1

52753

National Library
of CanadaBibliothèque nationale
du CanadaCANADIAN THESES
ON MICROFICHETHÈSES CANADIENNES
SUR MICROFICHÉ

0-315-04859-X

NAME OF AUTHOR/NOM DE L'AUTEUR JONATHAN CARL BEDDOES

TITLE OF THESIS/TITRE DE LA THÈSE ENVIRONMENTALLY ENHANCED FRACTURE
OF ALUMINUM ALLOYS

UNIVERSITY/UNIVERSITÉ CARLETON UNIVERSITY

DEGREE FOR WHICH THESIS WAS PRESENTED/
GRADE POUR LEQUEL CETTE THÈSE FUT PRÉSENTÉE M. ENG.

YEAR THIS DEGREE CONFERRED/ANNÉE D'OBTENTION DE CE DEGRÉ 1981

NAME OF SUPERVISOR/NOM DU DIRECTEUR DE THÈSE DR. W. WALLACE

Permission is hereby granted to the NATIONAL LIBRARY OF
CANADA to microfilm this thesis and to lend or sell copies
of the film.

The author reserves other publication rights, and neither the
thesis nor extensive extracts from it may be printed or other-
wise reproduced without the author's written permission.

*L'autorisation est, par la présente, accordée à la BIBLIOTHÈ-
QUE NATIONALE DU CANADA de microfilmer cette thèse et
de prêter ou de vendre des exemplaires du film.*

*L'auteur se réserve les autres droits de publication; ni la
thèse ni de longs extraits de celle-ci ne doivent être imprimés
ou autrement reproduits sans l'autorisation écrite de l'auteur.*

DATED/DATE OCT. 7 1981 SIGNED/SIGNÉ J. Beddoes

PERMANENT ADDRESS/RÉSIDENCE FIXE

2. DALE ST.KINGSTON, ONTARIOK7M 4H9

National Library of Canada
Collections Development Branch

Canadian Theses on
Microfiche Service

Bibliothèque nationale du Canada
Direction du développement des collections

Service des thèses canadiennes
sur microfiche

NOTICE

The quality of this microfiche is heavily dependent upon the quality of the original thesis submitted for microfilming. Every effort has been made to ensure the highest quality of reproduction possible.

If pages are missing, contact the university which granted the degree.

Some pages may have indistinct print especially if the original pages were typed with a poor typewriter ribbon or if the university sent us a poor photocopy.

Previously copyrighted materials (journal articles, published tests, etc.) are not filmed.

Reproduction in full or in part of this film is governed by the Canadian Copyright Act, R.S.C. 1970, c. C-30. Please read the authorization forms which accompany this thesis.

**THIS DISSERTATION
HAS BEEN MICROFILMED
EXACTLY AS RECEIVED**

AVIS

La qualité de cette microfiche dépend grandement de la qualité de la thèse soumise au microfilmage. Nous avons tout fait pour assurer une qualité supérieure de reproduction.

S'il manque des pages, veuillez communiquer avec l'université qui a conféré le grade.

La qualité d'impression de certaines pages peut laisser à désirer, surtout si les pages originales ont été dactylographiées à l'aide d'un ruban usé ou si l'université nous a fait parvenir une photocopie de mauvaise qualité.

Les documents qui font déjà l'objet d'un droit d'auteur (articles de revue, examens publiés, etc.) ne sont pas microfilmés.

La reproduction, même partielle, de ce microfilm est soumise à la Loi canadienne sur le droit d'auteur, SRC 1970, c. C-30. Veuillez prendre connaissance des formules d'autorisation qui accompagnent cette thèse.

**LA THÈSE A ÉTÉ
MICROFILMÉE TELLE QUE
NOUS L'AVONS REÇUE**

ENVIRONMENTALLY ENHANCED FRACTURE
OF HIGH STRENGTH ALUMINUM ALLOYS

by

JONATHAN CARL BEDDOES, B.ENG.

A Thesis

Submitted to the School of Graduate Studies

in Partial Fulfilment of the Requirements

for the Degree

Master of Engineering

CARLETON UNIVERSITY

August 1981

The undersigned hereby recommend to the Faculty
of Graduate Studies acceptance of this thesis, submitted
by Jonathan C. Beddoes, B.Eng., in partial fulfilment of
the requirements for the degree of Master of Engineering.

W. Wallace

Adjunct Professor
Dept. Mechanical and
Supervisor *Aeronautical Eng.*
for Professor H. C. de Mallerbe

J. L. Smith

Chairman, Dept. of Mech. &
Aero. Engineering

ABSTRACT

The stress corrosion failure of Grumman CS2F Tracker Aircraft wing-lock fittings is described. This failure exemplifies the problem of stress corrosion and corrosion fatigue in high strength aluminum alloys. In this thesis the nature of stress corrosion, corrosion fatigue and the effects of microstructural features on environmentally enhanced cracking of 7000 series aluminum alloys are reviewed. The question of whether corrosion fatigue is caused by cyclic stress corrosion is considered. A hydrogen embrittlement mechanism for stress corrosion crack growth is expounded.

The benefits of a new heat treatment option for 7075, retrogression and reaging, are discussed. The limits of retrogression and reaging, with respect to stress corrosion resistance and tensile strength of 7075 extruded bar and plate is determined by tensile tests, electrical conductivity measurements and stress corrosion crack growth rate tests. The results of electron microscopy of variously retrogressed and reaged heat treated samples are presented and correlated with the observed mechanical properties.

ACKNOWLEDGEMENTS

I would like to express my sincere appreciation for the encouragement and guidance of my supervisor, Dr. M.C. de Malherbe.

I particularly want to thank Dr. W. Wallace of the Structures and Materials Laboratory, National Research Council of Canada, for the invaluable technical help provided and the day to day supervision of this thesis. Through his cooperation it has been possible to perform the experimental work using the facilities of the National Research Council.

The assistance that Drs. R.L. Hewitt and K. Rajan provided for various aspects of the work is acknowledged. As well, thanks go to G. McPhee, T. Steel, T. Terada, and K. Allen for their help.

The financial assistance provided by Carleton University and the National Research Council of Canada has been appreciated.

Lastly, but most importantly, the continuous patience and support given by my wife, Judy, will always be remembered. As well, her hours spent typing the manuscript are very greatly appreciated.

TABLE OF CONTENTS

<u>ABSTRACT</u>	iii
<u>ACKNOWLEDGEMENTS</u>	iv
<u>LIST OF TABLES</u>	vii
<u>LIST OF FIGURES</u>	viii
<u>LIST OF APPENDICES</u>	xii
<u>1. INTRODUCTION</u>	1
<u>2. FRACTURE BY FATIGUE AND STRESS CORROSION</u>	3
2.1 General Nature of Fatigue Crack Growth in Aluminum Alloys	3
2.1.1 Environmental Effects	6
2.2 General Nature of Stress Corrosion Crack Growth in Aluminum Alloys	8
2.3 Microstructural Effects on Fracture of 7075	11
2.3.1 Constituent Particles	11
2.3.2 Dispersoid Particles	12
2.3.3 Precipitate Particles	12
<u>3. CORROSION FATIGUE - STRESS CORROSION INTERACTION</u>	19
3.1 Introduction	19
3.2 Linear Summation Model	20
3.3 Superposition Model	22
3.4 Process Competition Model	26
3.5 Comparison of Models	29
3.5.1 Crack Propagation by a Dual Process	30
3.5.2 Crack Propagation by a Single Process	34
3.6 Summary	37
<u>4. A MECHANISM FOR STRESS CORROSION CRACKING</u>	40
<u>5. RETROGRESSION AND REAGING</u>	47
<u>6. EXPERIMENTAL OUTLINE AND TECHNIQUE</u>	51

