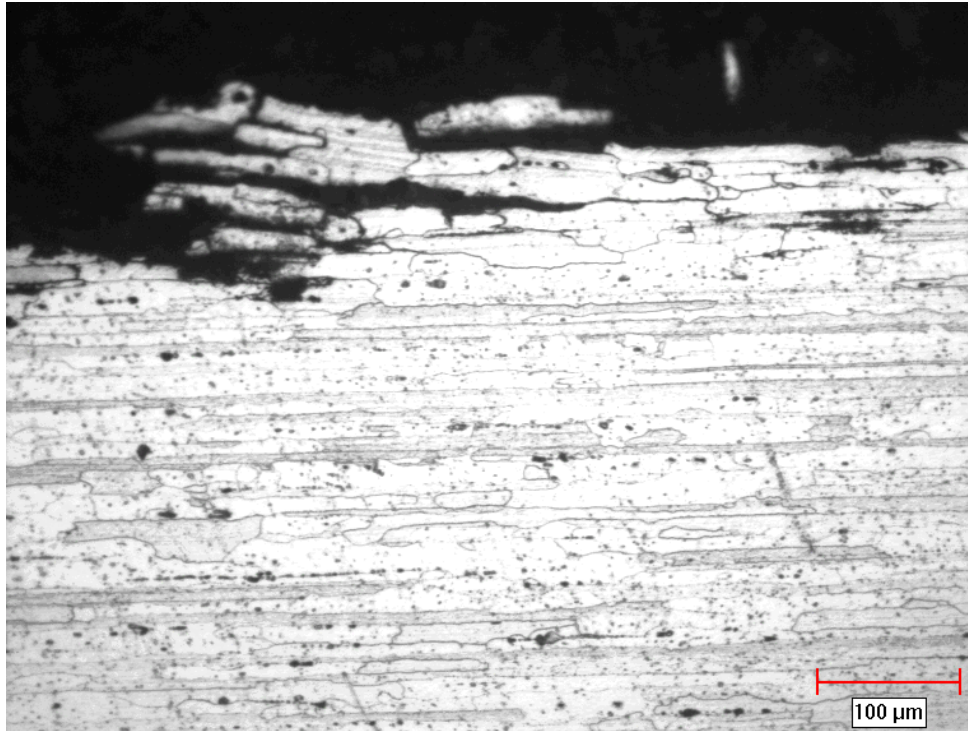


Figure 6.6: Optical micrograph of a section of an exfoliated sample showing intergranular corrosion

6.3.2 Stress Corrosion Cracking results

Tables 6.8 and 6.9 summarize SCC results obtained from double beam samples of 7075 and 7050 alloys respectively. The SCC behaviour of heat treated samples was measured in terms of crack length versus time. The data for these results are in appendix C. Since more than one samples were tested, Figure 6.7 compares change in crack length in each sample of $T_{F/Q} = 455$ and 370 °C 7075 alloy. This Figure shows the consistency and similarity in trends for results obtained from samples with the same heat treatment condition.

Table 6.8: Summary of results obtained from 7075 double beam samples

Heat Treatment	Time (h)	Crack opening displacement V_o (m)	Initial crack length a_o (m)	Final crack length a_f (m)	Initial stress intensity K_I (MPa $\sqrt{m}$)	Final stress intensity K_f (MPa $\sqrt{m}$)
T6	746	0.0004	0.018	0.117	24.18	1.01
T7	1346	0.0003	0.016	0.024	27.12	15.6
$T_{F/Q} = 455^\circ\text{C}$	746	0.0004	0.018	0.117	23.41	1.01
$T_{F/Q} = 425^\circ\text{C}$	746	0.0004	0.016	0.116	24.19	0.87
$T_{F/Q} = 405^\circ\text{C}$	1346	0.0004	0.018	0.116	24.37	1.00
$T_{F/Q} = 370^\circ\text{C}$	1446	0.0004	0.016	0.043	27.61	6.38

Table 6.9: Results obtained from 7050 double beam samples

Heat Treatment	Time (h)	Crack opening displacement V_o (m)	Initial crack length a_o (m)	Final crack length a_f (m)	Initial stress intensity K_I (MPa $\sqrt{m}$)	Final stress intensity K_f (MPa $\sqrt{m}$)
T6	746	0.0003	0.016	0.117	23.91	0.87
T7	1346	0.0003	0.016	0.024	26.93	14.54
T74	1346	0.0003	0.013	0.014	28.34	24.96
$T_{F/Q} = 455^\circ\text{C}$	1146	0.0004	0.018	0.116	23.89	1.03
$T_{F/Q} = 425^\circ\text{C}$	1346	0.0004	0.018	0.111	24.50	1.25
$T_{F/Q} = 405^\circ\text{C}$	1346	0.0003	0.015	0.096	24.28	1.39
$T_{F/Q} = 370^\circ\text{C}$	1346	0.0004	0.018	0.069	24.93	2.61

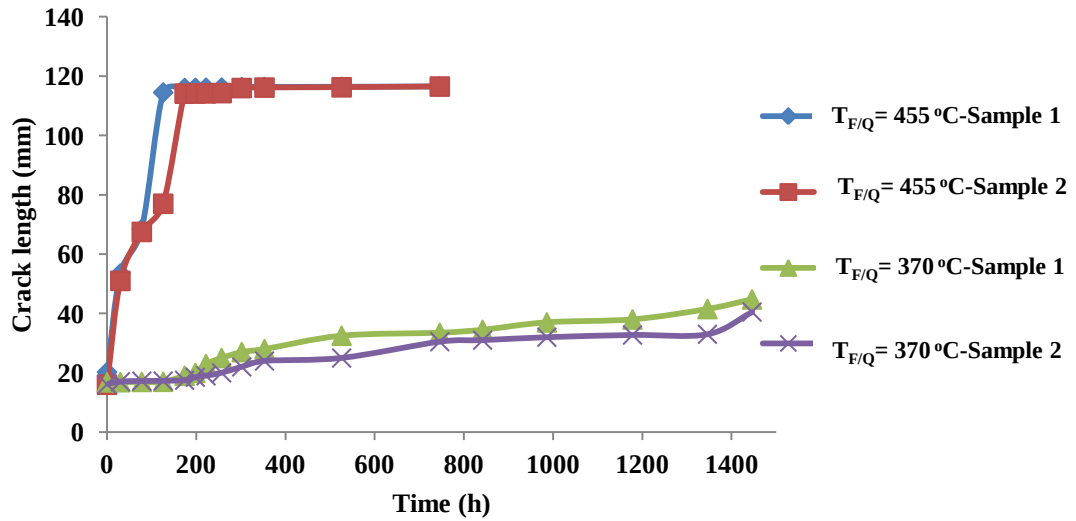


Figure 6.7: Comparism of crack length with time in two SCC samples for each of 7075 alloy treated at HTPP condition $T_{F/Q} = 455$ and 370 °C

Figures 6.8 and 6.9 show the effect of heat treatment on SCC resistance (of 7075 and 7050 alloys respectively) measured in terms of the crack length at time intervals. The results show that T6 temper is very susceptible to SCC while T7 is very resistant. The results show that HTPP improved the resistance to SCC in all the conditions tested. This is shown by the gradual decrease in the crack length as $T_{F/Q}$ decreases. It is evident from these results that HTPP heat treatment affects SCC resistance of both 7075 and 7050 alloy.

Figure 6.10 and 6.11 show the results obtained from crack growth rate (da/dt or V) versus stress intensity (K) for each of the tested condition in 7075 and 7050 alloy respectively. Data for all the samples and conditions tested are included in appendix C. The plots show the stages of SCC during the test. Figures 6.12 and 6.13 show the summary of V-K plot of all the tested conditions for 7075 and 7050 alloy

respectively. The results show that the corrosion rate decreased as the $T_{F/Q}$ decreased. This is shown in the decrease in the plateau velocity and threshold stress intensity as $T_{F/Q}$ decreases as shown in Figures 6.12 and 6.13. This shows that HTPP can improve SCC resistance in 7075 and 7050 alloys. In Figure 6.12, it appears that at $T_{F/Q} = 370$ °C and T7 conditions, the stress corrosion cracking of 7075 alloy remained in stage II. This shows that $T_{F/Q} = 370$ °C and T7 conditions are highly resistant to SCC. Figure 6.13 shows that similar trend of decrease in the plateau velocity and threshold stress intensity as $T_{F/Q}$ decreases was obtained in 7050 alloy. These results show that compared to T6 temper HTPP can achieve increase in SCC resistance but, none of the HTPP conditions achieved better SCC resistance compared to T7 and T74 tempers. From Figure 6.13, it appears that T74 temper has better SCC resistance compared to T7. The high SCC resistance in T74 temper can be attributed to the effect of cold work after heat treatment.

Generally, it is believed that 7050 alloy has higher SCC resistance than 7075 alloy. This is observed in all the conditions tested except for HTPP treated sample at $T_{F/Q} = 370$ °C. Figure 6.14 compares V-K plot of 7075 and 7050 alloys at $T_{F/Q} = 370$ °C condition. It is noteworthy that at $T_{F/Q} = 370$ °C, 7050 alloy showed a lesser SCC resistance compared to 7075 alloy. Similar observation is noticed in results shown in Figure 6.8, 6.9, 6.12 and 6.13. By comparing the corrosion results at $T_{F/Q} = 370$ °C to EC results in Figure 6.1, 7075 alloy has lesser EC compared to 7050 alloy. This shows that other factors are involved in determining SCC behaviour of different alloys. Further investigation is required to explain this anomaly in SCC behaviour of 7050 versus 7075 alloy.

The SCC behaviour observed in this research is shows similar trend to previous studies in the literature. By comparing this result to result obtained by Rajan et al for

SCC behaviour of 7075-T651 temper and Huang et al for 7055 at $T_{F/Q} = 455\text{ }^{\circ}\text{C}$, similar trend of increase in SCC resistance was observed in HTPP treated condition compared to the T651 temper [24, 28]. However, it appears that results obtained from this study show that the corrosion rate is faster compared to previous studies. The faster corrosion rate in the present work can be attributed to higher humidity of the environment and higher concentration of salt in the solution used in this study.

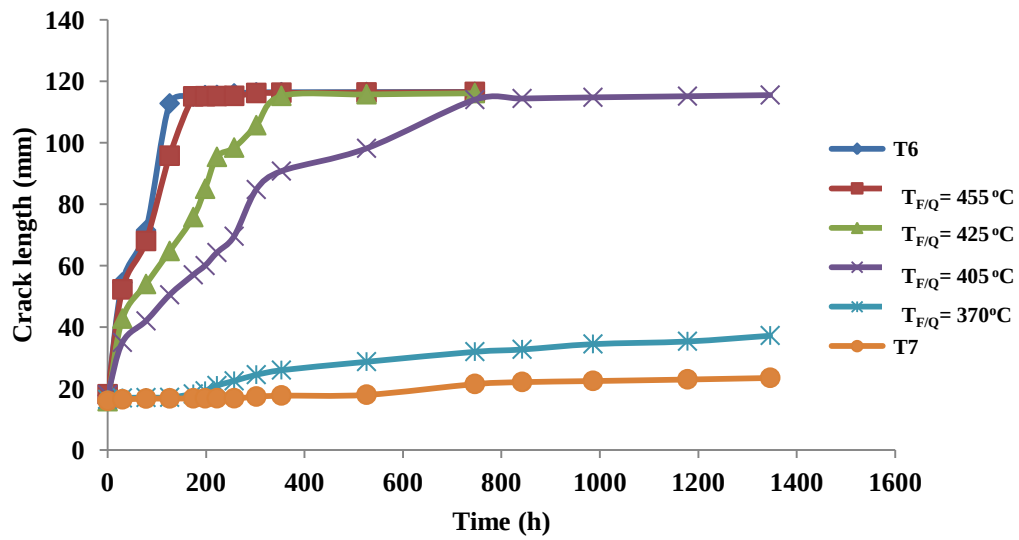


Figure 6.8: Average crack length with time of 7075 double beam samples

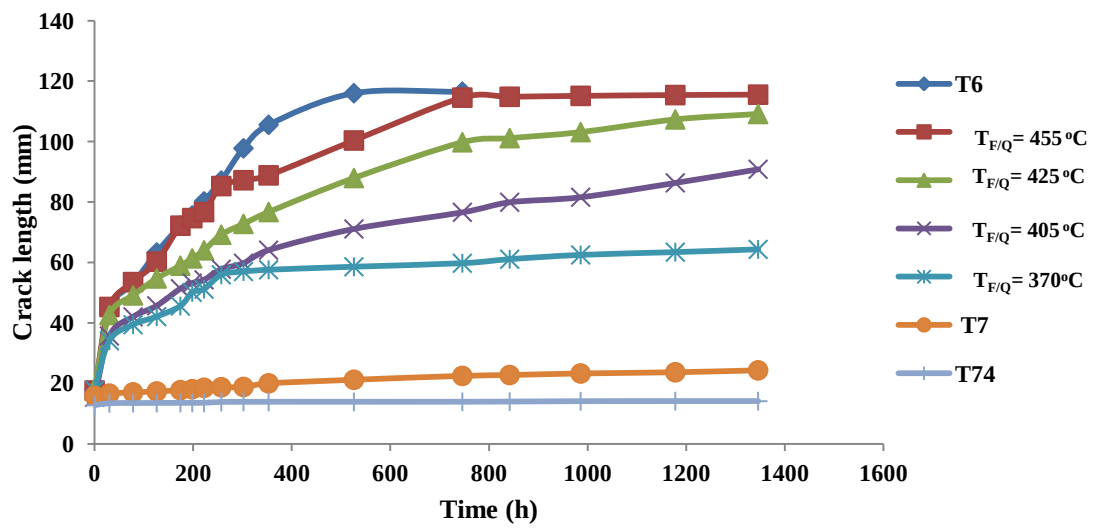


Figure 6.9: Average crack length with time of 7050 double beam samples

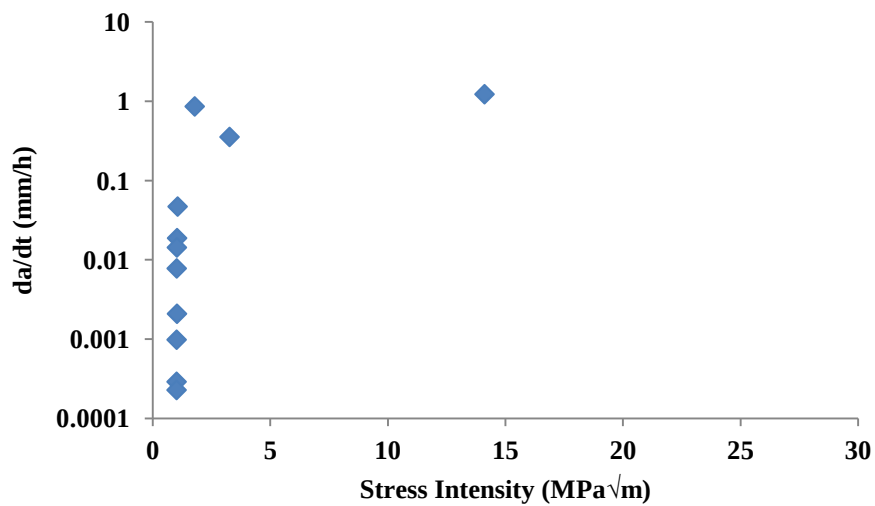


Figure 6.10 (a): Crack growth velocity vs stress intensity of 7075 alloy in T6 temper

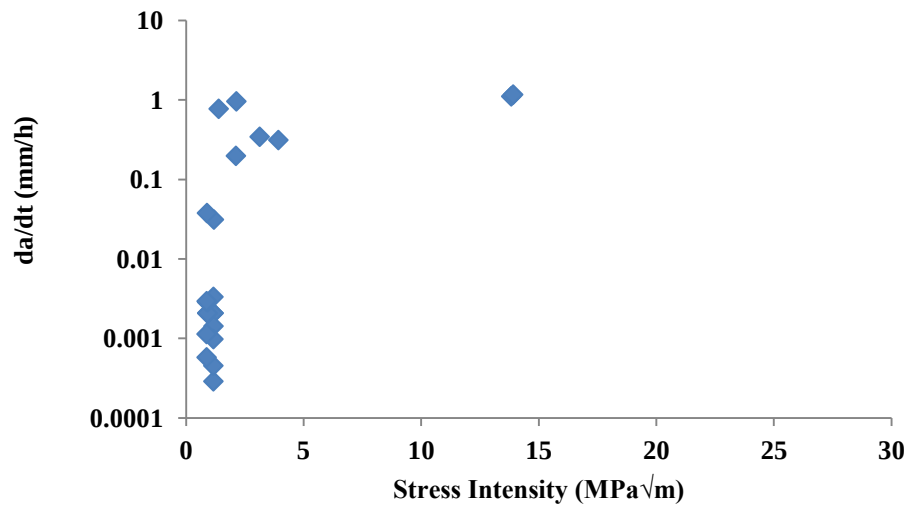


Figure 6.10 (b): Crack growth velocity vs stress intensity of HTPP treated 7075 alloy at $T_{F/Q} = 455\text{ }^{\circ}\text{C}$

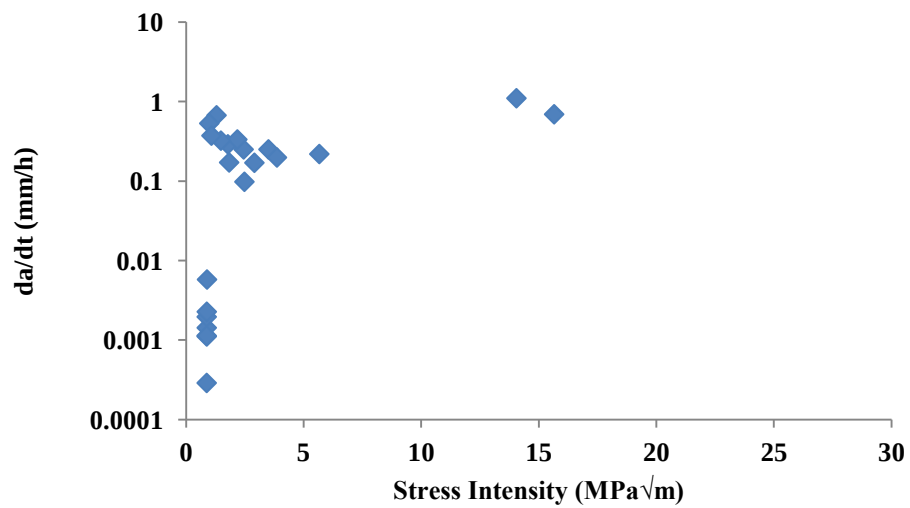


Figure 6.10 (c): Crack growth velocity vs stress intensity of HTPP treated 7075 alloy at $T_{F/Q} = 425\text{ }^{\circ}\text{C}$

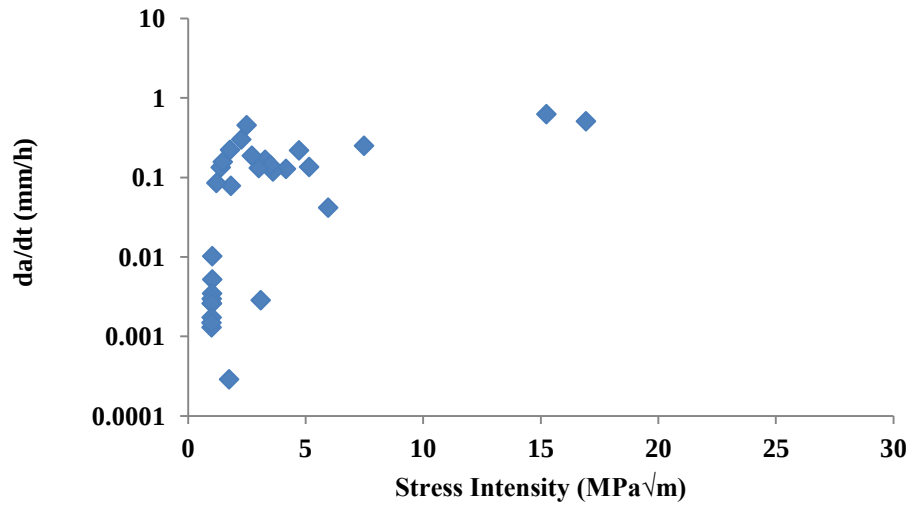


Figure 6.10 (d): Crack growth velocity vs stress intensity of HTPP treated 7075 alloy at $T_{F/Q} = 405^\circ\text{C}$