6.1 Outline of Study	51
6.2 Experimental Technique and Materials	53
6.2.1 Tensile Tests	53
6.2.2 Electrical Conductivity Measurements	54
6.2.3 Stress Corrosion Tests	54
6.2.4 Tracker Tests	58
6.2.5 Electron Microscopy	59
<u>7. RESULTS AND DISCUSSION</u>	60
7.1 Tracker Tests	60
7.2 Extruded Bar Material	61
7.2.1 Tensile Results	61
7.2.2 Electrical Conductivity Results	63
7.3 Plate Material	65
7.3.1 Tensile Results	66
7.3.2 Electrical Conductivity Results	68
7.3.3 Stress Corrosion Results	70
7.3.4 Fracture Toughness Results	72
7.4 Electron Microscopy Results	76
<u>8.0 CONCLUSIONS</u>	83
<u>REFERENCES</u>	86
<u>TABLES</u>	94
<u>FIGURES</u>	102
<u>APPENDICES</u>	154

LIST OF TABLES

- Table I Compositions of Some 7000 Series Aluminum Alloys.
- Table II Mechanical Properties of Some 7000 Series
Aluminum Alloys.
- Table III Heat Treatment Options For 7075.
- Table IV Stress Corrosion Results - Cina ⁵⁹.
- Table V Tensile Results of Samples Machined from Used
Double Cantilever Beam Specimens.
- Table VI Results of Samples Machined from 2.5 cm 7075 Plate.
- Table VII Electrical Conductivity of Heat Treated 2.5 cm
7075 Plate.
- Table VIII Short Transverse Fracture Toughness Results.

LIST OF FIGURES

1. The Grumman CS2F Tracker Aircraft, used in Canada for coastal surveillance.
2. a) Typical Tracker wing-lock attachment fittings.
b) Profile of stress corrosion cracking in wing-lock fitting shown in a.
3. Microstructure of the Tracker 7075-T6 plate wing-lock attachment fittings.
4. Fracture surface of Tracker wing-lock attachment fitting.
5. Standard compact-tension specimen for fatigue crack growth rate testing.
6. Schematic plot of a typical fatigue crack growth rate versus stress intensity plot for high strength materials
7. Diagrammatic representation of plane strain and plane stress crack growth in a compact-tension specimen.
8. Schematic showing the effects of corrosive environments on the position of a fatigue crack growth rate versus stress intensity curve.
9. Fatigue crack growth rate versus stress intensity range curve for 7075-T6 and T73 sheet in air and salt environments.
10. Fractograph showing various fatigue striation morphologies for 7075-T6.
11. Time-to-failure stress corrosion curve for 7075-T6 plate.
12. Schematic representation of a typical stress corrosion crack growth rate versus stress intensity plot for high strength aluminum alloys.
13. The effect of aging time or higher aging temperatures on the strength and stress corrosion resistance of 7075.
14. Computation procedure to obtain total crack growth.

15. Schematic of fatigue load cycles where (a) stress corrosion can occur throughout the whole load cycle, (b) stress corrosion occurs during a portion of the load cycle.
16. Schematic comparison of the crack growth curves for the three models for environmentally enhanced fatigue crack growth.
17. The effect of frequency on stress corrosion fatigue.
18. Results for Ti-8Al-1Mo-1V showing similar effects as figure 17.
19. a) Results similar to those of figure 17 and 18 for an aluminum alloy.
b) Results for the same material as (a) indicating a strong frequency effect at low stress intensities.
20. Electron micrograph of Ti-8Al-1Mo-1V, showing cleavage typical of cracking at 0.5 Hz in 3.5% NaCl solution at stress intensities above K_{Isc} .
21. Electron micrograph of Ti-8Al-1Mo-1V at stress intensities below K_{Isc} in 3.5% NaCl tested at 30 Hz.
22. Corrosion fatigue crack growth rates of 12Ni-5Cr-3Mo steel tested in 3% NaCl under various load profiles.
23. The effect of rise time on fatigue crack growth rates for A533B-1 steel tested at ΔK of 50 MPa $\sqrt{m}$ and a stress ratio of 0.05.
24. Effect of strain rate on susceptibility to stress corrosion cracking.
25. The effect of exposing Al-6%Zn-3%Mg to water vapour saturated air at 70°C for 72 hours after aging at various temperatures for 24 hours.
26. A model for stress corrosion crack growth.
27. Schematic representation of the changes in hardness during retrogression and reaging.
28. Hardness results of retrogressed 7075-T6 sheet obtained by Cina⁵⁹.

29. Typical stress corrosion crack growth rate curve.
30. Geometry of tensile specimens used for this thesis.
31. Geometry of double cantilever beam stress corrosion samples machined from 2.5 cm (1") 7075 plate.
32. Microstructure of 2.5 cm plate (7075-T651) used in this thesis.
33. Schematic showing the set-up used to determine the heat-up times to the retrogression temperatures of 180°C of double cantilever beam midsections.
34. Plot showing the decrease in stress intensity with increasing crack length for a double cantilever beam specimen.
35. Fracture surface of a broken open double cantilever beam stress corrosion sample.
36. Plot showing the effect of correcting stress corrosion crack growth rate results for crack tunnelling.
37. Photo showing 1 mm grid lines photoetched onto a double cantilever beam specimen.
38. Tensile yield strength of retrogressed 7075-T6 for retrogression times up to 60 minutes.
39. Yield strength of 7075-T6 retrogressed at 200°C for up to 5 hours and reaged.
40. Electrical conductivity of 7075-T6 retrogressed at 180°C for up to one hour and reaged.
41. Electrical conductivity of 7075-T6 retrogressed at 160°C , 180°C , 200°C , and 220°C for up to one hour.
42. The relative positions of short, intermediate and long time retrogression and reaging heat treatments, on the hardness or yield strength retrogression and reaging curves.
43. Stress corrosion crack growth rate versus stress intensity plots, for "short time" RRA treated double cantilever beam specimens.
44. Stress corrosion crack growth rate versus stress intensity plots, for "intermediate and long time" RRA treated double cantilever beam specimens.

45. Plot of fracture toughness of heat treated 7075 plate versus electrical conductivity.
46. a) Schematic showing variation of plastic zone size across a compact-tension or double cantilever beam specimen.
b) Drawing showing relative sizes of plane strain and plane stress plastic zones.
47. a) Profile of "pop-in" crack in a 7075-T6 double cantilever beam specimen.
b) Profile of a "pop-in" crack in a double cantilever beam specimen heat treated (T6+8h/180°C+RA) to a high toughness.
48. Electron micrograph of extruded bar 7075-T6.
49. a) Electron micrograph of 7075-T6 extruded bar retrogressed for 5 minutes at 220°C.
b) Electron micrograph of 7075-T6 extruded bar retrogressed as in a, plus 29 hours at 121°C.
50. Electron micrograph showing structure of 7075-T73 extruded bar.
51. Two examples of molecular hydrogen along grain boundaries in 7075-T6 extruded bar retrogressed for 5 minutes at 220°C and reaged.
52. Elastic strain field fringes along grain boundaries of artificially aged Al-4.4%Zn-1.5%Mg that have been attributed to hydrogen bubbles.

LIST OF APPENDICES

- A. Double Cantilever Beam Data Reduction.
- B. Procedure to Photoetch Grids Onto Double Cantilever Beam Specimens.
- C. Thin-Foil Preparation.
- D. Tensile Test Results.
- E. C-Ring Stress Corrosion Testing and Results.

1. INTRODUCTION

High strength 7000 series aluminum alloys, such as 7075-T6, have been used for aircraft structural parts for over twenty-five years. With the advent of the damage-tolerant design philosophy, the response to stress corrosion cracking and corrosion fatigue cracking of 7000 series aluminum alloys, as well as other high strength alloys, has become of interest. Although stress corrosion and corrosion fatigue have received widespread attention over the years, they are still problems that are encountered, as the following example demonstrates.