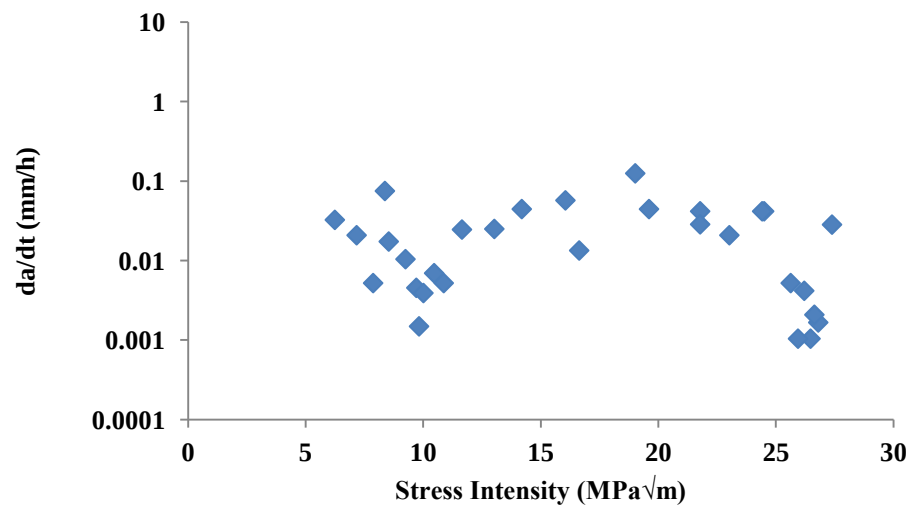


Figure 6.10 (e): Crack growth velocity vs stress intensity of HTPP treated 7075 alloy at $T_{F/Q} = 370^\circ\text{C}$

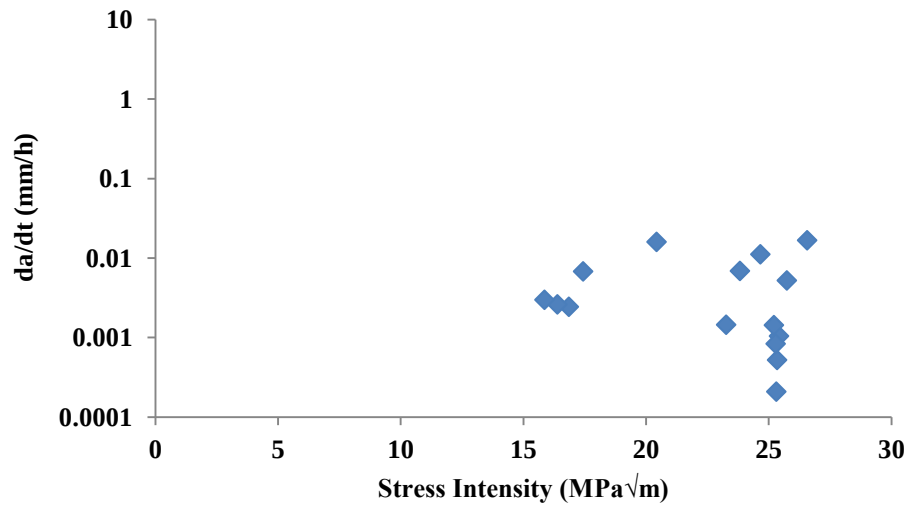


Figure 6.10 (f): Crack growth velocity vs stress intensity of 7075 alloy in T7 temper

Figure 6.10: Crack growth velocity vs stress intensity of 7075 samples showing (a) T6 (b) $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ (c) $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ (d) $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ (e) $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ (f) T7

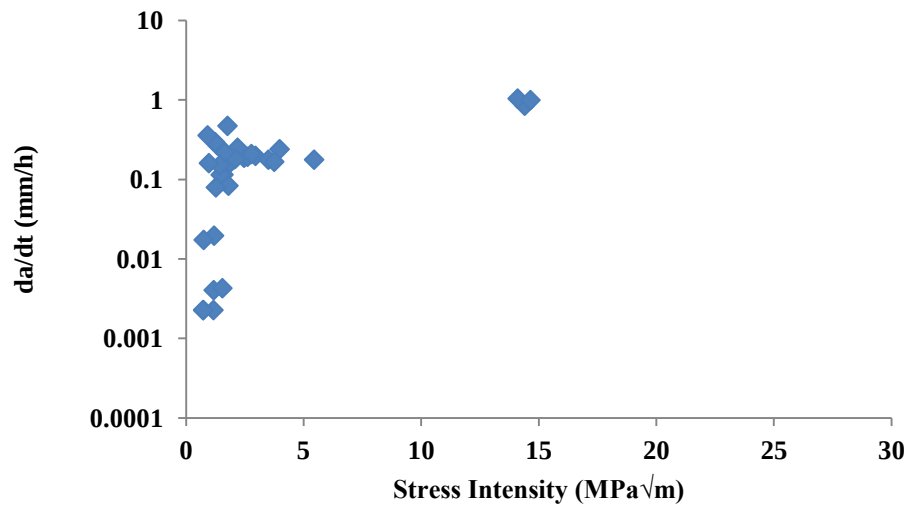


Figure 6.11 (a): Crack growth velocity vs stress intensity of 7050 alloy in T6 temper

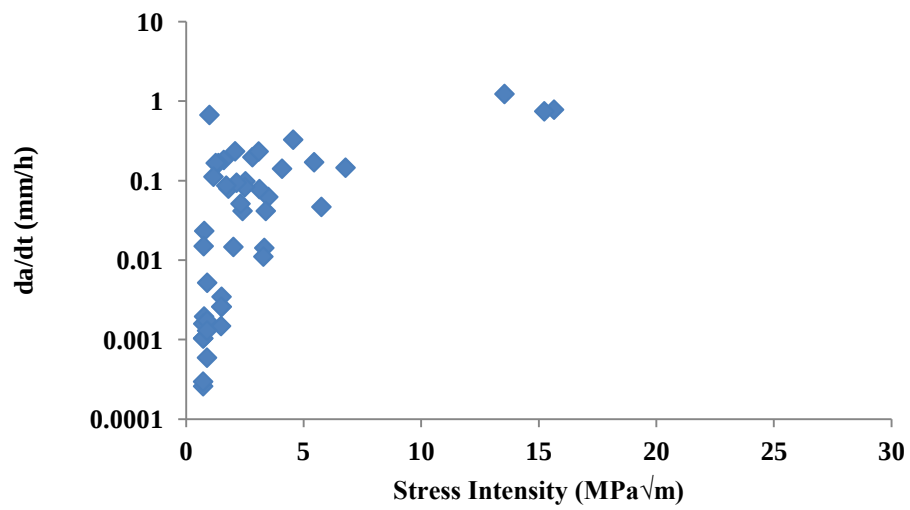


Figure 6.11 (b): Crack growth velocity vs stress intensity of HTPP treated 7050 alloy at $T_{F/Q} = 455$ °C

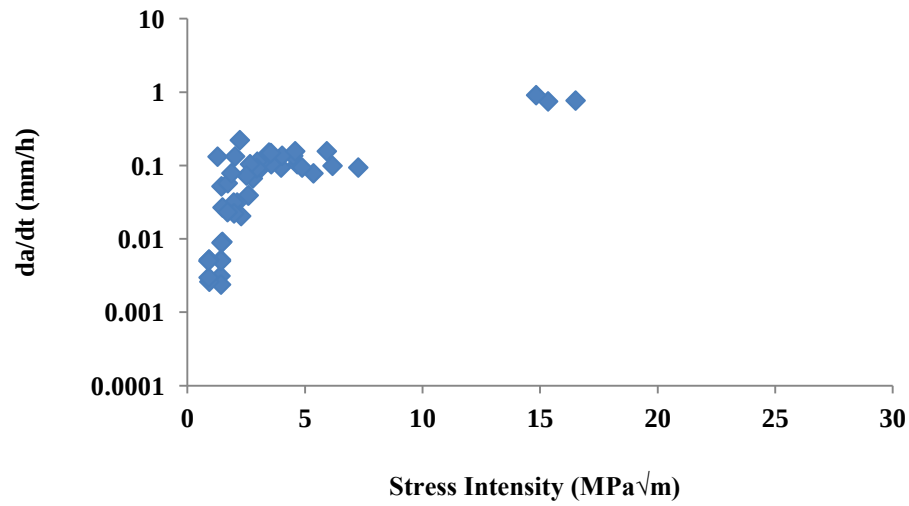


Figure 6.11 (c): Crack growth velocity vs stress intensity of HTPP treated 7050 alloy at $T_{F/Q} = 425\text{ }^{\circ}\text{C}$

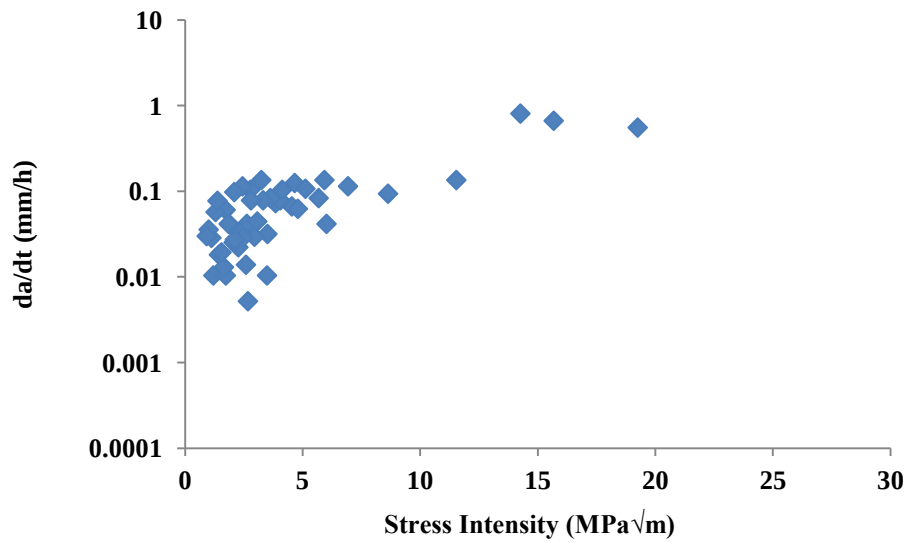


Figure 6.11 (d): Crack growth velocity vs stress intensity of HTPP treated 7050 alloy at $T_{F/Q} = 405\text{ }^{\circ}\text{C}$

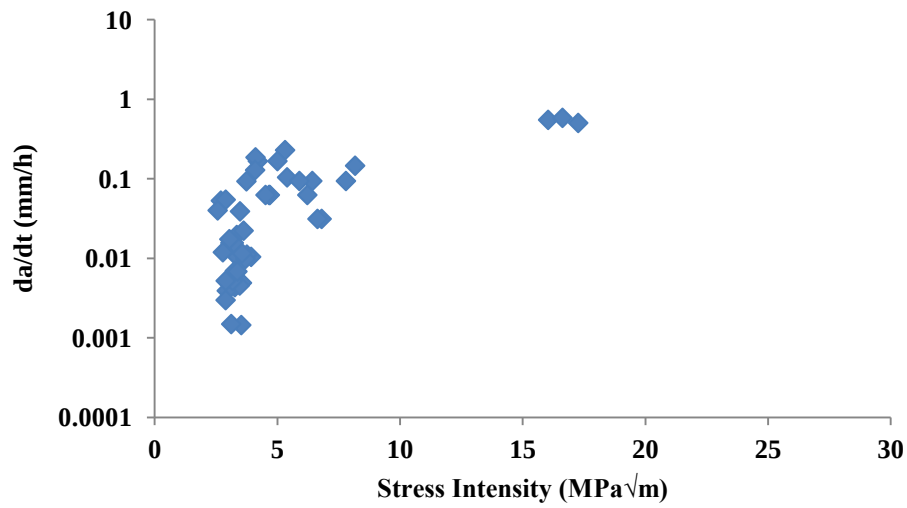


Figure 6.11 (e): Crack growth velocity vs stress intensity of HTPP treated 7050 alloy at $T_{F/Q} = 370^\circ\text{C}$

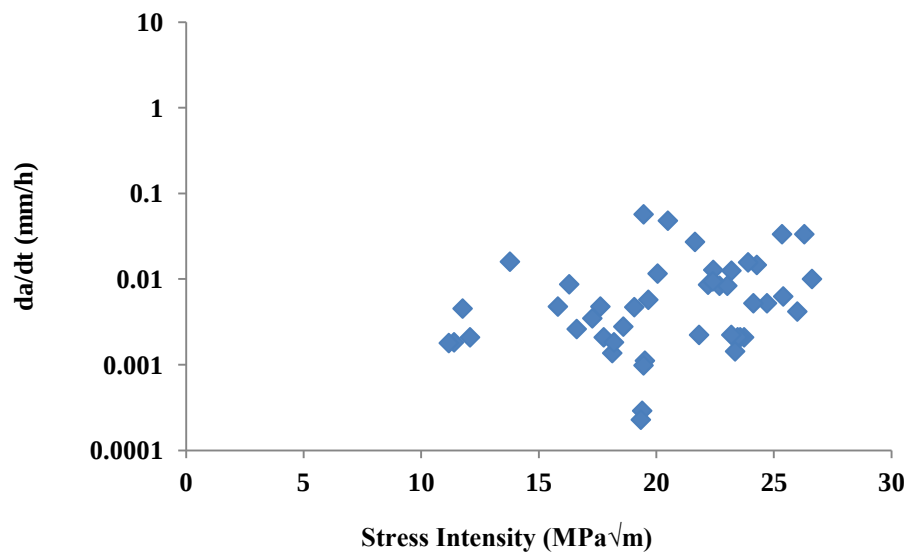


Figure 6.11 (f): Crack growth velocity vs stress intensity of 7050 alloy in T7 temper

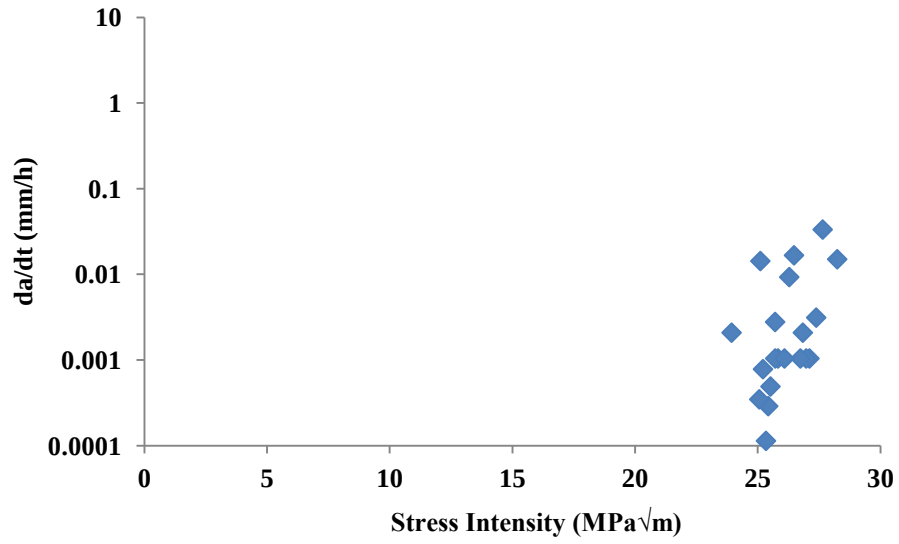


Figure 6.11 (g): Crack growth velocity vs stress intensity of 7050 alloy in T74

Figure 6.11: Crack growth velocity vs stress intensity of 7050 samples showing (a) T6 (b) $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ (c) $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ (d) $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ (e) $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ (f) T7 (g) T74

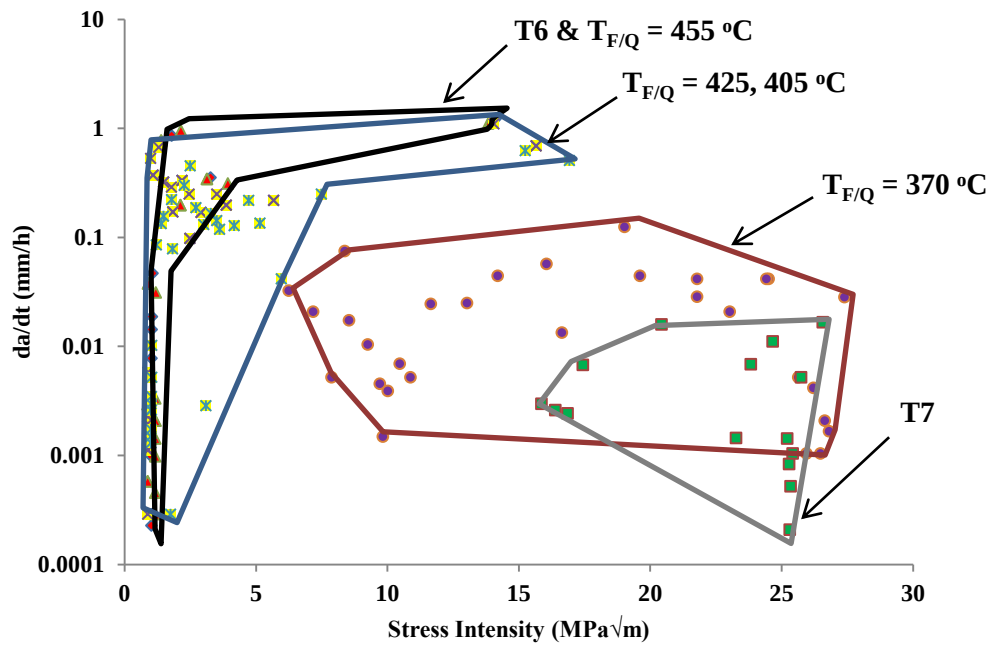


Figure 6.12: Summary of V-K plot for 7075 alloy

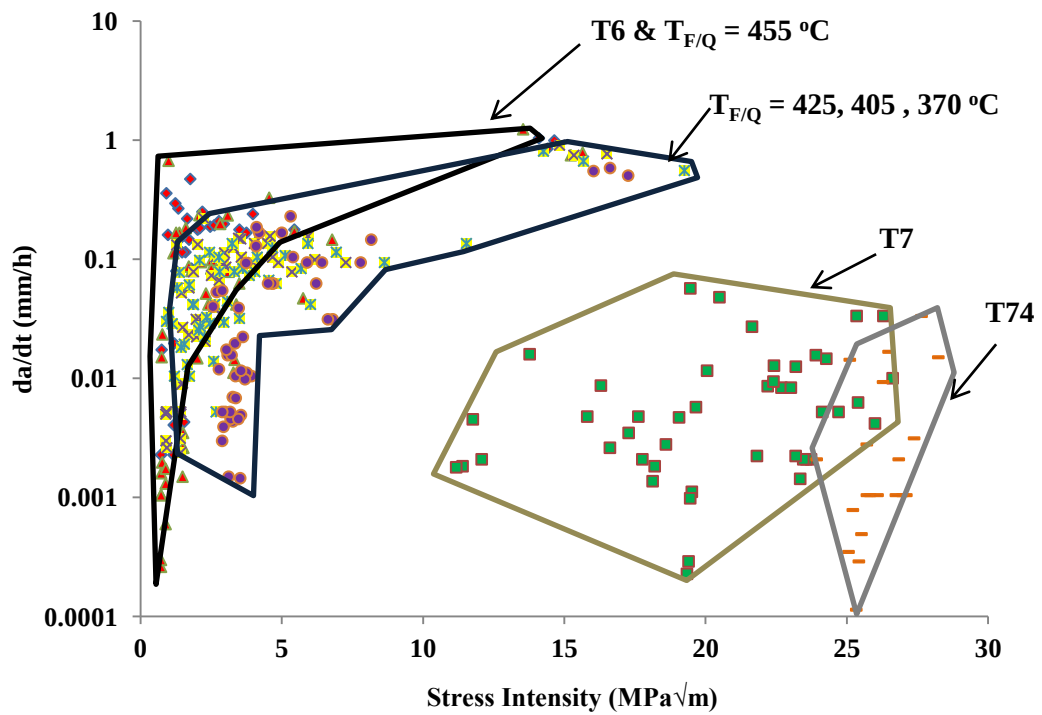


Figure 6.13: Summary of V-K plot for 7050 alloy

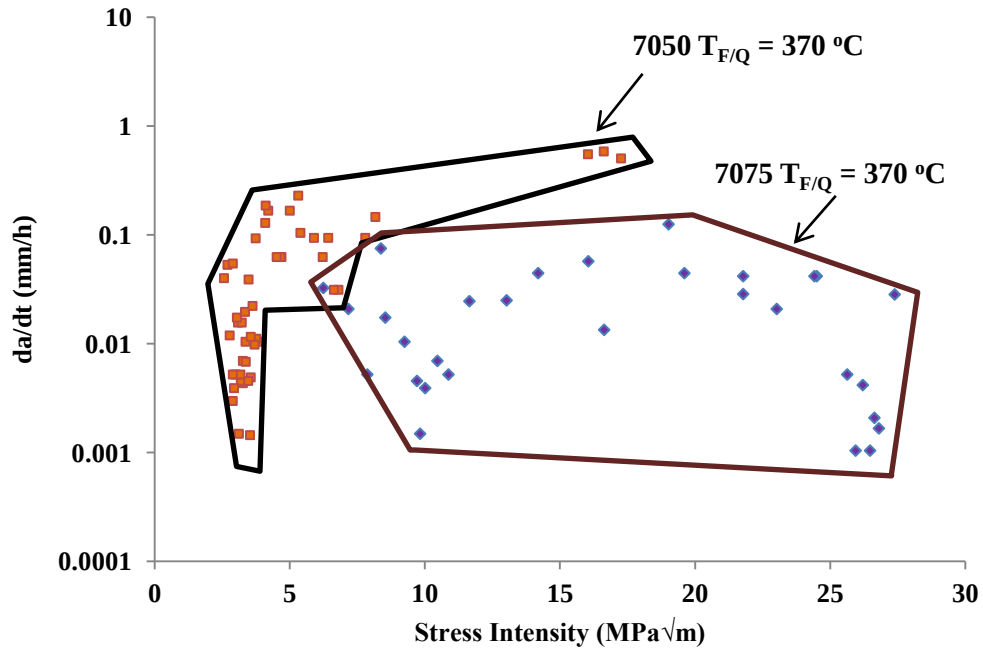


Figure 6.14: Comparison of SCC behaviour in 7050 and 7075 alloy at $T_{F/Q} = 370\text{ }^{\circ}\text{C}$

At the end of the experiment, the samples were broken by mechanical overload. Figure 6.15 shows the fractured surface of double beam samples of 7050 alloy in T6 and $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ condition after SCC test. As shown by the blue arrows in Figure 6.15 (b), the fractured surfaces show the initial crack initiated through mechanical loading. Addition of corrosive solution to the crack caused stress corrosion crack to propagate and grow with time. As shown in the $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ sample, this is a form of brittle failure accompanied by crack tunnelling (shown by red arrows). Crack tunneling is the difference in crack length at midsection of sample compared to the sample surface. Longer crack length can be observed at the mid-section due to a plane strain condition at the mid-section versus a plane stress condition at the surface.

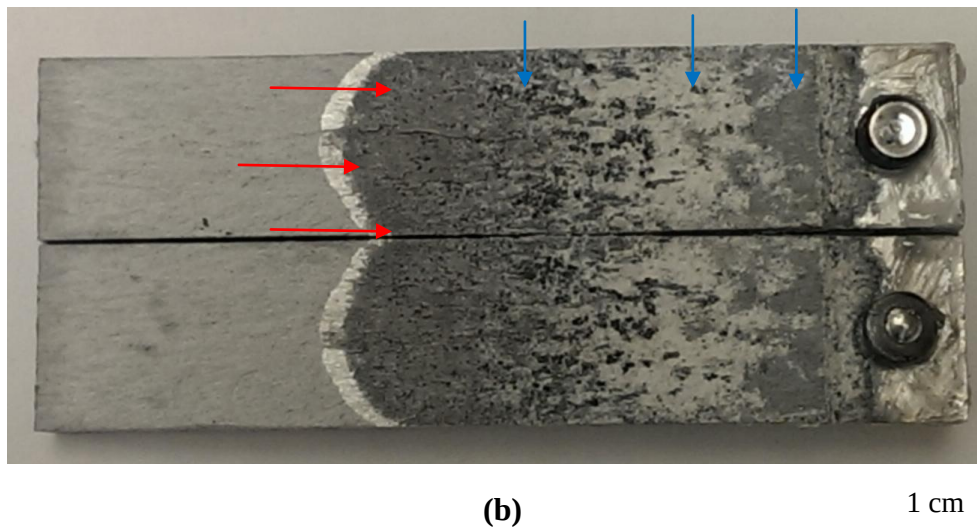
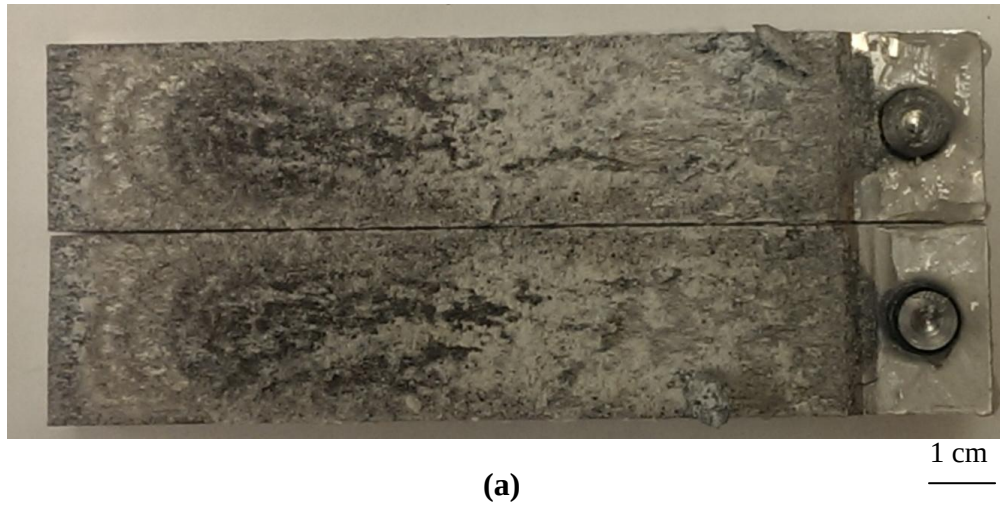


Figure 6.15: Double beam samples of 7050 alloy in (a) T6 (b) $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ condition after SCC testing. The blue arrows indicate crack growth from right to left. The red arrows indicate longer crack length in the mid-section compared to the surface (crack tunneling)

6.4 Results of microstructural analysis

Optical microscopy was used to analyze the as-received material as shown in Figure 6.16. From Figure 6.16 the elongated grains in the alloys are shown. TEM analysis of both alloys as shown in Figure 6.17 shows the effect of compositional modification on

the alloys. Both Figure 6.16 and 6.17 show the effect of compositional modification on the microstructure of the alloys. High concentration of constituent particles can be observed in 7075 alloy as shown in Figure 6.17 (a). Reducing the composition of iron and silicon (as shown in Table 5.1) reduced the constituent particles in 7050 alloy thereby improving the properties of the alloy and homogeneity of the microstructure.

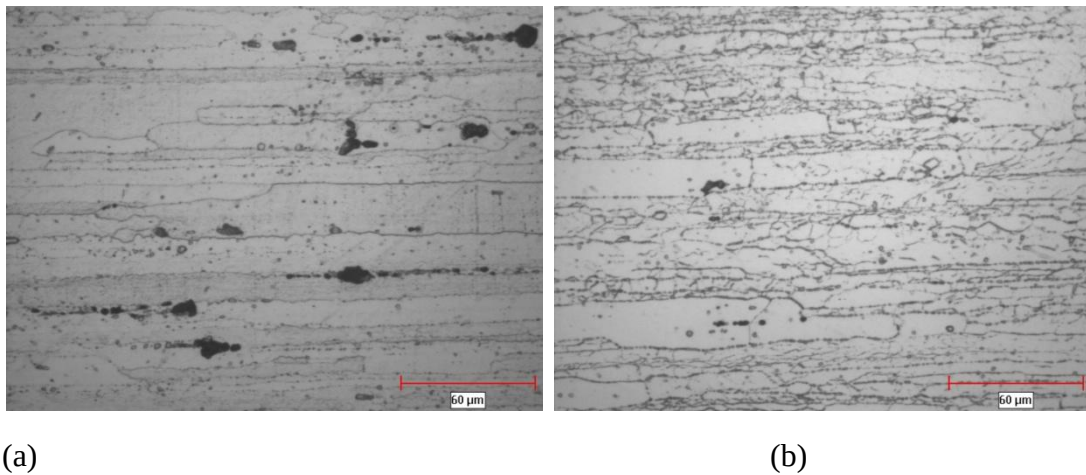


Figure 6.16: Optical images of as received (a) 7075-T651 and (b) 7050-T74 alloy in SL direction

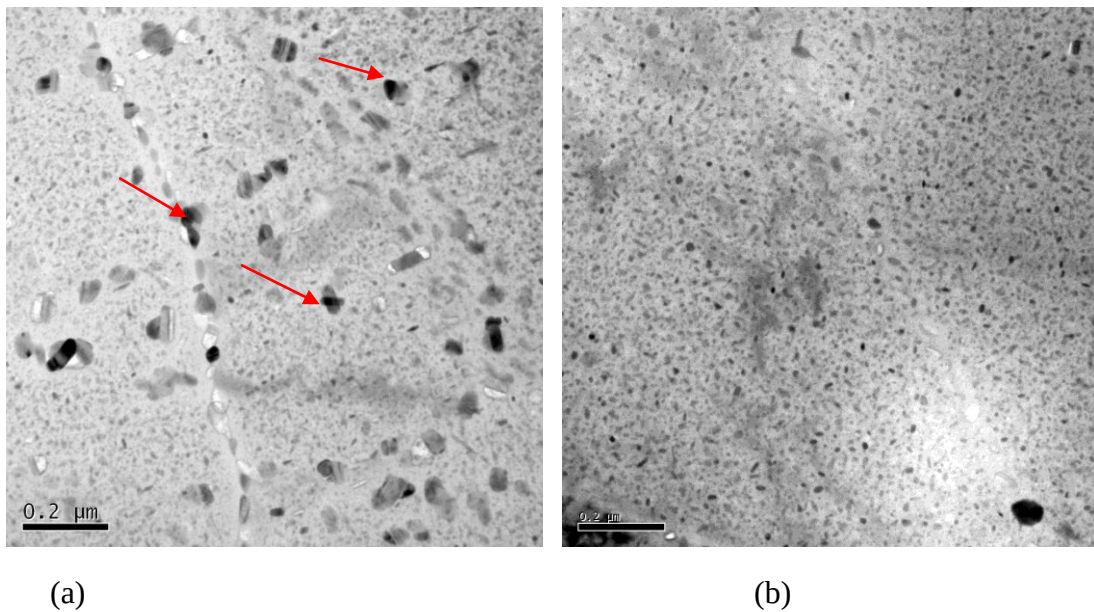
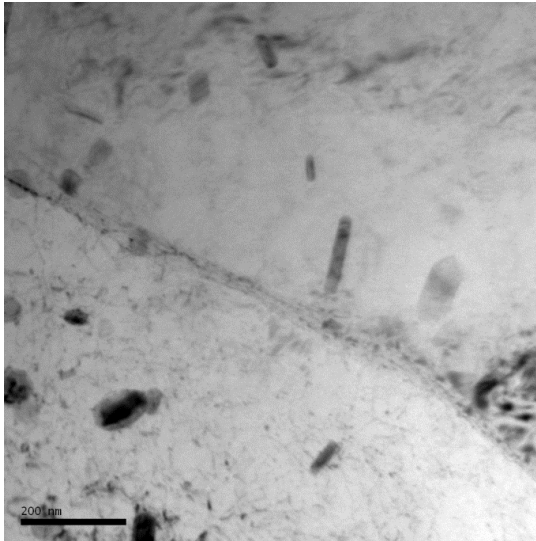


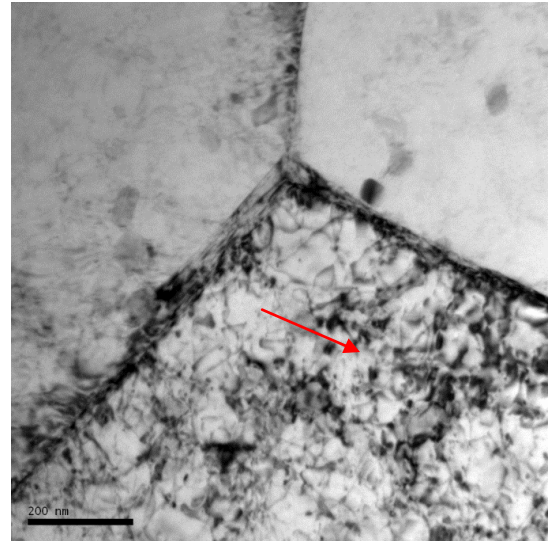
Figure 6.17: Effect of compositional modification on the microstructure of (a) 7075 and (b) 7050 alloy. Red arrows indicate large constituent particles in 7075 alloy

6.5 Microstructural analysis of unaged samples using TEM

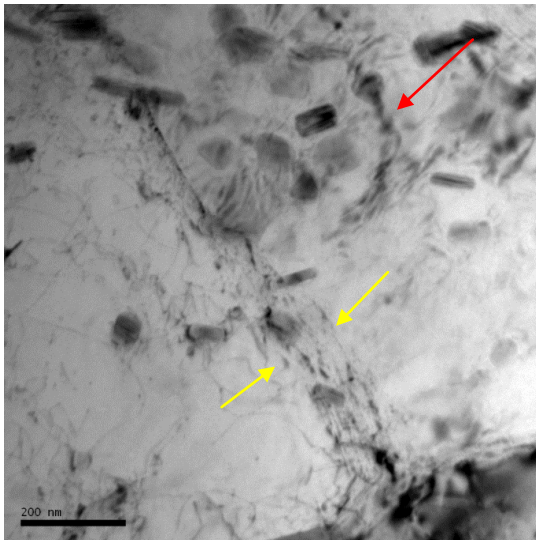
TEM was used to study grain boundary precipitate morphology of some selected tempers of 7075 and 7050 alloys in the unaged condition as shown in Figure 6.18 and 6.19 respectively. Red arrows indicate dislocation network while yellow arrows indicate grain boundary precipitation. Figure 6.18 (a) and (b) shows unaged 7075-T6 sample. In Figure 6.18 (b), the sample orientation shows high density of quenched-in dislocations. A similar result was obtained for 7050 alloy as shown in Figure 6.19 (b). High dislocation density is expected in HTPP treated sample of $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ due to quenching from a high $T_{F/Q}$ temperature. Figure 6.18 (c) and (d) shows HTPP treated sample of $T_{F/Q} = 405$ and $370\text{ }^{\circ}\text{C}$ respectively. It appears that the density of dislocations decreases as the $T_{F/Q}$ temperature decreases. This may be due to quenching from a lower temperature as $T_{F/Q}$ decreases. Also, decreasing $T_{F/Q}$ causes some heterogeneous precipitation at the grain boundary as shown in Figure 6.18 (c) and (d). A similar trend was observed in 7050 alloy as shown in Figure 6.19 (c) and (d). The contrast lines in Figure 6.19 (d) can be attributed to slight changes in thickness and bending of sample.



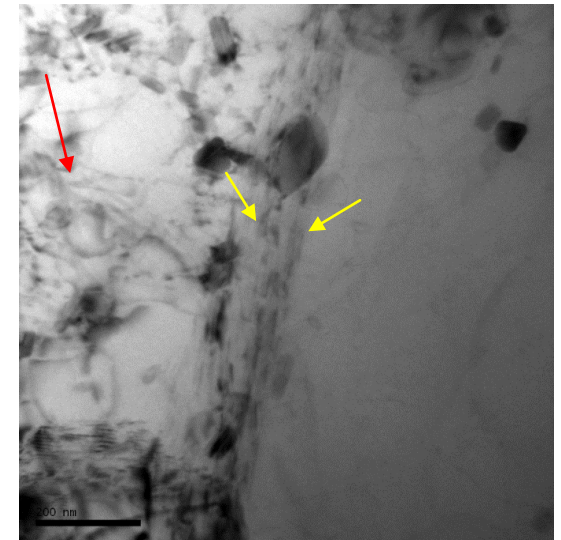
(a)



(b)

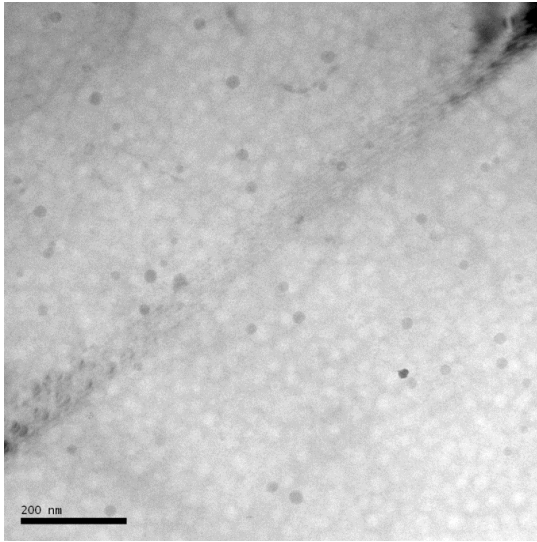


(c)

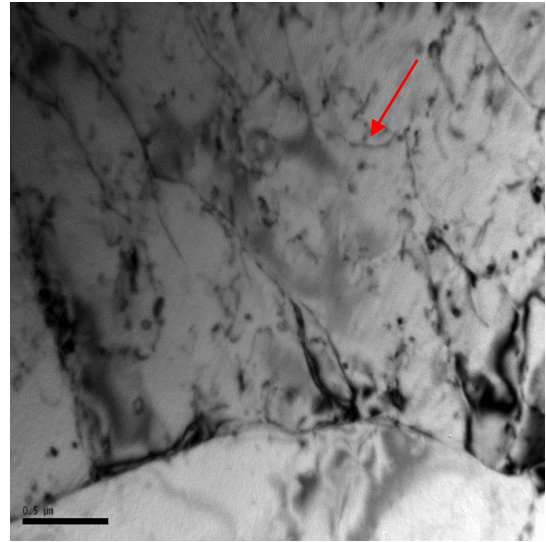


(d)

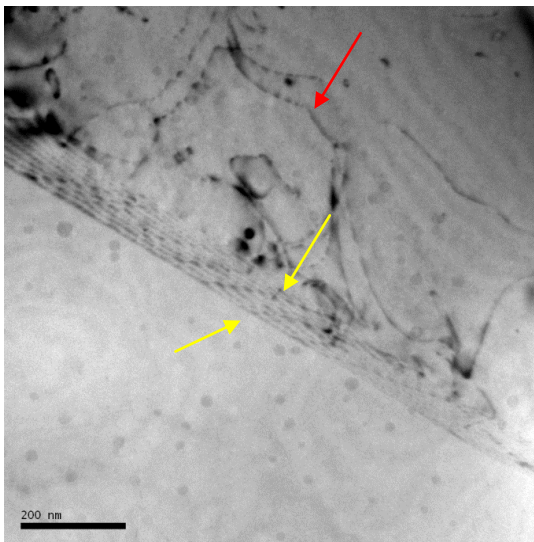
Figure 6.18: TEM analyses of unaged 7075 alloy in (a) and (b) T6 (c) $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ (d) $T_{F/Q} = 370\text{ }^{\circ}\text{C}$. Red arrows indicate dislocation network. Yellow arrows indicate precipitation along the grain boundary



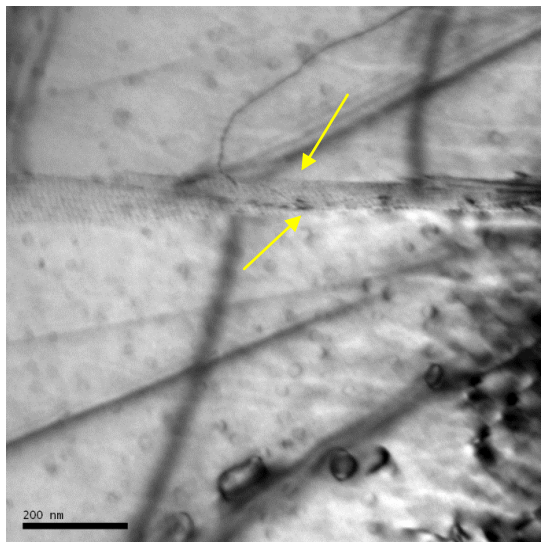
(a)



(b)



(c)



(d)

Figure 6.19: TEM analyses of unaged 7050 alloy in (a) and (b) T6 (c) $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ (d) $T_{F/Q} = 370\text{ }^{\circ}\text{C}$. Red arrows indicate dislocation network. Yellow arrows indicate precipitation along the grain boundary

6.6 Microstructural analysis of aged samples using TEM

After aging, TEM was used to analyse the precipitate morphology in the grain boundary region of the alloys. Figure 6.20 and 6.21 shows TEM analyses of the grain boundary region of 7075 and 7050 respectively. The results presented in this thesis were obtained after examining minimum of two grain boundaries. The grain boundaries are shown as either tilted or in planar view to the direction of TEM beam. As shown in Figure 6.20 (a), tilted view gives a partial plan view of the precipitates in the grain boundary region to provide more information on the density of precipitates along the grain boundary region. As shown in Figure 6.20 (f) planar view shows the precipitates lying in one plane.