The wing-lock attachment fittings of the Grumman CS2F Tracker aircraft, figure 1, cracked because of stress corrosion. The aircraft is used in Canada for maritime patrol and consequently is exposed to corrosive salt laden atmospheres. The wing-lock fittings, figure 2a, are machined from 7075-T6 plate and stress corrosion cracking occurred in a plane perpendicular to the short-transverse direction, figure 2b. This is the most stress corrosion susceptible orientation in plate material because of the elongated "pancake" shaped grains, figure 3, that result from the rolling operation. The stress corrosion fracture, figure 4, has the typical flat almost featureless appearance resulting from intergranular failure of the flat "pancake" grains. It

was this particular failure that led to some of the work described in this thesis. The Tracker failure described is typical of many stress corrosion problems. Failures under similar conditions have occurred as a result of corrosion fatigue ¹.

In this thesis the effects of microstructure on the environmentally enhanced fracture behaviour of 7075 are reviewed. Several proposed models for corrosion fatigue are outlined and the role of stress corrosion in the corrosion fatigue process discussed. A heat treatment, retrogression and reaging, which deviates from the aging-overaging treatments commercially used for 7075, is described. Its potential to shed light on the metallurgical factors required for good stress corrosion, and the corrosion fatigue-stress corrosion interaction is indicated. The experimental work concentrates on the more practical aspects of retrogression and reaging. That is, determining the limits of the process with respect to yield strength, stress corrosion and fracture toughness properties. Some of the microstructural changes occurring during retrogression and reaging are determined and correlated with the observed mechanical properties.

2. FRACTURE BY FATIGUE AND STRESS CORROSION

2.1 General Nature of Fatigue Crack Growth in Aluminum Alloys

Fatigue has long been recognized as a major cause of failure of metals. Consequently, it has been the topic of a vast amount of research. Due to the present use of the damage tolerant design philosophy for aircraft structures, interest is now being focused on fatigue crack growth rates. This section reviews the general nature of fatigue crack growth.

Typically, plots of fatigue crack growth rate (da/dN) versus the stress intensity range (ΔK) are generated by testing precracked center cracked tension or compact tension specimens, figure 5. Typically these plots have three distinct stages of crack growth, as shown in figure 6. Also, because of the sigmoidal shape of the da/dN vs ΔK plots two asymptotic values exist. At low ΔK values the fatigue crack growth rate approaches a threshold stress intensity (ΔK_0), below which no growth occurs. At high crack growth rates the stress intensity approaches the critical plane strain fracture toughness (K_{Ic}), above which unstable fracture occurs.

The threshold stress intensity is the minimum ΔK at which fatigue crack growth occurs. Below the threshold the crack tip

may remain closed during the whole fatigue cycle, resulting in no growth ². Threshold measurements may be very time consuming because of the slow growth rates involved, hence an "operational" definition is often used. The ΔK at which the fatigue crack growth rate is 10^{-10} m/cycle ^{3,4} or the highest ΔK at which no growth can be detected in 10^7 cycles ² have been used as "operational thresholds". The ΔK_0 is affected significantly by microstructural, environmental and loading variables.

Stage I fatigue crack growth has been reported by some to be strongly influenced by microstructure and mean stress ^{2,3,5} and by others to be independent of microstructure and also composition ⁶. At the low stage I stress intensities growth rates often correspond to less than one lattice spacing per cycle. Since the minimum crack growth rate possible is thought to be one interatomic spacing per cycle ³, the crack front must advance in a discontinuous manner. Consequently, growth rates may be dominated by local microstructural features, such as the constituent particles discussed in a latter section.

In unnotched specimens, stage I cracks propagate along favourable crystallographic planes at approximately 45° to the applied load (plane of maximum shear stress), by some unslipping or reverse slip process ⁷. As the crack extends shear growth

becomes more difficult because of the increasing tensile component (the crack causes a decrease in cross sectional area). This causes a transition to a crack approximately normal to the applied stress which marks the beginning of stage II crack growth ⁸.

For notched and precracked specimens stage II crack growth accounts for the largest percentage of the total fatigue life and corresponds to the linear portion of the da/dN versus ΔK curve. Microstructure and composition influence stage II crack growth in a very limited manner only ^{2, 6, 7}. Hence stage II crack growth can be modelled empirically by the Paris equation ⁹:

$$\frac{da}{dN} = m(\Delta K)^n \quad \dots (1)$$

where m and n are constants determined from experimental data.

During stage II crack growth in thick specimens, a plane strain stress state would usually predominate at the crack tip. As the plastic zone size approaches the same dimensions as the thickness, yielding in the thickness direction occurs, causing a transition to plane stress and stage III cracking. Since the thickness affects the plane strain to plane stress transition it is an important test parameter. The plane strain condition results in a slower crack growth rate because it is a more constrained condition, since there is no yielding

in the thickness direction ⁹.

The stress state affects the macroscopic failure. Failure occurs on the plane of maximum shear stress, which is normal to the applied load for plane strain and at 45° to the load for plane stress, figure 7.

The microscopic characteristics of plane stress and plane strain failures also differ. Fatigue striations are often found on the plane strain fracture surface and often growth rates can be correlated to the striation spacing ^{3,10}. Striations are rarely found on plane stress fracture surfaces. Ductile dimples are often present ^{8,11}, elongated in the direction of the tensile component. As the maximum cyclic stress intensity (K_{max}) approaches K_{Ic} , tensile fracture modes such as cleavage and ductile tearing become more prevalent ^{2,3}, and the fracture becomes similar to that of an overload failure, which occurs when K_{max} equals K_{Ic} .

2.1.1 Environmental Effects.

The effects of corrosive environments on fatigue crack growth rates are shown schematically in figure 8. The general trend is for the fatigue crack growth rate, for any value of ΔK , to be displaced to higher values and for the threshold stress intensity to be decreased. Perturbations

from the curve shown may occur depending on the material-environment combination. Corrosive environments range in severity from dry gas atmospheres to liquid metal environments. Laboratory air can cause growth rates ten times those occurring in a vacuum ¹², with more severe environments causing further crack acceleration, figure 9.

Both frequency and waveform exert large effects in corrosive environments. It has been shown that corrosion fatigue crack growth occurs only during the loading part of the fatigue cycle ^{13,14}, consequently slower frequencies cause faster growth rates. This point is discussed further in the next chapter.

The striation morphology changes with the severity of the environment ^{15,16}. Generally in a vacuum no striations are observed, in air and dry gases ductile striations occur and in more severe environments brittle striations form. However, both brittle and ductile striations may occur on the same fracture surface, depending on the individual grain orientation. The fracture morphologies (no striations, brittle and ductile striations) are shown in figure 10. Note that the brittle and ductile striations formed in the same air environment ¹⁶. Three factors which also influence striation morphology are; i) the strength of the interactions between material and environment, ii) the intrinsic

dislocation mobility, iii) the slip character, that is planar or wavy slip¹⁶.

2.2 General Nature of Stress Corrosion in Aluminum Alloys

Like fatigue, stress corrosion is well recognized as a failure mechanism in metals. Many aspects of stress corrosion have already been made apparent from the description of the Tracker wing-lock failures. It should be noted from that example, that in high strength aluminum plate, stress corrosion occurs by intergranular failure, most often when loading is in the short-transverse direction, which is normal to the elongated grain structure. Two general types of stress corrosion tests are common; time-to-failure and crack growth rate tests.

In time-to-failure (TTF) tests a smooth specimen, similar to a tensile specimen, is stressed in a corrosive environment and the time to failure recorded. Data from several samples can then be plotted as shown in figure 11¹⁷. It is clear that below a threshold stress (σ_{SCC}), failure will not occur. This threshold depends on the alloy, temper, loading direction with respect to the grain orientation, environment etc. Smooth specimen TTF data has been generated for most of the aluminum alloys in service, providing a wealth of data for comparison of new alloys or processes.