In Figure 6.20 and 6.21 (a) small precipitates are continuously distributed along the grain boundary in T6 treated samples as expected. Figure 6.20 and 6.21 (b)-(e) show that HTPP heat treatment affects the microstructure of both 7075 and 7050 alloy respectively. As shown by the yellow arrows, there is gradual increase in the size of the grain boundary precipitates as $T_{F/Q}$ temperature decreases in both 7075 and 7050 alloy respectively. Although Figure 6.20 (f) is in planar view, it is obvious that there are discontinuous, large precipitates along the grain boundary. Similar result was obtained for 7050 alloy as shown in Figure 6.21 (f).

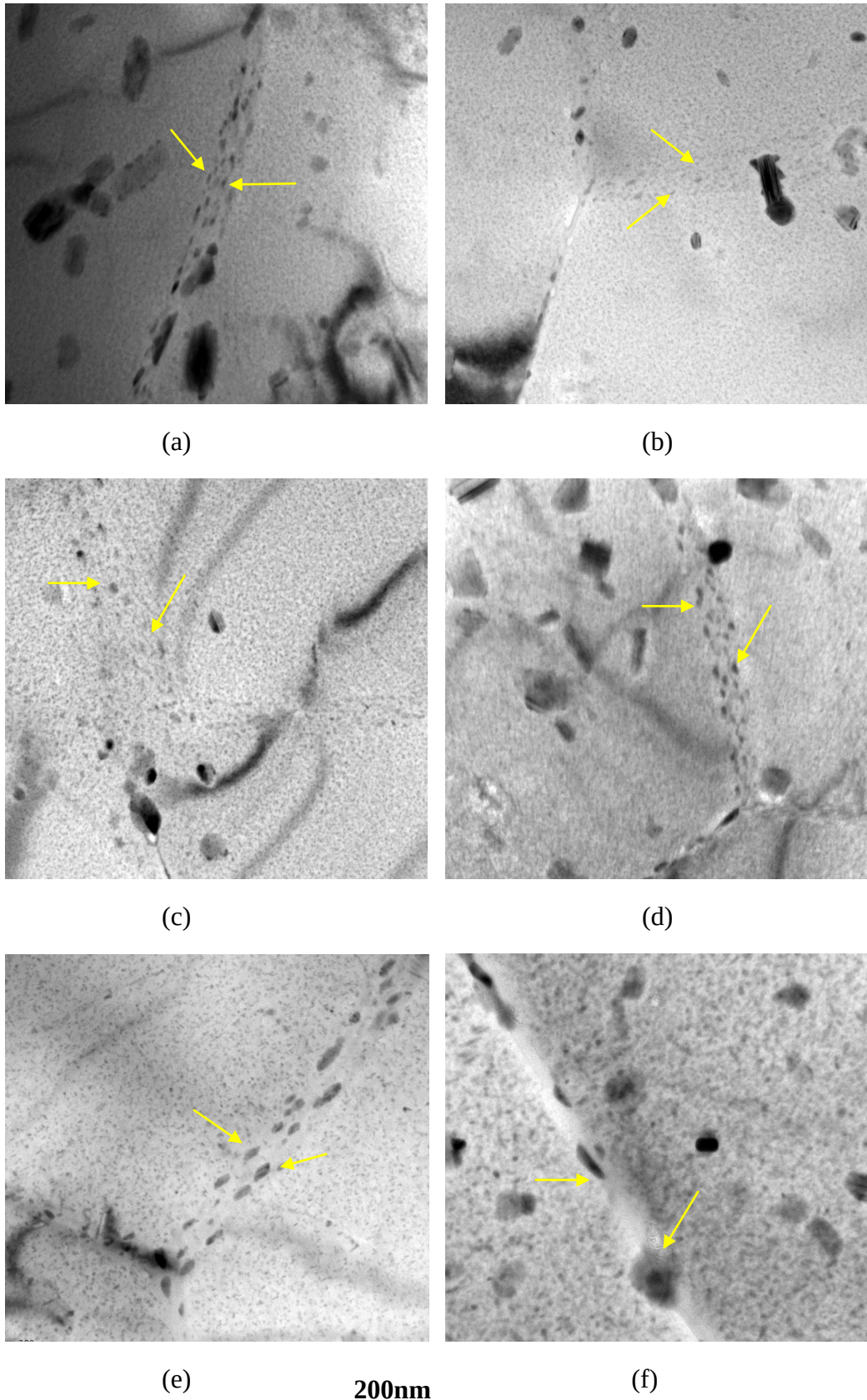


Figure 6.20: TEM analysis of grain boundary precipitate morphology of 7075 alloy in (a) T6 (b) $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ (c) $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ (d) $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ (e) $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ (f) T7. Yellow arrows indicate precipitation along the grain boundary

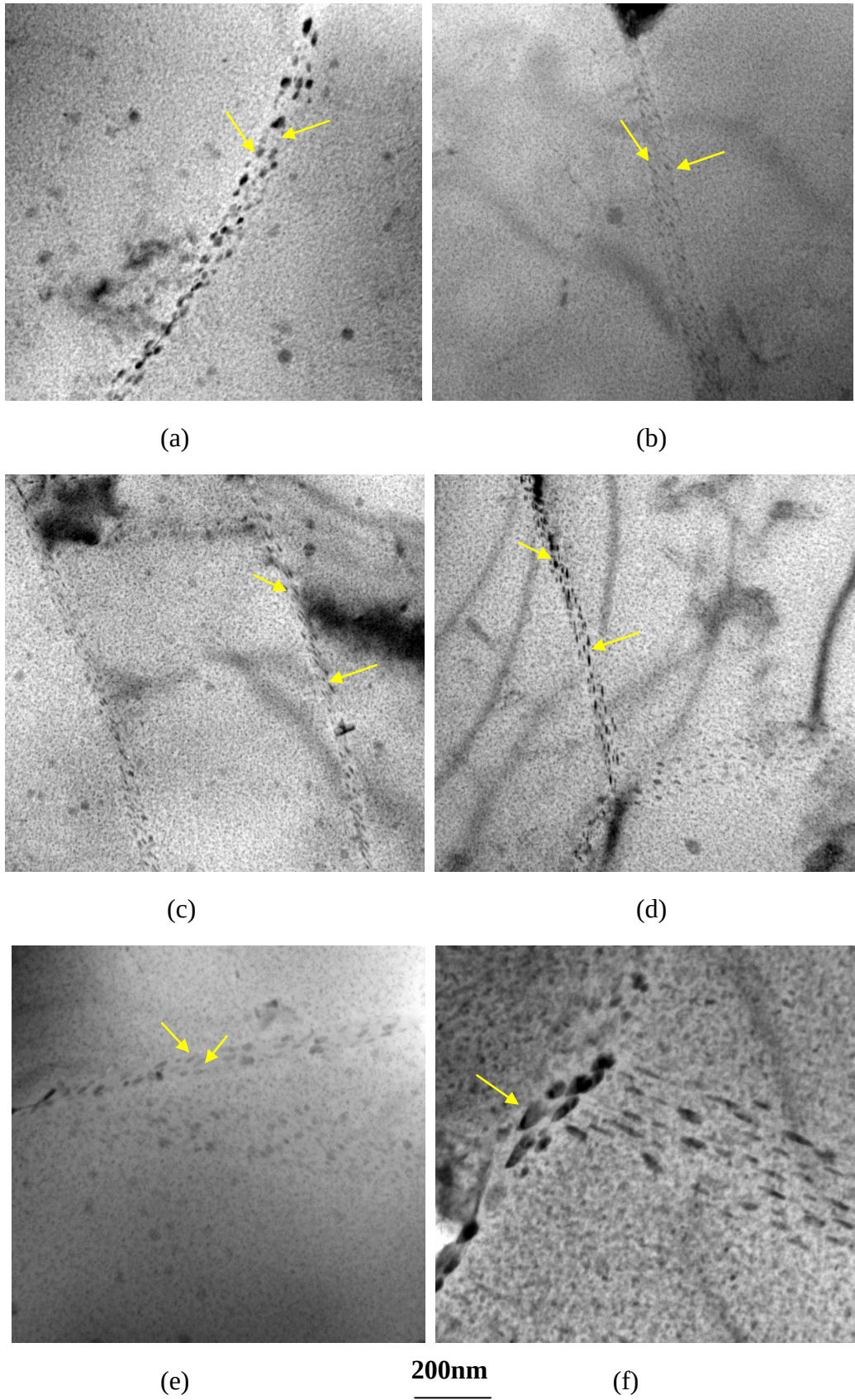


Figure 6.21: TEM analysis of grain boundary precipitate morphology of 7050 alloy in (a) T6 (b) $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ (c) $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ (d) $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ (e) $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ (f) T7. Yellow arrows indicate precipitation along the grain boundary

6.7 Energy Dispersive X-ray Spectrometry (EDS) analysis

Figures 6.22 and 6.23 show the mean solute concentration of Zn, Mg and Cu solutes segregated across two grain boundaries in 7075 and 7050 alloy respectively. Solute segregation at the grain boundary has been suggested to have influence on SCC resistance of Al-Zn-Mg-Cu alloys [60]. Decrease in solute segregation can be related to increase in solute concentration of grain boundary precipitates. The presence of copper in grain boundary precipitates has been reported in some SCC resistant alloys [63]. Results obtained in Figures 6.22 and 6.23 provide an overview of the amount of Zn, Mg and Cu segregated at the grain boundary. Note that some scatters are present in the result due to slight variation in solute concentration at different grain boundaries as shown in Figure 6.24. Variation of about 1nm was also observed in the position of the beam across the grain boundary. Corresponding data for Figure 6.22 and 6.23 are in appendix D.

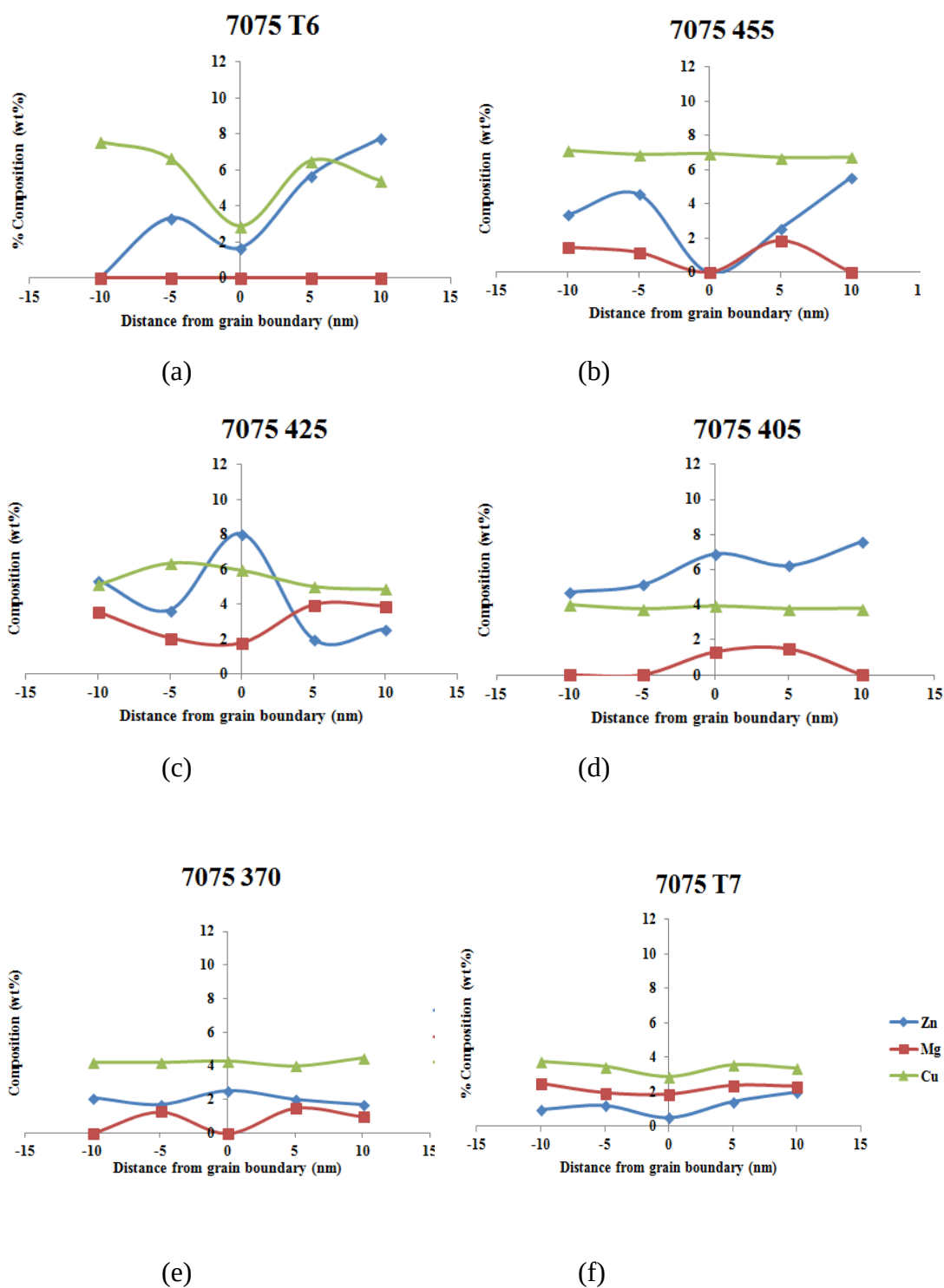


Figure 6.22: EDS analysis across grain boundary of 7075 samples showing (a) T6 (b) $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ (c) $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ (d) $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ (e) $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ and (f) T7. As shown in Figure 6.23 (f) the blue line represents Zn, red represents Mg and green represents Cu

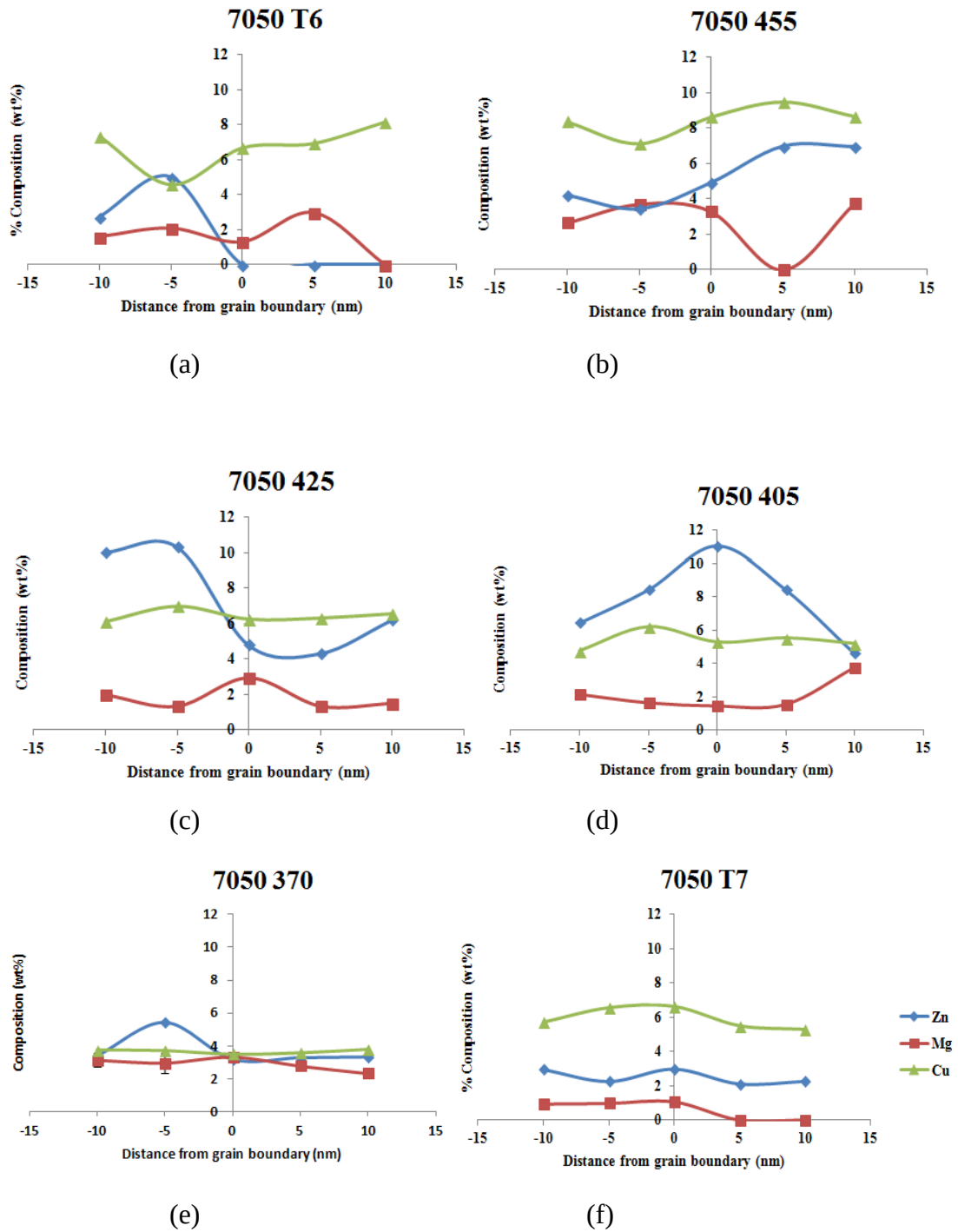


Figure 6.23: EDS analysis across grain boundary of 7050 samples showing (a) T6 (b) $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ (c) $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ (d) $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ (e) $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ and (f) T7. As shown in Figure 6.24 (f) the blue line represents Zn, red represents Mg and green represents Cu

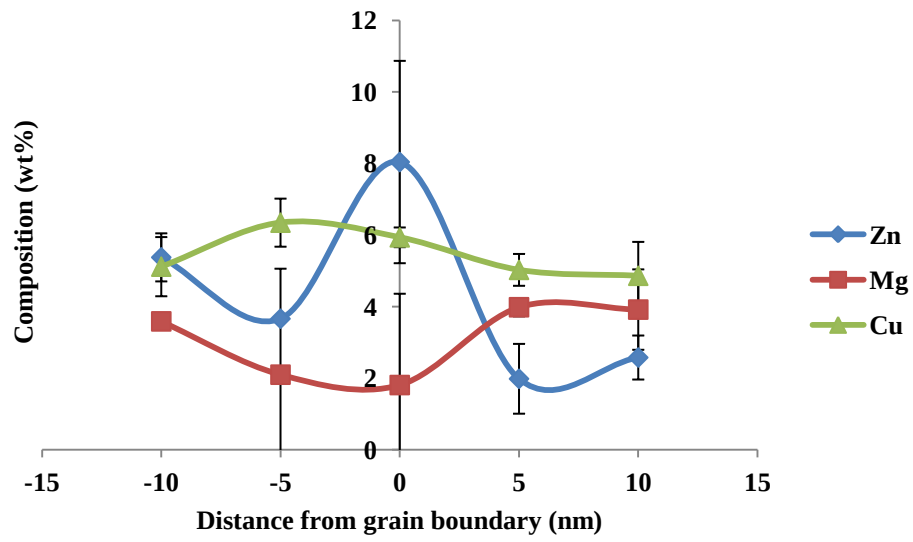


Figure 6.24: EDS analysis showing the extent of scatter in results for 7075 alloy at $T_{F/Q} = 425\text{ }^{\circ}\text{C}$

CHAPTER SEVEN

DISCUSSION OF RESULTS

7.1 Effect of heat treatment on electrical conductivity of 7075 and 7050 alloys

Figure 7.1 summarizes the effect of standard heat treatments and HTPP process on EC of aged 7050 and 7075 alloys. The result shows the EC of T6 as the lowest and T7/T74 temper as the highest. It is evident from this result that HTPP achieved increased EC in both alloys as $T_{F/Q}$ decreases and this provides a range of EC values between T6 and T7 temper. The increase in EC as $T_{F/Q}$ decreases may be explained by decrease in the concentration of solute due to loss of solute during pre-precipitation. Hence, there is decrease in resistance to flow of electrical current and an increase in EC. Another explanation for the increase in EC might be due to decrease in strain at the precipitate-matrix interface as the precipitates transform from coherent to semi-coherent or incoherent as $T_{F/Q}$ decreases. Further investigation will be required to confirm this explanation [77]. The higher EC observed in 7050 compared to 7075 alloy might be due to lower impurity levels in 7050 alloy. The arrows in Figure 7.1 indicates sharp increase in EC observed at about $T_{F/Q} = 445\text{ }^{\circ}\text{C}$ and $415\text{ }^{\circ}\text{C}$.

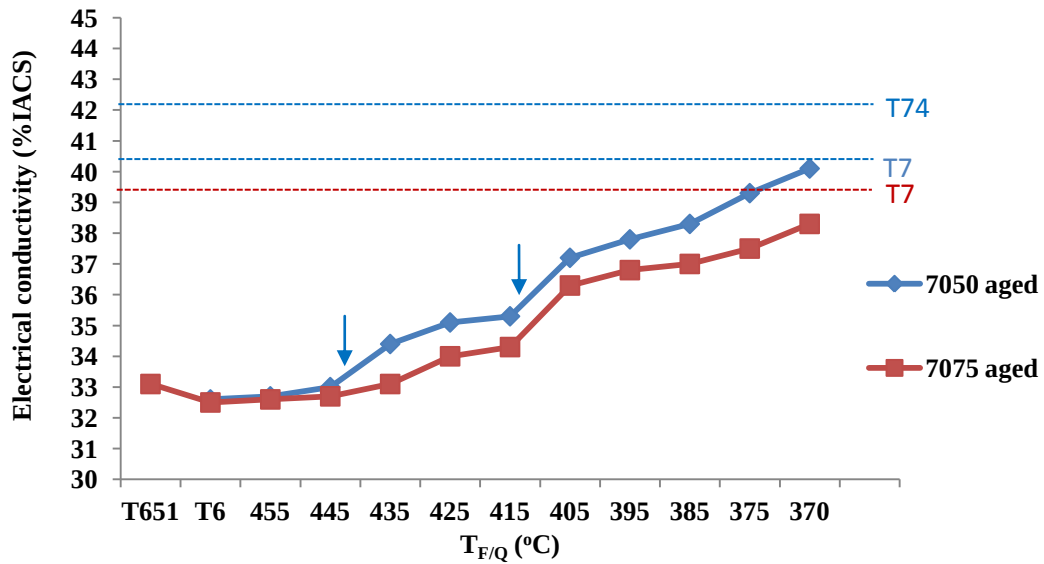


Figure 7.1: Effect of heat treatment on electrical conductivity of 7050 and 7075 alloy. Blue arrows indicate the sharp increase in electrical conductivity

7.2 Effect of heat treatment on hardness of 7050 and 7075 alloys

Figure 7.2 compares the effect of standard T6, T7 and HTPP heat treatment conditions on the hardness of both 7050 and 7075 alloys. Both alloys experienced loss of strength before aging. This is due to dissolution of strengthening phases and loss of solute atoms during pre-precipitation. The results show that subsequent aging of HTPP treated samples increases the hardness in both alloys. The increase in strength of the alloys after aging is due to formation of strengthening precipitate phase. However, strength decreases as the $T_{F/Q}$ temperature decreases. This is likely due to decrease in the solute atoms available for homogenous precipitation during aging. As $T_{F/Q}$ decreases more pre-precipitation occurs causing loss of solute atoms to heterogeneous precipitation during slow cooling to intermediate ($T_{F/Q}$) temperature.

However, gradual loss of hardness was observed as $T_{F/Q}$ decreased from 435 °C. This might be due to the solvus temperature being in that temperature range, causing more pre-precipitation to occur. Sharp changes in hardness comparable to the changes noted by the arrows in Figure 7.1 can be observed in Figure 7.2. From Figure 7.2, the hardness result of both alloys in the unaged condition show that significant changes in the hardness started at 445 °C. Thus, the solvus temperature can be estimated to be at 445-435 °C. Figure 7.3 shows that different phases can be nucleated during cooling below the solvus. Just below the solvus, the arrow shows that nucleation of η phase occurs and as the $T_{F/Q}$ temperature decreases, other phases will be formed. Thus, the sharp changes observed in EC and hardness might be related to nucleation of different phases. Further research using Differential Scanning Calorimetry (DSC) is required to confirm this conclusion.

It is evident that 7050 alloy can be treated to higher hardness than 7075 through HTPP. This can be related to the effect of compositional modification in replacement of Cr with Zr in 7050 to reduce quench sensitivity associated with 7075 alloy. In fact, $T_{F/Q}$ up to 385 °C in 7050 alloy achieves better hardness compared to T7 temper.

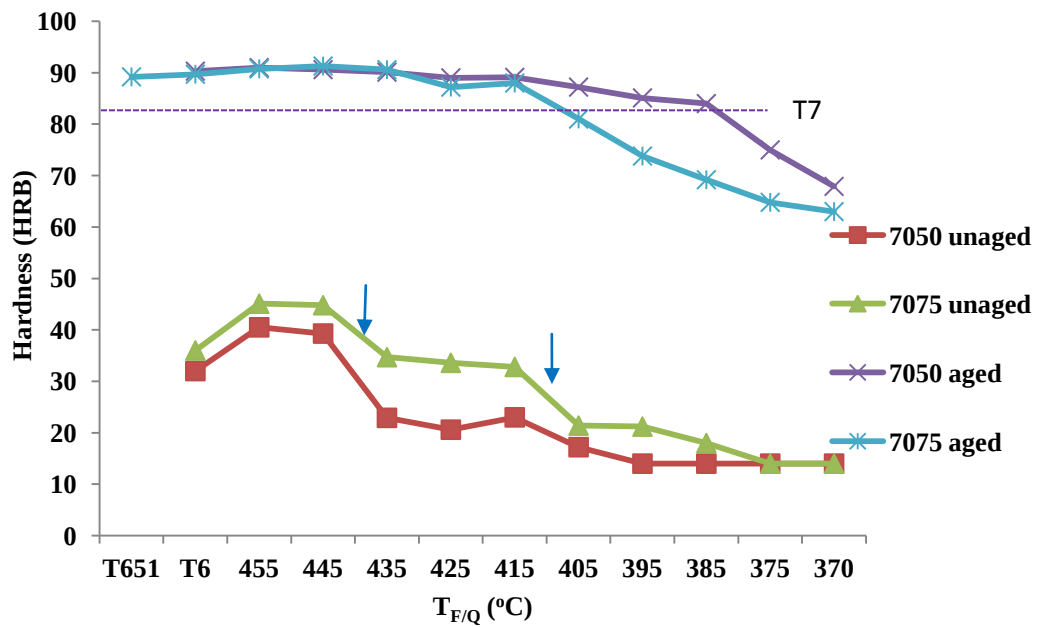


Figure 7.2: Effect of heat treatment on hardness of 7050 and 7075 alloys. Purple broken line indicates standard T7 temper. Blue arrows indicate sharp decrease in hardness

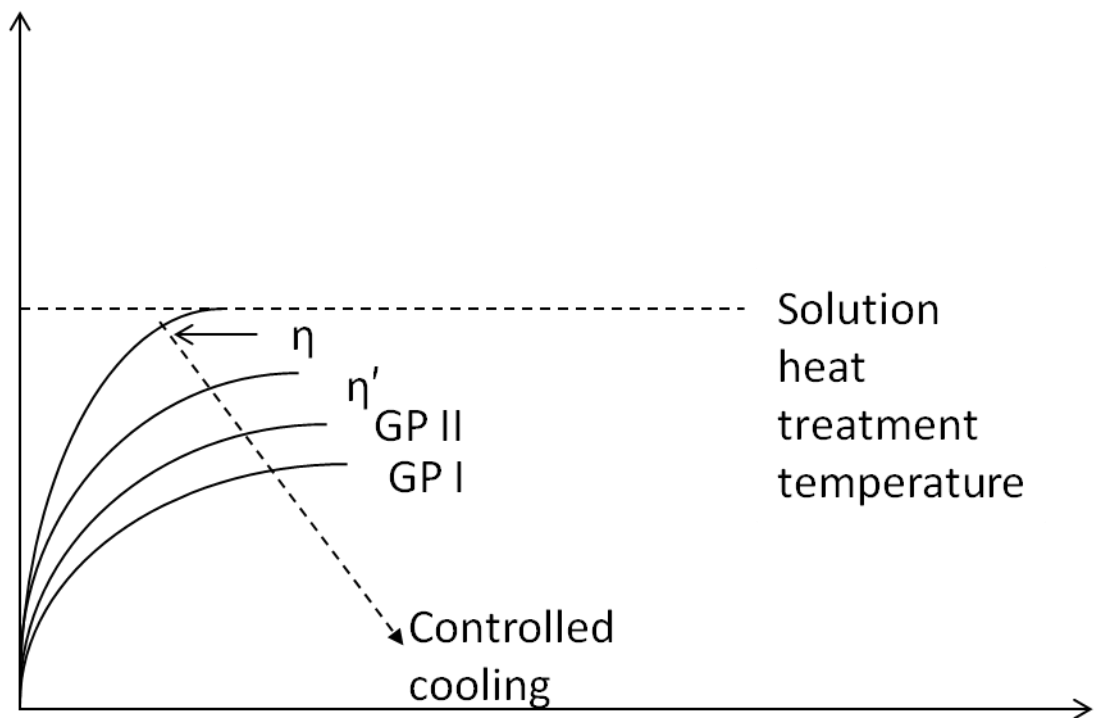


Figure 7.3: Nucleation of precipitate phases during controlled cooling possibly causing the marked changes observed in EC and hardness results

7.3 Exfoliation Corrosion (EXCO) versus Electrical conductivity of 7075 and 7050 alloy

Electrical conductivity is often used to assess and predict corrosion resistance in 7xxx alloys especially in T6 and T7 tempers. Figure 7.4 relates the exfoliation corrosion resistance of both alloys to electrical conductivity at different heat treatment conditions. The general trend observed in this result is that exfoliation corrosion susceptibility decreases as EC increases. EC obtained at $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ in 7050 alloy is almost the same as T7 temper but T7 showed higher resistance to exfoliation corrosion.

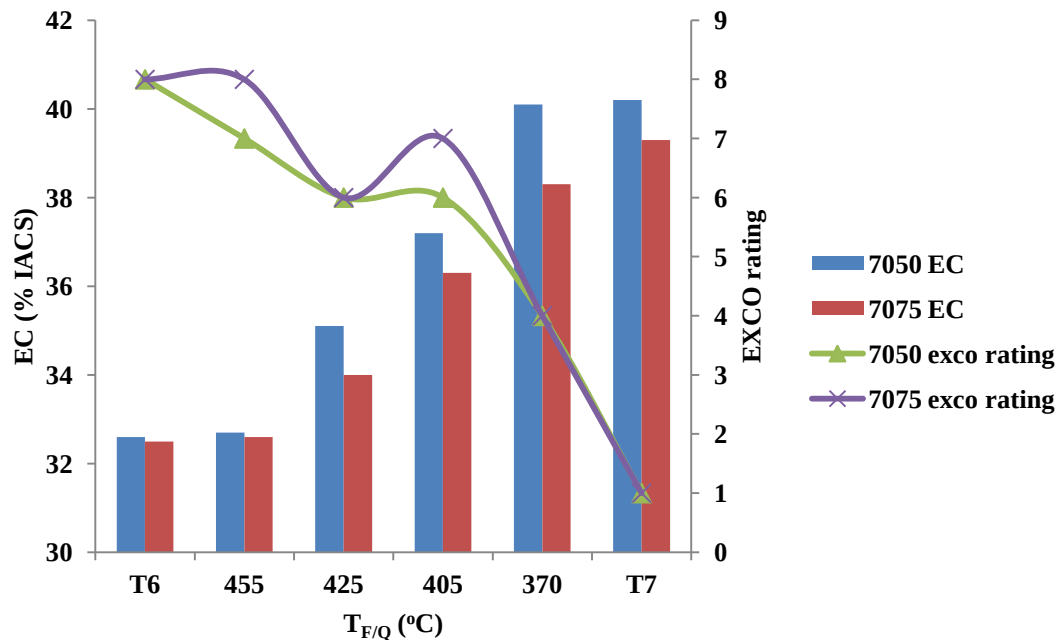


Figure 7.4: Effect of heat treatment on EC and EXCO resistance

7.4 Effect of HTPP on Stress corrosion cracking of 7075 and 7050 alloy

Figure 7.5 relates EC to SCC resistance in both alloys (using crack length measurement after 257 hours) for T7 and T6 tempers and the tested HTPP conditions. It is evident that there is a relationship between SCC resistance and EC measured as $T_{F/Q}$ decreases. Although the mechanism is not clear, these results show that SCC resistance in HTPP treated alloys is directly related to EC measurement. This is shown by decrease in crack length as EC increases. Figures 7.6 and 7.7 relate SCC resistance (based on the crack growth rate at stress intensity of $3 \text{ MPa}\sqrt{\text{m}}$) of 7075 and 7050 alloy in different heat treatment conditions to EC and the approximate time to reach stress intensity of $3 \text{ MPa}\sqrt{\text{m}}$ (corresponding to $\approx 69 \text{ mm}$ based on Figure 5.9). It is evident from these results that the crack growth rate is inversely proportional to EC and time to reach stress intensity of $3 \text{ MPa}\sqrt{\text{m}}$ in stage II of SCC. This confirms that SCC is directly related to EC. These results also show that each HTPP treatment has significant effect on the microstructure thereby increasing SCC resistance of the alloys. HTPP heat treatment reduces the crack growth rate, achieves increase in the EC and time involved in crack propagation and this relates to the observed increase in SCC resistance as the $T_{F/Q}$ temperature decreases.

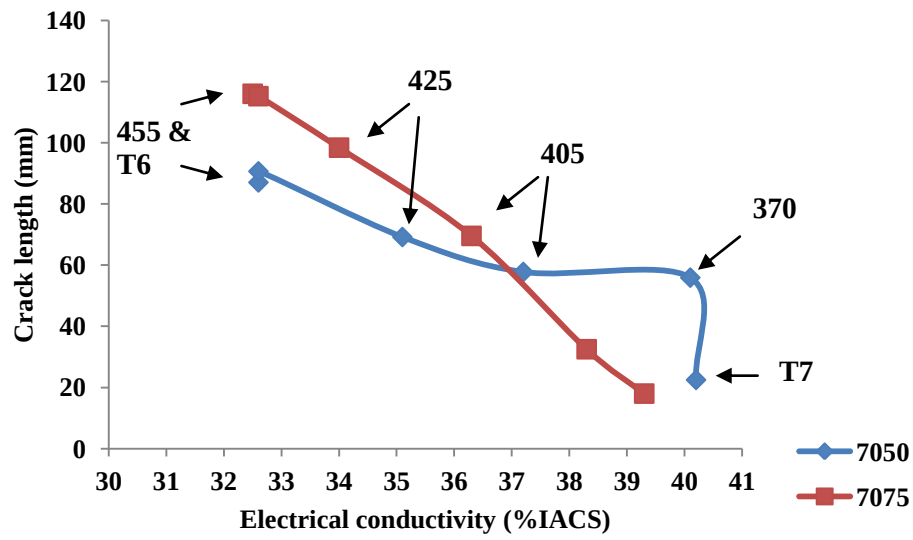


Figure 7.5: Relationship between EC and SCC resistance (using crack length measured after 257 hours) of 7075 and 7050 alloy

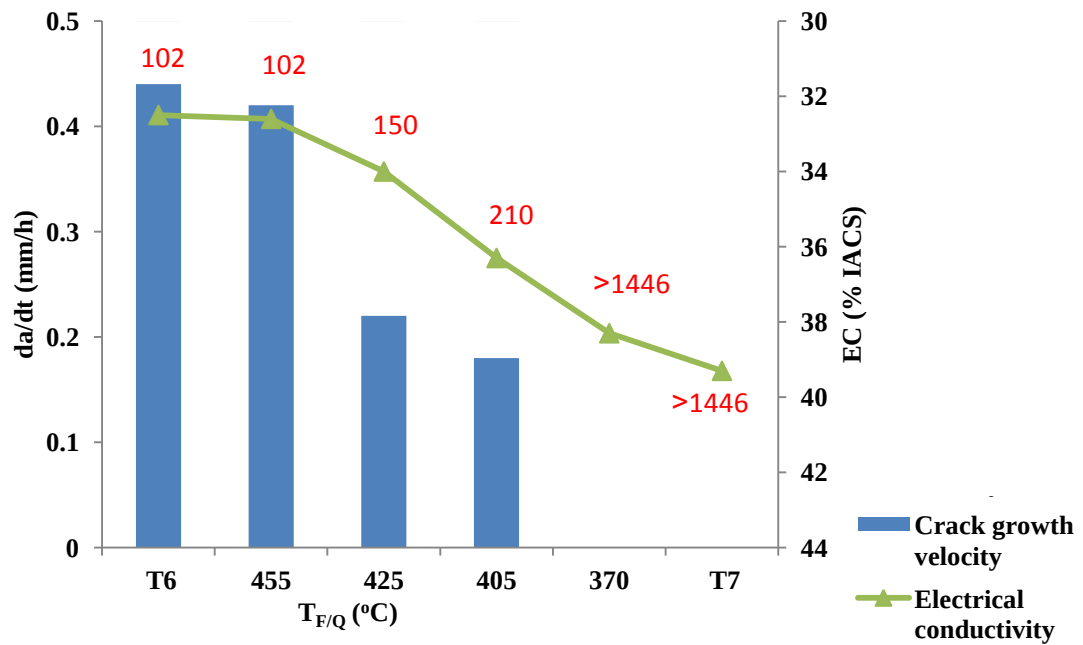


Figure 7.6: Summary of effect of heat treatment of 7075 alloy on electrical conductivity and crack growth velocity at 3 MPa√m. Numbers in red are the time (h) for stress intensity to decrease to 3 MPa√m (corresponding to a crack length of ≈ 69 mm from Figure 5.9)