Failure is usually defined as either complete fracture of the specimen or the appearance of a crack of some nominal length. Often metallographic examination of the fracture is necessary to verify that stress corrosion was the failure mechanism, rather than pitting or some other process. This type of stress corrosion test involves a sequence of phenomena, i.e., initiation, propagation of cracks and final failure.

Smooth specimen stress corrosion tests do not give time-to-failure of a flawed structure, which is of practical importance for damage tolerant design. Additionally, if an improvement in the TTF is observed for a new alloy or treatment, it cannot be readily determined whether the improvement is due to slower crack growth rates or an increased crack initiation time. However, the major drawback of smooth specimen TTF data is that they are markedly affected by the specimen fracture toughness¹⁸. If a high toughness and a low toughness material have equal stress corrosion crack initiation times and crack growth rates, they will, nevertheless, have different times to failure. This is because the crack must grow to larger dimensions in the tougher material before final failure will occur. By using precracked stress corrosion specimens the drawbacks of the TTF test are avoided, although only information about crack growth is obtained.

There are numerous specimen geometries for precracked stress corrosion tests. Generally, each specimen configuration contains a machined starter notch of known geometry. The test procedure involves precracking and then growing a stress corrosion crack from the tip of the precrack and recording the crack length with time, allowing crack growth rates to be determined. The crack growth rate is then plotted against the crack tip stress intensity, which is usually calculated from the specimen compliance.

A schematic plot of the stress corrosion crack growth rate versus stress intensity is shown in figure 12. A threshold stress intensity ($K_{I_{SCC}}$) below which no stress corrosion occurs is indicated in figure 12. The presence of a threshold stress intensity for stress corrosion has not been conclusively proven, with the lowest crack growth rate observed primarily dependent on the patience of the investigator²⁰. During stage I cracking the crack growth rate is strongly dependent on the stress intensity. While in stage II the crack growth rate is independent of stress intensity. The transition from stress intensity dependent to stress intensity independent crack growth could be due to a change in the controlling mechanism of stress corrosion. Stage I and stage II crack growth are practically the most important because most of the cracking time is spent in these two regions. Stage III cracking corresponds to final

overload failure and occurs at stress intensities close to the fracture toughness value of the material. Due to the fast crack growth rates in stage III the time spent in this cracking mode is small, consequently in many stress corrosion crack growth rate tests this region is not observed.

2.3 Microstructural Effects on Fracture of 7075.

The chemical compositions of some typical 7000 series aluminum alloys are listed in Table I ²¹, with mechanical properties given in Table II ^{21,22}. The high strengths of these alloys are obtained by aging treatments causing the formation of certain types of second phase particles.

In general three types of second phase particles are common; constituents, dispersoids, and precipitates. These are distinguished from one another by their size and composition.

2.3.1 Constituent Particles.

These particles have a composition of Mg_2Si or Al_7Cu_2Fe , are typically $>30 \mu m$ in their largest dimension and are not affected by heat treatment ²³. At low stress intensities an advancing crack passes around constituent particles and the overall crack growth is unaffected, although local increases or decreases in growth rate may

occur. At higher stress intensities particles ahead of the crack tip may cleave, causing microcracks ahead of the crack which result in accelerated and irregular crack growth ^{5, 23}.

2.3.2 Dispersoid Particles.

Dispersoids have a composition of $Al_{12}Mg_2Cr$ or $Al_{20}Mn_3Cu_2$ ²³ and form large (0.02 to 0.5 μm) incoherent particles. Dispersoids form by solid state precipitation and cannot be completely redissolved, although their size, spacing and volume fraction can be altered somewhat by heat treatment. Dispersoids ahead of the crack tip initiate microvoids which coalesce to void sheets, linking cracks caused by constituent particles cleaving. It follows then, that larger dispersoid spacing would result in better fracture resistance.

Constituents and dispersoids can be effectively controlled by altering composition. In particular, by decreasing the iron and silicon contents, e.g. alloy 7475, the volume fraction of constituents is decreased compared to alloy 7075. This improves the cracking behaviour, especially at high intensities.

2.3.3 Precipitate Particles.

During aging heat treatments of 7000 series aluminum

alloys. Guinier-Preston (GP) zones form, which are rich in zinc and magnesium. With further aging the GP zones coarsen, that is the smaller zones dissolve and the solute atoms join the larger clusters. When the zones reach a critical size more stable precipitates, which are semicoherent, form. The precipitation sequence goes through an intermediate stage (η') before the final equilibrium precipitate MgZn_2 (η) or $\text{Mg}_3\text{Zn}_3\text{Al}_2$ (T) forms ²⁴.

For alloy 7075 there are two commonly used commercial precipitation aging heat treatments, Table III. The T6 temper starts with a solution treatment typically for 1.5 hours at 460°C ²⁵. This is followed by a 24 hour age at 121°C resulting in a material condition with, what is generally considered to be the highest hardness and tensile strength attainable with 7075. In actual fact the T6 condition corresponds to a slight underage and further aging causes a slight increase in strength before overaging occurs. Service experience with the T6 temper, such as the Tracker problem described in Chapter I, has shown that it has poor stress corrosion resistance, consequently the T73 temper was developed.

The T73 temper has a solution treatment similar to

the T6 temper. A two stage aging treatment, shown in Table III, is then applied, resulting in an overaged precipitate condition. The overaging causes a tensile strength loss of approximately 15% compared to the T6 condition. However, the stress corrosion resistance is greatly improved as indicated by the threshold values in Table II. Between the T6 and T73 tempers, then, there is a trade-off of tensile strength and stress corrosion resistance. The T6 temper providing high strength and the T73 temper providing good stress corrosion resistance.

Microstructural differences between the T6 and T73 tempers are a result of the different degrees of precipitation of the two tempers. In the T6 temper GP zones with an average diameter of 20 to 35 Å, plus small amounts of the intermediate precipitate (η') are present. In the T73 temper metastable MgZn_2 is predominant ²⁶. The grain boundary precipitate free zone (PFZ) of the T6 temper also changes with overaging to the T73 condition. The PFZ is mechanically weak and has been reported to influence both the fatigue and stress corrosion cracking properties ^{20, 27}, but its influence is not completely understood. During overaging to the T73 temper the PFZ disappears, since there is sufficient diffusion time to replenish the PFZ with solute to nucleate precipitates ²⁷. These changes cannot be detected with light microscopy, and are difficult to detect with electron microscopy. However, it has been thought that the precipitate

condition can be determined indirectly from electrical conductivity measurements. Minimum electrical conductivities for the T6 and T73 tempers are given in Table II. Electrical conductivity is normally measured as a percentage of an International Annealed Copper Standard (% IACS).

In order for material to be considered as being in the T73 condition the electrical conductivity must be at least 38% IACS. However, if the conductivity is between 38 and 40 % IACS, then the yield strength must not be more than 11% higher than the minimum T73 yield strength²⁸. If the electrical conductivity is above 40% IACS the requirement on maximum yield strength is relaxed. It should be noted that by putting a maximum limit on the yield strength, the standard implies a trade-off between stress corrosion resistance and yield strength.

T6 material can be heat treated to the T73 condition by applying the second T73 age (8 hours at 177°C)²⁹. However, the electrical conductivity after the application of the second T73 age must have increased by at least 6% IACS, irrespective of the final conductivity.

By aging 7075 past the T6 condition, that is, increasing the degree of precipitation and/or overaging, the stress

corrosion resistance improves, which is shown schematically in figure 13³⁰. Also, by continued aging, the resistance to fatigue³¹ and corrosion fatigue¹³ improves, figure 9. However, when 7075 is aged beyond the T6 condition the yield strength decreases. Hence, the improving stress corrosion and fatigue properties resulting from overaging could be due to the changing precipitate morphology or the decreasing yield strength. The improving fracture resistance could also be due to several other parameters such as solute distribution, dislocation density or particle coherency none of which will be discussed in detail.