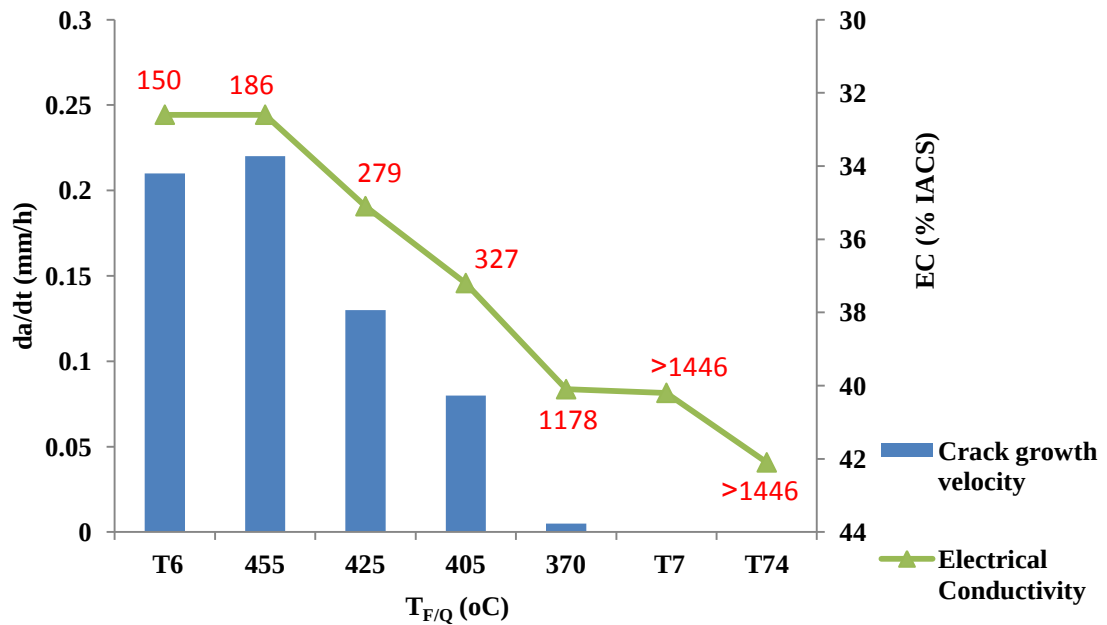


Figure 7.7: Summary of effect of heat treatment of 7050 alloy on electrical conductivity and crack growth velocity at 3 MPa√m. Numbers in red are the time (h) for stress intensity to decrease to 3 MPa√m (corresponding to a crack length of ≈ 69 mm from Figure 5.9)

7.5 Grain boundary precipitate versus SCC resistance

From Figure 6.20 (a) and 6.21 (a) small, discontinuous grain boundary precipitates in T6 can be related to poor corrosion resistance while the large grain boundary precipitates in T7 temper (Figure 6.20 (f) and 6.21 (f)) correlates with high SCC resistance obtained in both alloys. Figures 6.20 (b-e) and 6.21 (b-e) show that grain boundary precipitates gradually increased in size as $T_{F/Q}$ decreased during HTPP while Figure 7.5 shows that as $T_{F/Q}$ decreased, crack length decreased in both alloys. Summary of V-K plot of both alloys shown in Figures 6.12 and 6.13 show that HTPP treatment causes decrease in the plateau velocity as $T_{F/Q}$ decrease. $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ showed the best resistance to SCC of all the HTPP conditions tested. Results shown in Figures 6.12, 6.13, 7.5, 7.6 and 7.7 correspond to increase in size and discontinuity of grain boundary precipitates observed in Figure 6.21 and 6.22. These results correlate with studies by Li et al. (2008) that shows that HTPP can achieve higher SCC resistance [29]. In terms of the size of the grain boundary precipitates, studies have shown that large grain boundary precipitates can serve as nucleation site for hydrogen bubbles thereby reducing SCC by hydrogen embrittlement. Also, large grain boundary precipitates reduce the effect of SCC by anodic dissolution [77].

The gradual increase in SCC resistance as $T_{F/Q}$ decreases can also be related to increase in copper concentration of grain boundary precipitates. This is supported by EDS analysis in Figures 6.22 and 6.23 showing that as $T_{F/Q}$ decreases, there is decrease in copper solute segregated at the grain boundary. The increase in the potential of the grain boundary precipitate reduces SCC through anodic dissolution. The increase in homogeneity and uniformity of solute concentration at the grain boundaries as $T_{F/Q}$

decreases can also improve SCC resistance by reducing the formation of local galvanic cells that might cause SCC.

7.6 Effect of HTPP on microstructure

TEM analysis of the unaged samples in Figure 6.18 and 6.19 shows that dislocations were present in the unaged samples after quenching. These can be identified as quenched-in dislocations formed due to internal stress generated during quenching. It appears that there is decrease in dislocation density as $T_{F/Q}$ decreases. This shows that HTPP heat treatment can reduce the dislocation density in 7075 and 7050 alloys. Some preferential heterogeneous precipitation occurred at the grain boundary region in all the HTPP conditions observed due to prior slow cooling before quenching. TEM analysis in Figure 6.20 relates the morphology of grain boundary precipitates in HTPP treated conditions to standard T6 and T7 tempers of 7075 alloy. As shown in Figure 6.20 (a). The morphology of precipitates in the grain boundary region of T6 shows high concentration of small precipitates that are continuously distributed along the grain boundary. From Figure 6.20 (b)-(e) it is evident that the initial precipitation during quenching enabled more precipitation to occur at the grain boundary region after aging. This is evident from the increase in size of grain boundary precipitates as $T_{F/Q}$ temperature decreases from $T_{F/Q} = 455$ to 370 °C. The gradual increase in precipitate size agrees with the literature [26, 28]. This can be explained by increase in precipitation at the grain boundary due to prior pre-precipitation during slow cooling from SHT to a lower ($T_{F/Q}$) temperature. Figure 6.20 (f) shows the coarse precipitates that are discontinuously distributed along the grain boundary precipitate morphology in T7 temper. The increase in size of the precipitates is due to overaging treatment. Coarse discontinuous grain boundary precipitates are observed at $T_{F/Q} = 370$

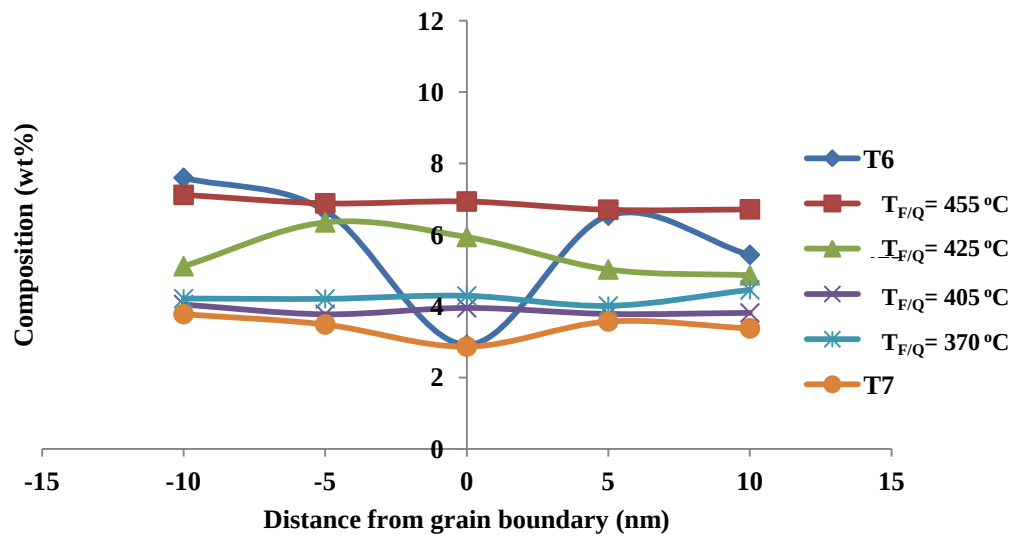
°C and T7 temper. Large discontinuous grain boundary precipitates has been related to high SCC resistance of T7 temper [40]. Similar result was observed in 7050 alloy as shown in Figure 6.21.

By comparison to unaged condition shown in Figure 6.18 and 6.19, it appears that subsequent aging after quenching of HTPP treated samples reduces the dislocation density. Thus, this results show that the $T_{F/Q}$ temperature used in HTPP treatment plays an important role in increasing the size of grain boundary precipitates and decreasing dislocation density.

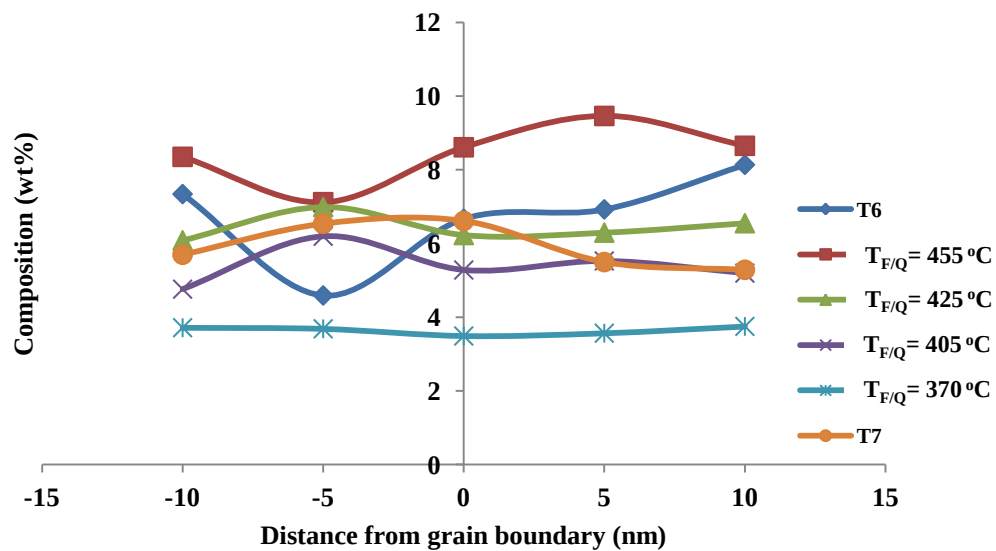
Using Energy Dispersive X-ray Spectrometry (EDS) analysis, concentration of solute elements segregated across grain boundary was analysed. Figure 6.22 and 6.23 show the results obtained from 7075 and 7050 alloy respectively. It is evident that decrease in $T_{F/Q}$ temperature causes increases homogeneity in the distribution of Zn, Mg and Cu across the grain boundary. This can be related to increase and uniformity in the diffusion of Zn, Mg and Cu into grain boundary precipitates. This trend is similar to results reported by Hepples et al. shown in Figure 4.8 [62]. Figure 7.8 shows that there is decrease in the amount of copper segregated in the HTPP treated samples as $T_{F/Q}$ decreases. This means increase in Cu concentration in the grain boundary precipitates. This result corresponds with the literature that HTPP can increase in Cu content at the grain boundary precipitates [26, 28]. Hepples et al. showed that copper segregated at grain boundary decreased in T7 and RRA compared to T6 [62].

In terms of Mg content, it appears that the amount of Mg segregated is higher in the HTPP treated conditions compared to T6. This correlates with literature on grain boundary segregation of Mg in 7150 alloy by Hepples et al. [62]. Although some studies show that, the presence of Mg can be related to SCC by hydrogen

embrittlement, this result confirms that Mg segregation may not be the dominant factor for SCC in these alloys [62, 64, 65].



(a)



(b)

Figure 7.8: Summary of grain boundary copper concentration in (a) 7075 and (b) 7050 alloy

CHAPTER EIGHT

CONCLUSIONS

This research has studied HTPP as a potential heat treatment approach to achieve a combination of high strength and corrosion resistance in 7075 and 7050 commercial alloys. This was achieved by using experimental procedure that involved electrical conductivity measurement, hardness measurement, exfoliation corrosion testing, stress corrosion cracking testing and analysis of grain boundary using TEM. The following conclusions can be made based on the results obtained from the experiments in this research:

- The intermediate temperature ($T_{F/Q}$) used during HTPP has a significant effect on the properties of both alloys after HTPP heat treatment.
- Both alloys exhibit a decrease in hardness as the $T_{F/Q}$ temperature decreased especially at $T_{F/Q} = 435\text{ }^{\circ}\text{C}$. However, 7050 can be heat treated to higher hardness than 7075 alloy. Zr addition in 7050 reduces the quench sensitivity during HTPP heat treatment.
- HTPP heat treatment achieves increase in SCC resistance as the $T_{F/Q}$ temperature decreased.
- Electrical conductivity varies directly with SCC resistance in both alloys. EC can thus be used to determine SCC resistance in 7050 and 7075 HTPP treated alloys.
- HTPP causes homogeneity of solute distribution at the grain boundary. Also, there is increase in discontinuity, size, and Cu concentration of grain boundary

precipitates as $T_{F/Q}$ decreases. Hence, increase in SCC resistance of HTPP treated samples can be attributed to a combination of these factors.

- None of the HTPP treated conditions achieved a good combination of strength and SCC resistance as standard T6 and T7 temper respectively. However, the range of $T_{F/Q}$ temperatures between 425 and 405 °C provides the best balance in strength and SCC resistance.

8.1 Recommendations for future work

Based on the results obtained in this research and some of the challenges experienced, the following recommendations are suggested for future work:

- The cause of stepwise changes observed in the hardness and EC results should be further investigated.
- The effect of multistep aging process in HTPP on the properties of 7050 and 7075 alloys should be investigated.
- To obtain sufficient data for V-K plot of HTPP treated samples, SCC test with slower rate is suggested using fatigue pre-cracked samples and test solution of 3.5% NaCl.

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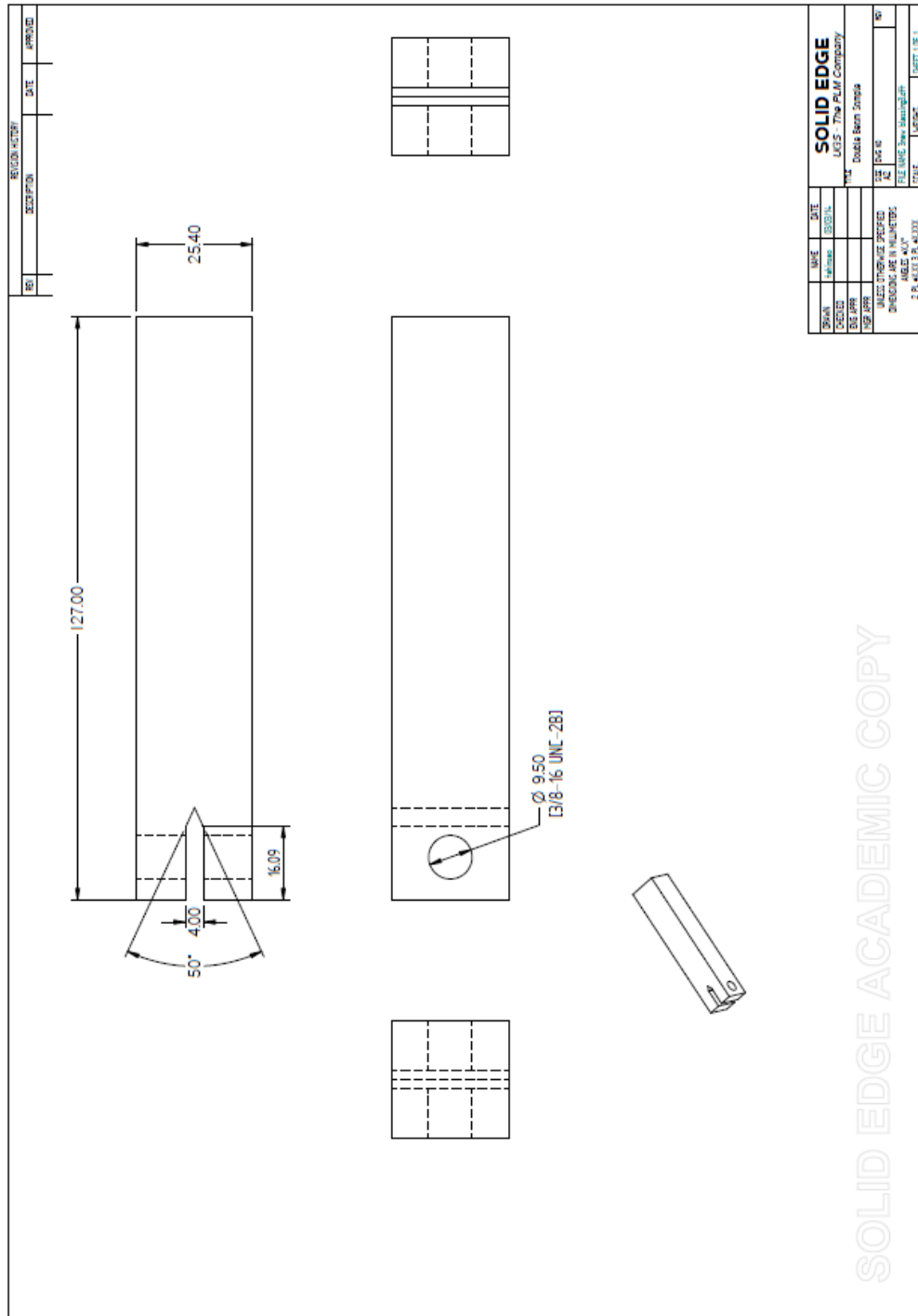
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APPENDIX

Appendix A: Double beam sample



**APPENDIX B: TEMPERATURE MEASUREMENT DURING SCC TESTING
[78]**

Date	Temperature (°C)	Date	Temperature (°C)
30-08-2013	22	01-10-2013	13
31-08-2013	22	02-10-2013	13
01-09-2013	22	03-10-2013	13
02-09-2013	22	04-10-2013	13
03-09-2013	21	05-10-2013	13
04-09-2013	21	06-10-2013	13
05-09-2013	21	07-10-2013	13
06-09-2013	21	08-10-2013	12
07-09-2013	20	09-10-2013	12
08-09-2013	20	10-10-2013	11
09-09-2013	20	11-10-2013	11
10-09-2013	19	12-10-2013	10
11-09-2013	19	13-10-2013	10
12-09-2013	19	14-10-2013	9
13-09-2013	19	15-10-2013	9
14-09-2013	18	16-10-2013	9
15-09-2013	18	17-10-2013	8
16-09-2013	18	18-10-2013	8
17-09-2013	17	19-10-2013	8
18-09-2013	17	20-10-2013	7
19-09-2013	17	21-10-2013	7
20-09-2013	16	22-10-2013	7
21-09-2013	16	23-10-2013	6
22-09-2013	16	24-10-2013	6
23-09-2013	16	25-10-2013	6
24-09-2013	15		
25-09-2013	15		
26-09-2013	14		
27-09-2013	14		
28-09-2013	14		
29-09-2013	13		
30-09-2013	13		

APPENDIX C: SCC DATA

Table 1: Data for crack length vs time for 7075 alloy

Time (h)	7075-T6	7075 $T_{F/Q} = 455\text{ }^{\circ}\text{C}$	7075 $T_{F/Q} = 425\text{ }^{\circ}\text{C}$	7075 $T_{F/Q} = 405\text{ }^{\circ}\text{C}$	7075 $T_{F/Q} = 370\text{ }^{\circ}\text{C}$	7075-T7
0	17.75	18.13	15.88	18.00	16.48	16.00
30	54.50	52.25	42.75	35.00	16.93	16.50
78	71.50	68.00	54.00	42.00	17.08	16.75
126	112.75	95.75	64.75	50.50	17.13	16.80
174	115.00	115.05	75.78	57.00	18.25	16.83
198	115.45	115.10	85.00	60.00	19.25	16.83
222	115.50	115.15	95.38	64.25	21.00	16.85
257	116.00	115.23	98.40	69.55	22.50	16.85
302	116.35	116.15	105.68	84.75	24.50	17.40
353	116.40	116.25	115.23	90.75	26.00	17.75
526	116.45	116.33	115.75	98.15	28.75	18.00
746	116.50	116.50	116.13	114.00	32.00	21.50
842				114.38	32.75	22.15
986				114.75	34.50	22.50
1178				115.13	35.38	23.00
1346				115.50	37.25	23.50