By fatigue testing an underaged and overaged 7000 type aluminum alloy, with identical yield strengths, the effect of precipitate morphology can be isolated from yield strength effects. In such testing³¹, it was found that overaged material had slower fatigue crack growth rates than underaged material, although they had the same tensile strength. In contrast material aged to peak hardness (equivalent to the T6 temper) and underaged material had similar fatigue crack growth rates but different tensile strengths. These results could be rationalized by the dislocation movement and slip characteristics of the material^{31,32}.

In the underaged and peak hardness (i.e. T6) aged

condition small dislocation shearable Guinier-Preston zones were present, resulting in inhomogeneous coarse planar slip on a macroscopic scale. Softening in the slipbands, due to strain localization, leads to crack nucleation by slip band decohesion. The planar slip character caused by dislocation pile-ups at barriers and the consequent high local stresses result in cracking. In overaged material (i.e. T73), coarser partly coherent or incoherent precipitates were present. In this condition shearing of precipitates by dislocations takes place, but Orowan looping of precipitates predominates. In this condition cross-slip results in homogeneous deformation and wavy slip, which depend on the precipitate morphology rather than the yield stress, causing fatigue crack growth rates which are lower for the overaged condition ^{13, 33}.

The response of 7000 series alloys with lower copper contents, e.g. alloy 7079, indicates that the stress corrosion resistance is not dependent on tensile strength. In these alloys overaging treatments cause a drop in tensile strength, but the stress corrosion resistance does not improve significantly until the tensile strength has been greatly reduced ²⁰. Thus, for low copper content alloys overaging treatments are not effective as a method of increasing stress corrosion resistance, even though the tensile strength decreases. The lack of a dependence of stress corrosion resistance on yield strength is further emphasized in figure 13. In region 2 of

of figure 13 both the yield strength and stress corrosion resistance are increasing. Thus, a decrease in yield strength is not a requirement for improving the stress corrosion resistance of 7000 aluminum alloys.

It is evident from the foregoing discussion that the degree of precipitation affects the stress corrosion resistance and corrosion fatigue properties of 7000 series aluminum alloys in a similar fashion. That is, overaging generally increases the resistance to these types of fracture. As a result of this similarity, and others to be discussed, it has been postulated that corrosion fatigue is partly or wholly due to a stress corrosion cracking process occurring under cyclic loads. The interaction between stress corrosion and corrosion fatigue has not yet been fully determined, although several models have been developed, and these are described and compared in the next chapter.

3. CORROSION FATIGUE - STRESS CORROSION INTERACTIONS

3.1 Introduction

It has been pointed out that aggressive environments can accelerate the rate of fatigue crack growth in high strength alloys. It is also generally accepted that the acceleration of fatigue crack growth may occur either by a process involving the synergistic action of corrosion and the plastic deformation process occurring at the fatigue crack tip - true corrosion fatigue -, or by a process of stress corrosion in aggressive environments under static loading conditions - stress corrosion fatigue. However, the stress intensity ranges (ΔK) over which each of these processes occur is not clear.

There are many similarities between corrosion fatigue and static stress corrosion cracking³⁴⁻³⁸. Nevertheless, it is uncertain whether corrosion fatigue and cyclic stress corrosion cracking represent two different phenomena, or simply two manifestations of the same phenomena. For example, it has been suggested that corrosion fatigue is merely an extrapolation of cyclic stress corrosion cracking³⁸. This implies that the threshold stress intensity for stress corrosion cracking under cyclic loading may be considerably

lower than the threshold observed under static conditions. There is evidence for a variety of materials that the threshold for cyclic stress corrosion cracking is a variable parameter that depends on the alloy and the test environment, as it is for static stress corrosion cracking. But the threshold also appears to be dependent on loading parameters, such as frequency and stress ratio ³⁵⁻⁴⁰. However in general, very little is known of the factors affecting cyclic stress corrosion cracking.

Three models for the acceleration of fatigue crack growth in aggressive environments have been proposed:

- 1) Linear Summation Model ³⁴
- 2) Superposition Model ³⁵
- 3) Process Competition Model ³⁹.

Each of the models relies on stress corrosion cracking to some extent to explain corrosion fatigue. The purpose of this chapter is to summarize each model and review results that shed light on the interaction between fatigue and stress corrosion cracking.

3.2 Linear Summation Model

The Linear Summation Model was derived from work on high strength steels. It was suggested that the processes of corrosion fatigue and stress corrosion cracking may be the same ³⁴, since nearly all the effects observed for crack growth

under sustained loads (such as the effects of water vapour, hydrogen and oxygen) were reproduced under fatigue loading conditions¹¹. Hence, it was postulated that the corrosion

fatigue crack growth rate was made up of two components,

i) the fatigue crack growth in an inert environment plus,

ii) the crack growth rate resulting from a sustained load in an aggressive environment, i.e., stress corrosion cracking.

The sustained load profile is adjusted to include the influence of the fatigue load profile. The procedure to obtain the total crack growth rate is as shown in figure 14 and is represented by:

$$\left(\frac{\Delta a}{\Delta N}\right)_{\text{corrosion fatigue}} = \left(\frac{\Delta a}{\Delta N}\right)_{\text{inert envir.}} + \int \frac{da}{dt} \cdot K(t) dt \dots (2)$$

Where da/dt is the stress corrosion crack growth rate in the corrosive environment, $K(t)$ is the stress intensity profile and the integral is computed over one load cycle.

The linear summation model (equation 2) does not account for accelerated fatigue crack growth below the threshold stress intensity (K_{Isc}) for static stress corrosion cracking which has been observed for some material-environment systems. Below K_{Isc} faster fatigue crack growth could be due to some other interaction between corrosion and fatigue. Accelerated

fatigue crack growth at stress intensities less than K_{Isc} is considered by the superposition model.

3.3 Superposition Model

This model attributes faster fatigue crack growth to a stress corrosion cracking mechanism; including corrosion fatigue at stress intensities less than K_{Isc} . It is suggested that the threshold for stress corrosion cracking under cyclic loads ($K_{Isc(f)}$) is less than K_{Isc} ³⁵.

The linear summation model is similar to the superposition model and considers the corrosion fatigue crack growth rate as the fatigue crack growth rate in an inert environment plus the stress corrosion crack growth rate. This latter is assumed to be discontinuous and be made up of a discrete number of "jumps" per cycle. The total crack growth rate is then given by:

$$\frac{da}{dN} = m(\Delta K)^n + (\text{no. of scc jumps/cycle}) \cdot \text{(average jump size)} \quad \dots (3)$$

Where the first term is the mechanical fatigue component, represented by the Paris equation and the second term is the discontinuous stress corrosion cracking component. Evidence supporting discontinuous stress corrosion cracking for 7000 type aluminum alloys has been presented⁴². To

determine the average jump size and the number of jumps per cycle, two cases are considered: 1) when stress corrosion cracking can occur throughout the whole fatigue cycle, i.e. $K_{\min} \geq K_{\text{lsc}(f)}$, (figure 15a), and 2) when stress corrosion cracking occurs during a portion of the fatigue cycle or $K_{\min} < K_{\text{lsc}(f)}$, (figure 15b). For case 1, the average jump size (l_{av}) was calculated³⁵ using the mean value theorem to be:

$$l_{\text{av}} = \frac{1}{(K_{\text{max}} - K_{\text{min}})} \int_{K_{\text{min}}}^{K_{\text{max}}} F(K) dK \quad \dots (4)$$

Where $F(K)$ represents the loading parameters. Since stress corrosion cracking can occur throughout the cycle then:

$$\left(\frac{da}{dN}\right)_{\text{scc}} = \frac{1}{F(K_{\text{max}} - K_{\text{min}})} \int_{K_{\text{min}}}^{K_{\text{max}}} F'(K) dK \quad \dots (5)$$

With f the cyclic frequency and $F'(K)$ the same as $F(K)$ but including a thermally activated component which controls the stress corrosion cracking incubation time. The diffusion of

hydrogen was considered the most likely thermally activated process³⁵. A hydrogen diffusion process gives a stress corrosion crack growth rate of:

$$\frac{da}{dN} = \left(\frac{K}{K_{1scc}} - 1 \right)^2 \cdot \frac{64 D_0 e^{-Q/RT}}{w} \quad \dots (6)$$

Where D_0 is the diffusion constant; w , the width of the grain exposed to the advancing crack front; Q , the activation energy, R , Boltzmann's Constant and T , the temperature (K). Equation (6) can be generalized for fatigue type loading by replacing K_{1scc} by $K_{1scc}(f)$. Equation (6) is then substituted for $F'(K)$ in equation (5) to give:

$$\left(\frac{da}{dN} \right)_{scc} = \frac{64 D_0 e^{-Q/RT}}{fw K} \int_{\Delta K \left[\frac{1}{\beta-1} \right]}^{\Delta K \left[\frac{\beta}{\beta-1} \right]} \left(\frac{K - K_{1scc}(f)}{K_{1scc}(f)} \right)^2 dK \dots (7)$$

Where $\beta = K_{max}/K_{min}$. This integrates to :

$$\begin{aligned} \frac{da}{dN}_{scc} = & \frac{64 D_0 e^{-Q/RT}}{3 fw K_{1scc}(f)} \left[\Delta K^2 \left\{ \frac{\beta^2 + \beta + 1}{\beta - 1} \right\} - 3 \Delta K K_{1scc}(f) \right. \\ & \left. \left\{ \frac{\beta + 1}{\beta - 1} \right\} + 3 K_{1scc}^2(f) \right] \quad \dots (8) \end{aligned}$$

which gives the average cyclic stress corrosion cracking growth rate.

When $K_{\min} < K_{\text{IscC}}(f)$ (see figure 15) some changes to the analysis are necessary. Firstly, the lower integration limit must be changed to account for the part of the load below $K_{\text{IscC}}(f)$, and secondly, the fraction of the fatigue cycle during which stress corrosion cracking can occur must be accounted for. The latter is incorporated by estimating a sine wave load by a triangle wave. Incorporating these changes into equation (5) yields:

$$\left(\frac{da}{dN}\right)_{\text{scc}} = \frac{1}{f} \left[\frac{K_{\max} - K_{\text{IscC}}(f)}{K_{\max} - K_{\min}} \right] \left[\frac{1}{K_{\max} - K_{\text{IscC}}(f)} \right] \int_{K_{\text{IscC}}(f)}^{K_{\max}} F'(K) dK \quad \dots (9)$$

As before substituting equation (7) into (9) and integrating gives the cyclic stress corrosion crack growth rate:

$$\left(\frac{da}{dN}\right)_{\text{scc}} = \frac{64 D_0 e^{-Q/RT}}{3 f w K_{\text{IscC}}^2(f)} \left[\frac{\left\{ \Delta K \left(\frac{\beta}{\beta-1} \right)^{-K_{\text{IscC}}(f)} \right\}^3}{\Delta K} \right] \quad \dots (10)$$

The crack growth rate from either equation (8) or (10)

can be summed with the inert environment fatigue crack growth rate to obtain the corrosion fatigue cracking rate.

The superposition model accounts for accelerated crack growth in a corrosive environment over a wider range of ΔK than the linear summation model by the introduction of $K_{lsc}(f)$. Since the superposition model accounts for accelerated crack growth at any stress intensity level by a stress corrosion cracking process, it ignores other interactions between corrosion and fatigue which might accelerate the rate of fatigue crack growth.

3.4 Process Competition Model

Austen and Walker³⁹ proposed that two separate corrosion fatigue processes exist. The first referred to as true corrosion fatigue explains accelerated crack growth at stress intensities below K_{lsc} and accounts for any synergistic action between the environment and fatigue. The second, stress corrosion fatigue, applies above K_{lsc} , and may be a combination of corrosion fatigue and stress corrosion cracking. Which of these two processes predominates depends on the frequency and stress ratio. High frequencies and low stress ratios favour the occurrence of true corrosion

fatigue. The frequency dependence exists because true corrosion fatigue is believed to be cycle dependent rather than time dependent, so that at high frequencies true corrosion fatigue crack growth per unit time should increase and tend to become the dominant mode of crack growth. In contrast, stress corrosion fatigue is believed to be time dependent and higher frequencies should not increase the rate of crack growth. The stress ratio effect occurs since the ΔK value corresponding to K_{Isc} (ΔK_{sc}) varies with stress ratio in a (1-R) relationship.

True corrosion fatigue and stress corrosion fatigue may be in competition, with the process resulting in the fastest growth rate predominating. A frequency or stress ratio representing the borderline between true corrosion fatigue and stress corrosion fatigue can be calculated using the Paris Equation:

$$\frac{da}{dN} = m(\Delta K)^n \quad \dots (1)$$

The rate of crack growth per cycle occurring due to stress corrosion cracking can be approximated using the plateau growth rate by allowing for the cyclic frequency:

$$\frac{da}{dN} = \left(\frac{da}{dt} \right)_{sc} \cdot \frac{1}{f} \quad \dots (11)$$

Also, the transition from true corrosion fatigue to stress corrosion fatigue is assumed to occur at:

$$\Delta K_{scc} = K_{lscc} (1-R) \quad \dots (12)$$

which means the transition occurs when the cyclic K_{max} equals K_{lscc} . Thus combining the above three equations gives:

$$\frac{da}{dt} \cdot \frac{1}{F} = m \left[K_{lscc} (1-R) \right]^n \quad \dots (13)$$

In equation (13) if either the frequency or stress ratio is fixed, then a value of the other can be calculated which represents the borderline between true corrosion fatigue and stress corrosion fatigue.

The effects of cyclic waveform are more accurately accounted for in the extended process competition model⁴³. In this model stress corrosion fatigue crack growth rate is based on the cyclic average stress intensity, rather than the maximum stress intensity. The average stress intensity will depend on the waveform applied, hence better prediction of waveform effects is obtained.

3.5 Comparison of Models

The three models presented above result in different shapes to da/dN vs ΔK curves, figure 16. Each model agrees with various experimental data to different extents and the relevance of any model depends on the material-environment system.

There are two ways that the three models interpret environmentally enhanced fatigue crack growth. Both the linear summation model and the superposition model consider it to be a result of stress corrosion cracking under a cyclic load. This does not take into account other interactions between corrosion and fatigue. The process competition model takes into account interactions between corrosion and fatigue, as well as stress corrosion cracking and fatigue. This model is the only one in which a separate process for corrosion fatigue, in addition to stress corrosion cracking is deemed to exist.

In both the superposition model and process competition model the stress corrosion cracking threshold under cyclic loads may be different from the threshold for static loads (K_{ISCC}) and is variable. In contrast, in the linear summation model the threshold for cyclic and monotonic loads is the

same and constant. The extent to which corrosion fatigue is attributable to a stress corrosion cracking process may partly be determined by knowing the value of the cyclic stress corrosion threshold. This at least may account for accelerated fatigue crack growth.