Table 2: Data for crack length vs time for 7050 alloy

Time (h)	7050-T6	7050 $T_{F/Q} = 455\text{ }^{\circ}\text{C}$	7050 $T_{F/Q} = 425\text{ }^{\circ}\text{C}$	7050 $T_{F/Q} = 405\text{ }^{\circ}\text{C}$	7050 $T_{F/Q} = 370\text{ }^{\circ}\text{C}$	7050-T7	7050-T74
0	15.93	17.63	18.38	15.37	17.70	15.83	12.75
30	44.70	45.27	42.60	35.67	34.07	16.60	13.40
78	53.03	53.50	49.10	42.00	39.40	17.00	13.47
126	63.28	60.27	54.72	45.67	42.07	17.32	13.50
174	72.20	72.17	58.93	51.33	45.57	17.73	13.52
198	75.53	74.67	61.35	53.17	50.07	18.07	13.53
222	80.20	76.67	64.02	54.25	51.15	18.53	13.55
257	87.083	85.27	69.18	57.75	55.90	18.72	13.82
302	97.77	87.20	72.77	59.75	56.98	18.80	13.86
353	105.53	88.82	76.68	64.08	57.57	20.00	13.87
526	116.00	100.35	87.93	71.08	58.57	21.18	13.88
746	116.50	114.53	99.77	76.58	59.73	22.47	13.89
842		114.82	101.13	79.92	61.07	22.75	13.95
986		115.12	103.13	81.58	62.48	23.27	14.06
1178		115.38	107.33	86.33	63.40	23.67	
1346		115.52	109.13	90.83	64.32	24.30	

Table 3: 7075-T6 sample

Time (h)	a _o (mm)	a _o (mm)	Ave. a _o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K _{ave} (MPa√m)
0	17.50	18.00	17.75		9.00	0.35	24.18	
30	53.50	55.50	54.50	1.230			4.04	14.11
78	71.00	72.00	71.50	0.350			2.49	3.27
126	113.00	112.50	112.75	0.850			1.08	1.79
174	115.00	115.00	115.00	0.047			1.04	1.06
198	115.50	115.40	115.45	0.019			1.03	1.04
222	115.50	115.50	115.50	0.002			1.03	1.03
257	116.00	116.00	116.00	0.014			1.02	1.03
302	116.20	116.50	116.35	0.008			1.02	1.02
353	116.30	116.50	116.40	0.001			1.02	1.02
526	116.40	116.50	116.45	0.0002			1.02	1.02
746	116.50	116.50	116.50	0.0002			1.01	1.01

Table 4: 7075-T7 sample

Time (h)	a _o (mm)	a _o (mm)	Ave. a _o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K _{ave} (MPa√m)
0	17.00	15.00	16.00		9.00	0.34	27.12	
30	17.00	16.00	16.50	0.017			26.00	26.56
78	17.00	16.50	16.75	0.005			25.47	25.74
126	17.00	16.60	16.80	0.001			25.36	25.42
174	17.00	16.65	16.83	0.000			25.31	25.34
198	17.00	16.66	16.83	0.000			25.30	25.31
222	17.00	16.70	16.85	0.000			25.26	25.29
257	17.00	16.80	16.90	0.001			25.16	25.21
302	18.00	16.80	17.40	0.011			24.16	24.66
353	18.50	17.00	17.75	0.006			23.49	23.82
526	19.00	17.00	18.00	0.001			23.03	23.26
746	22.00	21.00	21.50	0.015			17.82	20.43
842	22.30	22.00	22.15	0.007			17.05	17.43
986	22.50	22.50	22.50	0.002			16.65	16.86
1178	23.00	23.00	23.00	0.003			16.11	16.38
1346	23.00	24.00	23.50	0.003			15.60	15.86

Table 5: 7075-HTPP at $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ sample 1

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	20.00	20.50	20.25		9.00	0.4	22.89	
30	53.50	53.50	53.50	1.108			4.77	13.83
78	68.00	69.00	68.50	0.313			3.08	3.93
126	113.50	115.50	114.50	0.958			1.20	2.14
174	116.00	116.00	116.00	0.031			1.17	1.19
198	116.00	116.10	116.05	0.002			1.17	1.17
222	116.00	116.20	116.1	0.002			1.17	1.17
257	116.00	116.30	116.15	0.001			1.17	1.17
302	116.10	116.5	116.3	0.003			1.16	1.16
353	116.20	116.5	116.35	0.001			1.16	1.16
526	116.30	116.5	116.4	0.000			1.16	1.16
746	116.50	116.5	116.5	0.000			1.16	1.16

Table 6: 7075-HTPP at $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ sample 2

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	16.50	15.50	16.00		9.00	0.30	23.93	
30	50.00	52.00	51.00	1.167			3.89	13.91
78	68.50	66.50	67.50	0.344			2.37	3.13
126	77.00	77.00	77.00	0.198			1.87	2.12
174	114.10	114.10	114.10	0.773			0.90	1.39
198	114.10	114.20	114.15	0.002			0.90	0.90
222	114.20	114.20	114.20	0.002			0.90	0.90
257	114.30	114.30	114.30	0.003			0.90	0.90
302	116.00	116.00	116.00	0.038			0.88	0.89
353	116.20	116.10	116.15	0.003			0.87	0.88
526	116.30	116.20	116.25	0.001			0.87	0.88
746	116.50	116.50	116.5	0.001			0.87	0.87

Table 7: 7075-HTPP at $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ sample 1

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	15.50	16.50	16.00		9.00	0.30	23.93	
30	49.00	49.00	49.00	1.100			4.17	14.05
78	61.00	61.00	61.00	0.250			2.84	3.50
126	73.50	72.50	73.00	0.250			2.06	2.45
174	86.80	87.00	86.90	0.290			1.50	1.78
198	103.00	103.00	103.00	0.671			1.09	1.30
222	115.50	116.00	115.75	0.531			0.88	0.99
257	115.60	116.00	115.80	0.001			0.88	0.88
302	115.70	116.00	115.85	0.001			0.88	0.88
353	116.00	115.90	115.95	0.002			0.88	0.88
526	116.00	116.00	116.00	0.000			0.88	0.88
746	116.00	116.50	116.25	0.001			0.87	0.87

Table 8: 7075-HTPP at $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ sample 2

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	15.50	16.00	15.75		9.00	0.30	24.45	
30	35.00	38.00	36.50	0.692			6.86	15.65
78	46.00	48.00	47.00	0.219			4.48	5.67
126	55.50	57.50	56.50	0.198			3.25	3.87
174	62.00	67.30	64.65	0.170			2.56	2.91
198	66.00	68.00	67.00	0.098			2.40	2.48
222	75.00	75.00	75.00	0.333			1.96	2.18
257	81.00	81.00	81.00	0.171			1.70	1.83
302	94.00	97.00	95.50	0.322			1.26	1.48
353	115.0	114.0	114.50	0.373			0.90	1.08
526	116.0	115.0	115.50	0.006			0.88	0.89
746	116.0	116.0	116.00	0.002			0.88	0.88

Table 9: 7075-HTPP at $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ sample 1

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	18.50	19.00	18.75		9.00	0.38	24.29	
30	37.00	38.00	37.50	0.625			6.18	15.24
78	39.00	40.00	39.50	0.042			5.73	5.96
126	45.50	46.50	46.00	0.135			4.56	5.15
174	52.80	51.50	52.15	0.128			3.76	4.16
198	55.00	55.00	55.00	0.119			3.46	3.61
222	59.00	59.00	59.00	0.167			3.09	3.28
257	59.10	59.10	59.10	0.003			3.09	3.09
302	78.00	81.00	79.50	0.453			1.90	2.49
353	83.50	83.50	83.50	0.078			1.74	1.82
526	83.50	83.60	83.55	0.000			1.74	1.74
746	113.00	113.00	113.00	0.134			1.03	1.39
842	113.50	113.50	113.50	0.005			1.03	1.03
986	114.00	114.00	114.00	0.003			1.02	1.02
1178	114.50	114.50	114.50	0.003			1.01	1.01
1346	115.00	115.00	115.00	0.003			1.00	1.01

Table 10: 7075-HTPP at $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ sample 2

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	17.50	17.00	17.25		9.00	0.34	24.45	
30	33.00	32.00	32.50	0.508			9.39	16.92
78	46.00	43.00	44.50	0.250			5.57	7.48
126	55.00	55.00	55.00	0.219			3.86	4.72
174	63.2	60.50	61.85	0.143			3.14	3.50
198	64.00	66.00	65.00	0.131			2.87	3.01
222	70.00	69.00	69.50	0.188			2.55	2.71
257	80.00	80.00	80.00	0.300			1.98	2.26
302	90.00	90.00	90.00	0.222			1.59	1.78
353	98.00	98.00	98.00	0.157			1.36	1.48
526	113.0	112.5	112.7	0.085			1.05	1.20
746	115.0	115.0	115.0	0.010			1.01	1.03
842	115.0	115.5	115.2	0.003			1.01	1.01
986	115.5	115.5	115.5	0.002			1.00	1.00
1178	116.0	115.5	115.7	0.001			1.00	1.00
1346	116.0	116.0	116.0	0.001			0.99	1.00

Table 11: 7075-HTPP at $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ sample 1

Time (h)	a_0 (mm)	a_0 (mm)	Ave. a_0 (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	17.00	16.60	16.80		10.00	0.36	26.86	
30	17.00	16.70	16.85	0.002			26.75	26.80
78	17.00	16.90	16.95	0.002			26.53	26.64
126	17.00	17.00	17.00	0.001			26.42	26.47
174	19.00	19.00	19.00	0.042			22.58	24.50
198	20.00	20.00	20.00	0.042			20.97	21.78
222	23.00	23.00	23.00	0.125			17.06	19.02
257	25.00	25.00	25.00	0.057			15.03	16.05
302	27.00	27.00	27.00	0.044			13.35	14.19
353	28.00	28.00	28.00	0.020			12.61	12.98
526	33.00	32.00	32.50	0.025			9.95	11.65
746	33.00	34.00	33.50	0.005			9.47	9.71
842	34.00	35.00	34.50	0.010			9.03	9.25
986	37.00	37.00	37.00	0.017			8.04	8.53
1178	38.00	38.00	38.00	0.005			7.70	7.87
1346	40.00	43.00	41.50	0.021			6.64	7.17
1446	44.50	45.00	44.75	0.033			5.84	6.24

Table 12: 7075-HTPP at $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ sample 2

Time (h)	a_0 (mm)	a_0 (mm)	Ave. a_0 (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	16.30	16.00	16.15		9.00	0.36	28.35	
30	17.50	16.50	17.00	0.028			26.42	27.39
78	17.50	16.90	17.20	0.004			25.99	26.21
126	17.50	17.00	17.25	0.001			25.89	25.94
174	17.50	17.50	17.50	0.006			25.37	25.63
198	18.50	18.50	18.50	0.042			23.46	24.42
222	19.00	19.00	19.00	0.021			22.58	23.02
257	20.00	20.00	20.00	0.029			20.97	21.78
302	22.00	22.00	22.00	0.044			18.23	19.60
353	24.00	24.00	24.00	0.039			16.00	17.12
526	26.00	24.00	25.00	0.013			15.03	16.63
746	30.00	31.00	30.50	0.025			11.02	13.02
842	31.00	31.00	31.00	0.005			10.73	10.87
986	32.00	32.00	32.00	0.007			10.20	10.46
1178	32.50	33.00	32.75	0.004			9.82	10.01
1346	33.00	33.00	33.00	0.001			9.82	9.82
1446	40.00	41.00	40.50	0.075			6.92	8.37

Table 13: 7050-T6 sample 1

Time (h)	a _o (mm)	a _o (mm)	Ave. a _o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K _{ave} (MPa√m)
0	21.20	19.70	20.45		9.7	0.4	22.56	
30	46.30	45.30	45.80	0.845			6.24	14.40
78	54.30	54.30	54.30	0.177			4.65	5.44
126	65.80	65.80	65.80	0.240			3.31	3.98
174	74.30	76.30	75.30	0.198			2.59	2.95
198	80.30	79.30	79.80	0.188			2.33	2.46
222	86.30	85.30	85.80	0.250			2.04	2.19
257	102.30	102.30	102.30	0.471			1.48	1.76
302	114.30	114.30	114.30	0.267			1.20	1.34
353	115.30	115.30	115.30	0.020			1.18	1.19
526	116.00	116.00	116.00	0.004			1.17	1.18
746	116.50	116.50	116.50	0.002			1.16	1.16

Table 14: 7050-T6 sample 2

Time (h)	a _o (mm)	a _o (mm)	Ave. a _o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K _{ave} (MPa√m)
0	14.70	13.00	13.85		9.50	0.25	24.18	
30	44.50	45.50	45.00	1.038			4.02	14.10
78	52.50	54.50	53.50	0.177			2.98	3.50
126	62.00	63.50	62.75	0.193			2.25	2.62
174	72.00	70.50	71.25	0.177			1.79	2.02
198	75.00	74.50	74.75	0.146			1.64	1.72
222	77.5	77.50	77.50	0.115			1.54	1.59
257	82.50	80.50	81.50	0.114			1.40	1.47
302	94.50	95.00	94.75	0.294			1.06	1.23
353	113.00	113.00	113.00	0.358			0.77	0.92
526	116	116.00	116.00	0.017			0.73	0.75
746	116.50	116.50	116.50	0.002			0.72	0.73

Table 15: 7050-T6 sample 3

Time (h)	a _o (mm)	a _o (mm)	Ave. a _o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K _{ave} (MPa√m)
0	13.50	13.50	13.50		8.70	0.25	25.00	
30	43.30	43.30	43.30	0.993			4.29	14.65
78	51.30	51.30	51.30	0.167			3.21	3.76
126	59.80	62.80	61.30	0.208			2.35	2.78
174	69.30	70.80	70.05	0.182			1.85	2.10
198	70.80	73.30	72.05	0.083			1.76	1.80
222	77.30	77.30	77.30	0.219			1.55	1.65
257	77.30	77.60	77.45	0.004			1.54	1.54
302	84.50	84.00	84.25	0.151			1.32	1.43
353	88.30	88.30	88.30	0.079			1.21	1.27
526	116.00	116.00	116.00	0.160			0.73	0.97
746	116.50	116.50	116.50	0.002			0.72	0.73

Table 16: 7050-T7 sample 1

Time (h)	a _o (mm)	a _o (mm)	Ave. a _o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K _{ave} (MPa√m)
0	13.00	12.00	12.50		10.00	0.25	27.59	
30	14.00	13.00	13.50	0.033			25.00	26.29
78	14.00	13.50	13.75	0.005			24.41	24.71
126	14.00	14.00	14.00	0.005			23.84	24.134
174	14.00	14.20	14.10	0.002			23.62	23.73
198	14.00	14.30	14.15	0.002			23.51	23.57
222	14.00	14.40	14.20	0.002			23.40	23.46
257	14.00	14.50	14.25	0.001			23.30	23.35
302	14.20	14.50	14.35	0.002			23.08	23.19
526	18.00	16.00	17.00	0.011			18.35	20.71
746	17.30	17.30	17.30	0.001			17.91	18.13
842	17.50	17.50	17.50	0.002			17.62	17.76
986	18.00	18.00	18.00	0.003			16.94	17.28
1178	18.50	18.50	18.50	0.003			16.29	16.61
1346	19.30	19.30	19.30	0.005			15.33	15.81

Table 17: 7050-T7 sample 2

Time (h)	a _o (mm)	a _o (mm)	Ave. a _o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K _{ave} (MPa√m)
0	15.30	11.70	13.50		9.70	0.27	27.00	
30	15.30	12.30	13.80	0.010			26.24	26.62
78	15.30	12.70	14.00	0.004			25.75	26.00
126	15.30	13.30	14.30	0.006			25.04	25.40
174	15.30	14.70	15.00	0.015			23.50	24.27
198	15.30	15.30	15.30	0.013			22.89	23.19
222	15.30	15.70	15.50	0.008			22.49	22.69
257	15.30	16.30	15.80	0.009			21.91	22.20
302	15.30	16.50	15.90	0.002			21.72	21.82
526	22.30	18.30	20.30	0.020			15.39	18.56
746	25.30	22.30	23.80	0.016			12.15	13.77
842	25.50	22.50	24.00	0.002			12.00	12.08
986	26.30	23.00	24.65	0.005			11.52	11.76
1178	27.00	23.00	25.00	0.001			11.28	11.40
1346	23.30	27.30	25.30	0.002			11.07	11.17

Table 18: 7050-T7 sample 3

Time (h)	a _o (mm)	a _o (mm)	Ave. a _o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K _{ave} (MPa√m)
0	24.00	19.00	21.50		9.70	0.5	26.20	
30	25.00	20.00	22.50	0.033			24.49	25.35
78	25.50	21.00	23.25	0.016			23.31	23.90
126	26.00	21.30	23.65	0.008			22.72	23.02
174	26.20	22.00	24.10	0.009			22.08	22.40
198	26.50	23.00	24.75	0.027			21.20	21.64
222	26.80	25.00	25.90	0.048			19.77	20.49
257	27.00	25.20	26.10	0.006			19.54	19.66
302	27.10	25.20	26.15	0.001			19.48	19.51
526	27.20	25.30	26.25	0.000			19.37	19.43
746	27.30	25.30	26.30	0.000			19.31	19.34
842	27.50	26.00	26.75	0.005			18.81	19.06
986	28.00	26.30	27.15	0.003			18.38	18.60
1178	28.00	27.00	27.50	0.002			18.02	18.20
1346	28.30	28.30	28.30	0.005			17.22	17.62

Table 19: 7050-HTPP at $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ sample 1

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	16.00	12.00	14.00		9.00	0.25	23.84	
30	51.00	51.00	51.00	1.233			3.24	13.54
78	62.00	59.00	60.50	0.198			2.40	2.82
126	72.00	71.50	71.75	0.234			1.77	2.09
174	81.00	80.00	80.50	0.182			1.44	1.60
198	85.00	84.00	84.50	0.167			1.31	1.38
222	89.00	88.00	88.50	0.167			1.21	1.26
257	112.00	112.00	112.00	0.671			0.78	0.99
302	113.10	113.00	113.05	0.023			0.77	0.77
353	113.30	113.00	113.15	0.002			0.77	0.77
526	115.50	116.00	115.75	0.015			0.73	0.75
746	116.00	116.20	116.10	0.002			0.73	0.73
842	116.00	116.40	116.20	0.001			0.73	0.73
986	116.30	116.40	116.35	0.001			0.73	0.73
1178	116.40	116.4	116.40	0.000			0.73	0.73
1346	116.50	116.40	116.45	0.000			0.73	0.73

Table 20: 7050-HTPP at $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ sample 2

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	23.00	23.00	23.00		9.00	0.50	23.70	
30	46.00	47.00	46.50	0.783			7.60	15.65
78	54.00	53.00	53.50	0.146			5.96	6.78
126	57.00	54.50	55.75	0.047			5.55	5.76
174	73.00	70.00	71.50	0.328			3.56	4.55
198	73.00	73.00	73.00	0.063			3.43	3.50
222	74.00	74.00	74.00	0.042			3.35	3.39
257	75.00	74.00	74.50	0.014			3.31	3.33
302	76.00	74.00	75.00	0.011			3.27	3.29
353	79.00	79.00	79.00	0.078			2.97	3.12
526	96.00	96.00	96.00	0.098			2.08	2.53
746	114.00	113.00	113.50	0.080			1.52	1.80
842	114.00	113.50	113.75	0.003			1.52	1.52
986	114.50	114.00	114.25	0.003			1.50	1.51
1178	115.00	114.50	114.705	0.003			1.49	1.50
1346	115.00	115.00	115.00	0.001			1.48	1.49

Table 21: 7050-HTPP at $T_{F/Q} = 455\text{ }^{\circ}\text{C}$ sample 3

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	15.50	16.30	15.90		9.70	0.30	24.14	
30	38.30	38.30	38.30	0.747			6.33	15.23
78	45.30	47.70	46.50	0.171			4.56	5.45
126	51.8	54.80	53.30	0.142			3.60	4.08
174	64.00	65.00	64.50	0.233			2.57	3.09
198	65.00	68.00	66.50	0.083			2.43	2.50
222	68.00	67.00	67.50	0.042			2.37	2.40
257	69.30	69.30	69.30	0.051			2.26	2.32
302	73.80	73.3	73.55	0.094			2.03	2.15
353	74.30	74.30	74.30	0.015			1.99	2.01
526	89.30	89.30	89.30	0.087			1.43	1.71
746	114.00	114.00	114.00	0.112			0.91	1.17
842	114.50	114.50	114.50	0.005			0.89	0.90
986	115.00	114.50	114.75	0.002			0.89	0.89
1178	115.00	115.00	115.00	0.001			0.89	0.89
1346	115.00	115.20	115.10	0.001			0.89	0.89

Table 22: 7050-HTPP at $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ sample 1