3.5.1. Crack Propagation by a Dual Process

There are experimental results which indicate that a process other than stress corrosion cracking occurs during corrosion fatigue ^{13,37,39,44-46}. For example: alloy Ti-6Al-4V is immune to stress corrosion cracking in distilled water but shows considerable acceleration of fatigue crack growth in the same environment ⁴⁴, indicating there may be a synergistic interaction between corrosion and fatigue, other than stress corrosion cracking. Corrosive environments can lead to acceleration of fatigue crack growth at stress intensities below the static stress corrosion cracking threshold ^{13,14,36,39,44,46,47}. Evidence that this is not due to stress corrosion cracking has been obtained for 7000 type aluminum alloys by studying the effect of waveform below the static stress corrosion cracking threshold ^{13,47}. For square waves no effect of frequency on corrosion fatigue crack growth rates below K_{Isc} was observed over two orders of magnitude (0.1 Hz to 10 Hz). Hence crack growth rates

are independent of hold times at maximum load. If stress corrosion cracking were contributing to crack growth in this regime, it would occur during the hold time at maximum load and would increase with increasing hold time. The failure of the linear summation model to account for a synergistic interaction between corrosion and fatigue below K_{Isc} is well documented^{37,38,40,44}. The superposition model does consider accelerated fatigue crack growth rates below the static stress corrosion cracking threshold, but it models them with a stress corrosion process, when in fact, the physical process occurring may be something different.

The process competition model separates corrosion fatigue into true corrosion fatigue and stress corrosion fatigue³⁴. The data for 835M30 steel, figure 17, shows the two regions clearly. The transition from true corrosion fatigue to stress corrosion fatigue occurs at a stress intensity of $6 \text{ MPa}\sqrt{\text{m}}$ ($5.5 \text{ ksi}\sqrt{\text{in}}$), for $R=0.5$. The K_{Isc} for 835M30 steel is $14.3 \text{ MPa}\sqrt{\text{m}}$ ($13 \text{ ksi}\sqrt{\text{in}}$), so the observed transition agrees moderately well with the transition of $7.2 \text{ MPa}\sqrt{\text{m}}$ ($6.5 \text{ ksi}\sqrt{\text{in}}$) predicted by equation (12).

Fatigue tests on Ti-8Al-1Mo-1V (figure 18) result in crack growth curves very similar to those of the steel alloy of figure 17⁴⁶. As before the transition from true corrosion

fatigue to stress corrosion fatigue is clearly marked by the sudden increase in crack growth rate for the slower frequencies. The 3.8 mm(0.15") and 6.86 mm(0.27") thick mill annealed sheet tested, exhibited a transition stress intensity, ΔK_{SCC} , of 18.7 MPa $\sqrt{m}$ (17 ksi $\sqrt{in}$). This value agrees moderately well with static stress corrosion threshold measurements, K_{ISCC} , of 22 MPa $\sqrt{m}$ (20 ksi $\sqrt{in}$) and 23.1 MPa $\sqrt{m}$ (21 ksi $\sqrt{in}$) for mill annealed sheet of 4.1 mm(0.16") and 12.7 mm(0.5") thickness respectively ⁴⁸. Agreement between ΔK_{SCC} and K_{ISCC} would be expected since the test stress ratio was zero (see equation 12).

Curves similar to those of figure 17 and 18 have been observed in an aluminum alloy (Al-Cu-Mg) for square wave loading, figure 19 ⁴⁷. The similar crack growth rates below K_{ISCC} for the two frequencies have already been discussed. However, the faster crack growth rate for the slower frequency (longer hold times at maximum load) above K_{ISCC} suggest that a stress corrosion cracking component is present. In contrast, the data of figure 19 for triangular waveforms indicate a pronounced frequency effect in the threshold region, with crack growth rates at 0.1 Hz being 5 to 6 times faster than at 10 Hz ⁴⁷. The reasons for these differences are not known. This behaviour is contrary to that predicted by the process competition model.

Fractography shows that different fracture surface

morphologies may prevail above and below K_{1SCC} ⁴⁶. At stress intensities above K_{1SCC} in a titanium alloy, cleavage occurs on the same crystallographic plane as static stress corrosion cracking. In this ΔK range the fracture morphology shown in figure 20 dominated, suggesting that most of the crack propagation was due to stress corrosion cracking, and indicating that a stress corrosion fatigue process occurred. At higher crack rates or high frequencies, the fracture surface was a mixture of cleavage and striations. This could be due to the increasing relative importance of mechanical intensities and frequencies¹³.

At stress intensities less than K_{1SCC} cleavage fracture occurred on two planes, one of which is not common to stress corrosion cracking⁴⁶, and fatigue striations were present, figure 21. The differences between fracture surfaces of figures 20 and 21 would seem to indicate that different cracking processes occur above and below K_{1SCC} .

It appears that corrosion fatigue could be made up of two processes - true corrosion fatigue and stress corrosion fatigue. The transition from true corrosion fatigue and stress corrosion fatigue occurs at a cyclic stress corrosion cracking threshold. The transition could occur when $(da/dN)_{TCF} = (da/dN)_{SCF}$ ⁴³. If this is the case, then the process which predominates is the one giving the fastest crack growth

rate ^{37,39,46}. The transition could also occur when $\Delta K = K_{Isc} (1-R)$ ³⁹ which is when the cyclic K_{max} equals K_{Isc} .

3.5.2 Crack Propagation by a Single Process

It has been postulated that stress corrosion cracking is the cause of accelerated corrosion fatigue at all stress intensity levels. This type of interaction is implicit in the linear summation and superposition models ^{34,35}. This requires that stress corrosion cracking and corrosion fatigue occur by similar mechanisms, and some results lend support to this.

Similar crack morphologies for stress corrosion cracking and corrosion fatigue have been reported for 18Ni maraging steel ³⁴ and mild steel ³⁷. Also, for these alloys, nearly all the effects observed for crack growth under sustained loads were reproduced for fatigue ⁴¹; e.g. similar crack initiation times and crack velocities ³⁷.

A fatigue test in which the crack tip strain rate was held constant over the entire stress intensity range (by continually altering frequency) was applied to an aluminum alloy of the Al-Cu-Mg type ⁴⁷. When the results were plotted,

da/dN vs ΔK , the shape of the curves was similar to stress corrosion crack growth curves (da/dt vs K). This strongly suggests that corrosion fatigue crack growth is an extension of stress corrosion cracking.

The effects of cyclic waveform on corrosion fatigue crack growth rates in a maraging steel below K_{Isc} have been studied¹⁴, figure 22. From figure 22 two points should be noted. Firstly; the square and negative sawtooth waveforms caused no acceleration of crack growth rates above those observed in air. Secondly; the fatigue crack growth rate is increased by the same amount for triangular and positive sawtooth waves, which had the same rise times. The former point indicates that no corrosive action occurs during constant or decreasing tensile stresses, while the latter shows that similar loading rise times cause equivalent amounts of fatigue crack acceleration. Hence, corrosive processes act only during the rise time of the cycle¹⁴. The influence of rise time on corrosion fatigue crack growth rates in A533B-1 steel below K_{Isc} is shown in figure 23³⁶. The cyclic crack growth rate increases to a maximum for a rise time of approximately 30 seconds and then decreases, indicating a strong dependence on loading rise time or loading (strain) rate. The critical rise time effect could correspond to the strain rate effect observed in stress corrosion cracking⁴⁹,

as shown in figure 24 ³⁶. The results indicate that at slow strain rates passive film formation occurs quickly enough to suppress corrosion processes involved in stress corrosion cracking and at high strain rates pure mechanical failure is observed. Hence, the condition for corrosion fatigue crack growth is that rupture processes at the crack tip must provide a supply of bare metal faster than it is removed by passivation. In addition to the steel alloy ³⁶, a frequency dependent stress corrosion cracking-repassivation competition process has been proposed for other alloys ^{38,40}.

None of the proposed models for corrosion ~~fatigue~~ satisfactorily explain, quantitatively, the frequency effects below K_{Isc} described above.