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	16.00	15.50	15.75		9.00	0.30	24.45	
30	43.00	43.00	43.00	0.908			5.21	14.83
78	50.00	51.00	50.50	0.156			3.96	4.58
126	57.50	57.70	57.60	0.148			3.14	3.55
174	63.00	61.00	62.00	0.092			2.76	2.95
198	65.00	64.00	64.50	0.104			2.57	2.67
222	66.00	66.50	66.25	0.073			2.45	2.51
257	74.00	74.00	74.00	0.221			2.01	2.23
302	75.00	75.00	75.00	0.022			1.96	1.98
353	79.00	79.00	79.00	0.078			1.78	1.87
526	83.00	83.00	83.00	0.023			1.63	1.71
746	112.00	112.00	112.00	0.132			0.94	1.28
842	112.00	112.50	112.25	0.003			0.93	0.93
986	113.00	113.00	113.00	0.005			0.92	0.93
1178	114.00	114.00	114.00	0.005			0.91	0.91
1346	114.50	114.50	114.50	0.003			0.90	0.90
1446	115.00	115.00	115.00	0.005			0.89	0.89

Table 23: 7050-HTPP at $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ sample 2

Time (h)	a_0 (mm)	a_0 (mm)	Ave. a_0 (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{\text{m}}$)	K_{ave} (MPa $\sqrt{\text{m}}$)
0	21.00	22.00	21.50		10.00	0.48	25.15	
30	44.00	45.00	44.50	0.767			7.87	16.51
78	49.00	49.00	49.00	0.094			6.67	7.27
126	53.00	54.50	53.75	0.099			5.68	6.17
174	57.00	58.00	57.50	0.078			5.04	5.36
198	58.50	61.00	59.75	0.094			4.71	4.88
222	61.00	65.00	63.00	0.135			4.29	4.5
257	68.5	67.00	67.75	0.136			3.77	4.03
302	75.00	74.00	74.50	0.15			3.17	3.47
353	81.00	79.50	80.25	0.113			2.77	2.97
526	86.00	88.00	87.00	0.039			2.39	2.58
746	91.00	92.00	91.50	0.020			2.18	2.29
842	95.00	94.00	94.50	0.031			2.05	2.12
986	99.00	99.00	99.00	0.031			1.88	1.97
1178	110.00	110.00	110.00	0.057			1.55	1.72
1346	115.00	114.00	114.50	0.027			1.44	1.49
1446	115.00	115.00	115.00	0.005			1.43	1.43

Table 24: 7050-HTPP at $T_{F/Q} = 425\text{ }^{\circ}\text{C}$ sample 3

Time (h)	a_0 (mm)	a_0 (mm)	Ave. a_0 (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{\text{m}}$)	K_{ave} (MPa $\sqrt{\text{m}}$)
0	18.20	17.60	17.90		9.00	0.35	23.90	
30	39.80	40.80	40.30	0.747			6.78	15.34
78	47.80	47.80	47.80	0.156			5.07	5.93
126	52.30	53.30	52.80	0.104			4.27	4.67
174	57.30	57.30	57.30	0.094			3.70	3.99
198	59.80	59.80	59.80	0.104			3.43	3.57
222	62.80	62.80	62.80	0.125			3.15	3.29
257	65.80	65.80	65.80	0.086			2.89	3.02
302	68.80	68.80	68.80	0.067			2.67	2.78
353	70.80	70.80	70.80	0.039			2.54	2.61
526	93.80	93.80	93.80	0.133			1.52	2.03
746	95.80	95.8	95.80	0.009			1.46	1.49
842	96.50	96.80	96.65	0.009			1.44	1.45
986	97.00	97.80	97.40	0.005			1.42	1.43
1178	98.00	98.00	98.00	0.003			1.40	1.41
1346	98.00	98.80	98.40	0.002			1.46	1.43
1446	103.60	103.60	103.60	0.052			1.44	1.45

Table 25: 7050-HTPP at $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ sample 1

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	14.00	14.50	14.25		9.00	0.25	23.30	
30	38.00	39.00	38.50	0.808			5.23	14.26
78	44.00	45.00	44.50	0.125			4.10	4.66
126	47.50	48.50	48.00	0.073			3.60	3.85
174	54.50	54.50	54.50	0.135			2.89	3.24
198	57.00	57.00	57.00	0.104			2.67	2.78
222	58.00	58.00	58.00	0.042			2.59	2.63
257	62.00	62.00	62.00	0.114			2.30	2.44
302	63.00	63.00	63.00	0.022			2.23	2.27
353	68.00	68.00	68.00	0.098			1.95	2.09
526	80.00	77.00	78.50	0.061			1.50	1.73
746	84.00	81.00	82.50	0.018			1.37	1.44
842	89.00	87.00	88.00	0.057			1.22	1.30
986	90.00	89.00	89.50	0.010			1.183	1.20
1178	95.00	95.00	95.00	0.029			1.06	1.12
1346	101.0	101.00	101.0	0.03571			0.95	1.00
1446	104.0	104.00	104.0	0.03			0.90	0.92

Table 26: 7050-HTPP at $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ sample 2

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	15.20	19.50	17.35		9.50	0.35	24.97	
30	33.50	34.5	34.00	0.555			13.53	19.25
78	40.50	40.5	40.50	0.135			9.54	11.54
126	45.00	45.00	45.00	0.094			7.73	8.63
174	50.50	50.50	50.50	0.115			6.13	6.93
198	51.50	51.50	51.50	0.042			5.90	6.02
222	53.50	53.50	53.50	0.083			5.47	5.68
257	57.50	57.00	57.25	0.107			4.77	5.11
302	60.50	60.00	60.25	0.067			4.31	4.54
353	64.50	64.00	64.21	0.078			3.79	4.05
526	70.00	69.50	69.75	0.032			3.26	3.50
746	76.50	76.00	76.25	0.030			2.69	2.95
842	76.50	77.00	76.75	0.005			2.66	2.67
986	79.00	78.50	78.75	0.014			2.52	2.59
1178	85.00	85.00	85.00	0.033			2.17	2.34
1346	89.00	89.50	89.25	0.025			1.96	2.06
1446	93.40	93.40	93.40	0.042			1.79	1.88

Table 27: 7050-HTPP at $T_{F/Q} = 405\text{ }^{\circ}\text{C}$ sample 3

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K_{ave} (MPa√m)
0	13.00	16.00	14.50		9.00	0.27	24.59	
30	34.00	35.00	34.50	0.667			6.77	15.68
78	41.00	41.00	41.00	0.135			5.08	5.93
126	44.00	44.00	44.00	0.063			4.51	4.80
174	49.00	49.00	49.00	0.104			3.75	4.13
198	51.00	51.00	51.00	0.083			3.50	3.63
222	51.00	51.50	51.25	0.010			3.47	3.49
257	54.00	54.00	54.00	0.079			3.17	3.32
302	56.00	56.00	56.00	0.044			2.97	3.07
353	60.00	60.00	60.00	0.078			2.63	2.81
526	65.00	65.00	65.00	0.029			2.28	2.46
746	71.00	71.00	71.00	0.027			1.95	2.12
842	75.00	75.00	75.00	0.042			1.76	1.86
986	77.00	76.00	76.50	0.010			1.70	1.73
1178	79.00	79.00	79.00	0.013			1.61	1.65
1346	82.00	82.50	82.25	0.019			1.49	1.55
1446	90.00	90.00	90.00	0.078			1.26	1.37

Table 28: 7050-HTPP at $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ sample 1

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K_{ave} (MPa√m)
0	19.00	19.00	19.00		9.50	0.40	25.09	
30	35.50	35.50	35.50	0.550			6.98	16.03
78	40.50	39.50	40.00	0.094			5.87	6.42
126	45.00	45.00	45.00	0.104			4.92	5.39
174	48.00	48.00	48.00	0.063			4.46	4.69
198	51.50	52.50	52.00	0.167			3.94	4.20
222	51.50	53.00	52.25	0.010			3.91	3.93
257	55.50	55.50	55.50	0.093			3.56	3.74
302	57.00	57.50	57.25	0.039			3.39	3.48
353	58.00	58.50	58.25	0.020			3.30	3.34
526	59.00	59.00	59.00	0.004			3.23	3.26
746	60.00	60.00	60.00	0.005			3.15	3.19
842	62.00	61.00	61.50	0.016			3.02	3.08
986	62.50	62.00	62.25	0.006			2.96	2.99
1178	63.00	63.00	63.00	0.004			2.91	2.94
1346	63.50	63.50	63.50	0.003			2.87	2.89
1446	68.80	68.80	68.80	0.053			2.52	2.70

Table 29: 7050-HTPP at $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ sample 2

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	15.00	20.00	17.50		8.50	0.35	24.67	
30	34.50	35.50	35.00	0.583			8.57	16.62
78	38.50	40.50	39.50	0.094			7.01	7.79
126	40.50	41.50	41.00	0.0313			6.59	6.80
174	44.50	43.50	44.00	0.063			5.85	6.22
198	49.50	49.50	49.50	0.229			4.78	5.31
222	51.00	51.00	51.00	0.063			4.54	4.66
257	57.50	57.50	57.50	0.186			3.68	4.11
302	58.50	58.50	58.50	0.022			3.57	3.62
353	59.00	58.50	58.75	0.005			3.54	3.55
526	59.00	59.00	59.00	0.001			3.51	3.53
746	60.00	60.00	60.00	0.005			3.41	3.46
842	61.00	61.00	61.00	0.010			3.31	3.36
986	62.00	62.00	62.00	0.007			3.22	3.27
1178	63.00	63.00	63.00	0.005			3.13	3.17
1346	63.50	63.00	63.25	0.001			3.11	3.12
1446	68.70	68.70	68.70	0.055			2.68	2.89

Table 30: 7050-HTPP at $T_{F/Q} = 370\text{ }^{\circ}\text{C}$ sample 3

Time (h)	a_o (mm)	a_o (mm)	Ave. a_o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa $\sqrt{m}$)	K_{ave} (MPa $\sqrt{m}$)
0	16.00	17.20	16.60		9.80	0.33	25.03	
30	31.20	32.20	31.70	0.503			9.49	17.26
78	38.20	39.20	38.70	0.146			6.84	8.17
126	40.20	40.20	40.20	0.031			6.42	6.63
174	45.20	44.20	44.70	0.094			5.37	5.89
198	48.20	49.20	48.70	0.167			4.63	5.00
222	50.20	50.20	50.20	0.063			4.40	4.52
257	54.20	55.20	54.70	0.129			3.79	4.09
302	54.20	56.20	55.20	0.011			3.73	3.76
353	55.20	56.20	55.70	0.010			3.67	3.70
526	57.20	58.20	57.70	0.012			3.45	3.56
746	59.20	59.20	59.20	0.007			3.29	3.37
842	60.20	61.20	60.70	0.016			3.15	3.22
986	63.20	63.20	63.20	0.017			2.93	3.04
1178	64.20	64.20	64.20	0.005			2.85	2.89
1346	66.20	66.20	66.20	0.012			2.70	2.78
1446	70.20	70.20	70.20	0.040			2.43	2.56

Table 31: 7050 T74-1

Time (h)	a _o (mm)	a _o (mm)	Ave. a _o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K _{ave} (MPa√m)
0	11.80	12.30	12.05		9.70	0.25	28.89	
30	12.20	12.80	12.50	0.015			27.59	28.24
78	12.20	13.10	12.65	0.003			27.17	27.38
126	12.20	13.20	12.70	0.001			27.04	27.11
174	12.30	13.20	12.75	0.001			26.90	26.97
198	12.30	13.30	12.80	0.002			26.77	26.84
222	12.30	13.35	12.83	0.001			26.70	26.74
257	13.00	13.30	13.15	0.009			25.86	26.28
302	13.25	13.30	13.28	0.003			25.55	25.71
353	13.30	13.30	13.30	0.000			25.49	25.52
526	13.30	13.40	13.35	0.000			25.36	25.43
746	13.30	13.45	13.375	0.000			25.30	25.33
842	13.40	13.50	13.45	0.001			25.12	25.21
986	13.50	13.50	13.50	0.000			25.00	25.06

Table 32: 7050 T74-2

Time (h)	a _o (mm)	a _o (mm)	Ave. a _o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K _{ave} (MPa√m)
0	14.50	14.70	14.60		9.30	0.30	27.07	
30	14.70	15.50	15.10	0.017			25.88	26.48
78	14.70	15.60	15.15	0.001			25.77	25.83
126	14.70	15.70	15.20	0.001			25.65	25.72
174	14.70	15.70	15.20	0			25.65	25.65
198	14.70	15.70	15.20	0			25.65	25.65
222	14.70	15.70	15.20	0			25.65	25.65
257	15.70	15.70	15.70	0.014			24.56	25.12
302	15.70	15.70	15.70	0			24.56	24.56
353	15.70	15.70	15.70	0			24.56	24.56
526	15.70	15.70	15.70	0			24.56	24.56
746	15.70	15.70	15.70	0			24.56	24.56
842	16.00	16.00	15.70	0			23.93	24.24
986	16.00	16.00	16.00	0.002			23.93	23.93

Table 33: 7050-T74 sample 3

Time (h)	a _o (mm)	a _o (mm)	Ave. a _o (mm)	da/dt (mm/h)	Co (mm)	Vo (mm)	K (MPa√m)	K _{ave} (MPa√m)
0	11.60	11.60	11.60		9.40	0.24	29.07	
30	12.60	12.60	12.60	0.033			26.22	27.65
78	12.60	12.60	12.60	0			26.22	26.22
126	12.60	12.60	12.60	0			26.22	26.22
174	12.60	12.60	12.60	0			26.22	26.22
198	12.60	12.60	12.60	0			26.22	26.22
222	12.60	12.60	12.60	0			26.22	26.22
257	12.60	12.60	12.60	0			26.22	26.22
302	12.60	12.60	12.60	0			26.22	26.22
353	12.60	12.60	12.60	0			26.22	26.22
526	12.60	12.60	12.60	0			26.22	26.22
746	12.60	12.6	12.60	0			26.22	26.22
842	12.80	12.60	12.70	0.001			25.96	26.09
986	12.80	12.60	12.70	0			25.96	25.96

APPENDIX D: EDS ANALYSIS DATA

Table 1: 7075-T6

Distance from grain boundary (nm) Point 1	Zn (wt%)	Mg (wt%)	Cu (wt%)
10	0	0	9.22
5	0	2.48	7.37
0	0	2.58	6.69
-5	9.99	4.17	0
-10	0	3.19	6.25
Point 2			
10	0	0	7.05
5	0	3.46	6.48
0	0	0	6.65
-5	0	0	9.18
-10	5.45	0	8.43

Table 2: 7075-T7

Distance from grain boundary (nm) Point 1	Zn (wt%)	Mg (wt%)	Cu (wt%)
10	1.31	0	5.3
5	0	0	5.54
0	2.45	0	7.38
-5	0	0	6.59
-10	1.44	0	5.49
Point 2			
10	3.25	0	5.27
5	4.25	0	5.45
0	3.54	2.18	5.84
-5	4.54	1.99	6.48
-10	4.5	1.92	5.9

Table 3: 7075-HTPP at $T_{F/Q} = 455\text{ }^{\circ}\text{C}$

Distance from grain boundary (nm) Point 1	Zn (wt%)	Mg (wt%)	Cu (wt%)
10	0	0	7.64
5	0	0	5.42
0	0	0	7.24
-5	0	0	7.49
-10	3.45	0	6
Point 2			
10	0	3.62	6.41
5	0	0	6.69
0	0	0	6.11
-5	0	0	6.52
-10	0	0	7.63

Table 4: 7075-HTPP at $T_{F/Q} = 425\text{ }^{\circ}\text{C}$

Distance from grain boundary (nm) Point 1	Zn (wt%)	Mg (wt%)	Cu (wt%)
10	6.97	0	5.72
5	4.73	0	4.81
0	5.46	3.3	6.31
-5	14.8	0	8.21
-10	14.75	3.93	6.36
Point 2			
10	5.46	2.97	7.38
5	3.92	2.67	7.78
0	4.13	2.53	6.15
-5	5.89	2.65	5.77
-10	5.35	0	5.81

Table 5: 7075-HTPP at $T_{F/Q} = 405\text{ }^{\circ}\text{C}$

Distance from grain boundary (nm) Point 1	Zn (wt%)	Mg (wt%)	Cu (wt%)
10	3.83	3.17	5.75
5	6.23	3.05	5.76
0	12.72	2.86	5.27
-5	9.79	3.24	6.52
-10	6.17	0	3.85
Point 2			
10	5.4	4.3	4.64
5	10.69	0	5.29
0	9.39	0	5.29
-5	7.11	0	5.87
-10	6.75	4.25	5.67

Table 6: 7075-HTPP at $T_{F/Q} = 370\text{ }^{\circ}\text{C}$

Distance from grain boundary (nm) Point 1	Zn (wt%)	Mg (wt%)	Cu (wt%)
10	3.71	2.29	3.47
5	3.8	2.66	3.79
0	3.08	3.31	3.24
-5	2.65	2.5	3.44
-10	2.15	2.82	3.35
Point 2			
10	2.91	2.34	4.03
5	2.72	2.87	3.34
0	3.24	3.3	3.74
-5	8.22	3.4	3.93
-10	4.6	3.42	4.08

Table 7: 7050-T6

Distance from grain boundary (nm) Point 1	Zn (wt%)	Mg (wt%)	Cu (wt%)
10	6.94	0	5.73
5	4.57	0	5.82
0	3.34	0	5.83
-5	0	0	7.26
-10	0	0	7.3
Point 2			
10	8.56	0	5.14
5	6.74	0	7.25
0	0	0	0
-5	6.6	0	6.09
-10	0	0	7.89

Table 8: 7050-T7

Distance from grain boundary (nm) Point 1	Zn (wt%)	Mg (wt%)	Cu (wt%)
10	1.94	2.22	3.3
5	1.32	2.82	3.94
0	0	2.03	2.61
-5	1.18	2.38	3.64
-10	0	2.49	3.69
Point 2			
10	1.98	2.5	3.45
5	1.5	2	3.2
0	0.97	1.76	3.12
-5	1.19	1.58	3.33
-10	1.91	2.56	3.86

Table 9: 7050-HTPP at $T_{F/Q} = 455\text{ }^{\circ}\text{C}$

Distance from grain boundary (nm) Point 1	Zn (wt%)	Mg (wt%)	Cu (wt%)
10	6.62	0	5.82
5	0	3.68	6.87
0	0	0	7.33
-5	3.19	2.31	6.74
-10	0	0	7.47
Point 2			
10	4.47	0	7.6
5	5.13	0	6.53
0	0	0	6.53
-5	5.97	0	7.02
-10	6.73	2.91	6.76

Table 10: 7050-HTPP at $T_{F/Q} = 425\text{ }^{\circ}\text{C}$

Distance from grain boundary (nm) Point 1	Zn (wt%)	Mg (wt%)	Cu (wt%)
10	2.14	4.71	4.19
5	1.29	3.8	5.34
0	10.04	0	5.74
-5	3.7	4.19	5.87
-10	5.85	3.47	5.7
Point 2			
10	3.01	3.12	5.53
5	2.67	4.16	4.71
0	6.04	3.61	6.13
-5	3.62	0	6.82
-10	4.9	3.7	4.53

Table 11: 7050-HTPP at $T_{F/Q} = 405\text{ }^{\circ}\text{C}$

Distance from grain boundary (nm) Point 1	Zn (wt%)	Mg (wt%)	Cu (wt%)
10	9.53	0	4.03
5	7.33	0	3.77
0	9.58	0	3.41
-5	4.89	0	3.83
-10	4.54	2.67	3.73
Point 2			
10	5.72	0	3.59
5	5.18	2.99	3.79
0	4.27	2.65	4.49
-5	5.43	0	3.71
-10	4.95	0	4.36

Table 12: 7050-HTPP at $T_{F/Q} = 370\text{ }^{\circ}\text{C}$

Distance from grain boundary (nm) Point 1	Zn (wt%)	Mg (wt%)	Cu (wt%)
10	1.98	0	4.91
5	2.21	3.01	4.58
0	2.66	0	4.96
-5	1.7	0	5.03
-10	2.15	0	4.95
Point 2			
10	1.41	2.02	3.99
5	1.82	0	3.43
0	2.43	0	3.61
-5	1.69	2.57	3.37
-10	2.1	0	3.47