The K_{Isc} value may be lowered under cyclic loads because of continued rupture of a passive film during the rise time. This would make the cyclic K_{Isc} dependent on load ratio and frequency. A lower cyclic K_{Isc} may also be due to the cyclic crack tip plastic zone having a higher defect density than the monotonic plastic zone ³⁵. This would make material more crack sensitive due to its work hardened condition. Also, the cyclic plastic zone could provide more dissolution sites because of emerging dislocations and other defects.

Crack growth above the cyclic K_{Isc} is similar for both the static and cyclic conditions, that is stress corrosion cracking is the damaging mechanism. Reasonable agreement should then be possible between observed crack growth rates and those predicted by the linear summation model and superposition model in this stress intensity range ^{36,38}. Differences between actual and modelled results could be due to electrochemical differences at the crack tip between static and cyclic situations ^{36,38,44}.

A repassivation-stress corrosion cracking competition may exist. Below the cyclic K_{Isc} passivation occurs and virtually no accelerated crack growth takes place. Above the cyclic K_{Isc} a strain rate dependent stress corrosion cracking mechanism occurs and this accounts for faster crack growth rates. The value of the cyclic K_{Isc} depends on the load ratio and frequency. This process therefore gives results which agree qualitatively with those predicted by linear summation and superposition models.

3.6 Summary

Three models for accelerated fatigue crack growth rates in corrosive environments have been reviewed and compared. Two models, the linear summation model and superposition

model, explain faster fatigue crack growth, by considering it as a combination of two components; fatigue crack growth in an inert environment and a stress corrosion cracking component. The process competition model postulates that corrosion fatigue and stress corrosion cracking are in competition and that crack growth occurs by the faster process.

The value of the cyclic stress corrosion cracking threshold is dependent on load parameters in both the superposition and process competition models. In contrast, the transition to cyclic stress corrosion cracking predicted by the linear summation model is independent of loading and is equivalent to K_{1SCC} .

The linear summation model does not attempt to explain accelerated crack growth at stress intensities below K_{1SCC} . The superposition model considers faster crack growth below K_{1SCC} to be a result of a stress corrosion process. Further results indicate that a competition between repassivation and cyclic stress corrosion cracking occurs, where repassivation occurs below the cyclic K_{1SCC} and stress corrosion cracking occurs above it. These results lend qualitative support to the superposition model. The process competition model predicts a true corrosion fatigue -

stress corrosion fatigue competition. True corrosion fatigue takes place below the cyclic stress corrosion crack threshold, while stress corrosion fatigue may occur above it, with the process giving the fastest crack growth predominating.

4. A MECHANISM FOR STRESS CORROSION CRACKING

In the previous chapter it was shown that corrosion fatigue is caused, at least partly, by stress corrosion cracking. Before a corrosion fatigue - stress corrosion interaction can be explicitly defined, the mechanism of stress corrosion cracking must be determined. To date, several stress corrosion mechanisms have been proposed and these have been reviewed extensively by Parkins⁴⁹ and Speidel²⁰. However there is no universally accepted theory or mechanism for stress corrosion cracking.

Hydrogen embrittlement has often been suggested as a mechanism for stress corrosion cracking as well as corrosion fatigue. There are several reasons why a hydrogen embrittlement mechanism should be considered^{50,51}. Possibly the strongest reason is because the stage II stress corrosion crack growth rate of 7075 in air is proportional to humidity. Another factor indicating a hydrogen embrittlement mechanism, is the similar intergranular and discontinuous nature of stress corrosion and hydrogen embrittlement cracking.

The slip character, that is planar or wavy slip, has been correlated to the susceptibility to hydrogen embrittlement³⁰. Decreasing slip planarity tends to reduce hydrogen

transport to grain boundaries. As was pointed out in chapter 2, the T6 condition of 7075 exhibits a planar slip character and has low stress corrosion resistance, while the T73 condition exhibits wavy slip and is essentially immune to stress corrosion. Wavy slip, then, appears to be beneficial for both hydrogen embrittlement and stress corrosion resistance. As was discussed, it is predominantly the grain interior precipitate structure that determines the type of slip occurring. Hence, the hydrogen embrittlement reaction is in part dependent on the grain interior precipitate morphology.

It would be expected that the grain boundary precipitate morphology would influence intergranular cracking and as a result stress corrosion. The grain boundary precipitate could play a role in the accumulation of hydrogen at grain boundaries or, it could affect intergranular crack nucleation and propagation.

Dislocations associated with large MgZn_2 grain boundary precipitates have been observed in the stress corrosion susceptible T6 condition. In contrast, the stress corrosion resistant T73 condition has been reported to contain fewer dislocations⁸². Pitting corrosion occurring at the large MgZn_2 precipitates, which are anodic to the surrounding matrix, were deemed responsible for stress corrosion crack

initiation. The pitting corrosion rates for T6 and T73 material were the same, although no stress corrosion cracking occurred in the T73 material. Consequently, an interaction between dislocations and the large grain boundary MgZn_2 precipitates, at which the pits form, was thought to cause an additional stress concentration factor, sufficient to allow crack growth under existing stress levels. Crack propagation could be temporarily halted if another large grain boundary precipitate were to be encountered. This would blunt the crack tip until further pitting occurred and the dislocation - precipitate interaction once again caused the load, stress, to be raised sufficiently to reinitiate growth⁵³. In this mechanism, the presence of dislocations at large grain boundary MgZn_2 precipitates determines whether stress corrosion occurs. It is important to note that discontinuous crack growth is predicted, with the large grain boundary MgZn_2 precipitates having a major effect on the cracking behaviour. A discontinuous mode of stress corrosion crack growth is supported by fractographic evidence^{42, 53}, and stress wave emission analysis³⁵.

High strength aluminum alloys do not suffer any degradation of mechanical properties when exposed to dry hydrogen gas⁵⁴. Thus, molecular hydrogen does not embrittle aluminum alloys. However, this does not discount the possibility of

atomic hydrogen - formed by hydrolysis in wet gases or aqueous solutions - from embrittling aluminum alloys, causing the observed degraded mechanical properties which result after exposure to wet gases or aqueous solutions. The evolution of hydrogen during stressing of Al-Zn-Mg alloys exposed to water vapour supports the atomic hydrogen embrittlement mechanism ⁵⁵.

The embrittling atomic hydrogen may form bubbles of molecular hydrogen or remain in solid solution primarily along grain boundaries ^{56,57}. The hydrogen pressure in the bubbles may be sufficient to cause intergranular failure, although it is thought that hydrogen in interstitial solid solution is more damaging, because of a lowering of the cohesive strength of the atomic bonds ⁵⁷.

The formation of intergranular hydrogen bubbles is affected by the grain boundary precipitate size ⁵⁸. An Al-6Zn-3Mg alloy aged for 24 hours at various temperatures was exposed to water vapour saturated air (w.v.s.a.) and the resulting properties compared to similarly aged material not exposed to w.v.s.a., figure 25. The properties for unexposed material follow the normal trend for age hardened systems, with the material aged for 24 hours at 120°C, (nominally the T6 condition) having the highest yield strength.

It is clear from figure 25 that material aged at the higher temperatures and exposed to w.v.s.a. had a smaller fractional loss in tensile ductility.

Microstructural examination revealed that at grain boundary precipitates larger than 200 Å, molecular hydrogen bubbles had formed. In contrast at grain boundary precipitates smaller than 200 Å, no hydrogen bubbles were observed, therefore the hydrogen must have been held in interstitial solid solution. At higher aging temperatures larger grain boundary precipitates form; hence a greater number of the particles are larger than the critical 200 Å size. The smaller fractional loss in tensile ductility of material aged at higher temperatures and exposed to w.v.s.a. indicates that the presence of hydrogen in the form of bubbles is not as detrimental as hydrogen in solid solution. When the ternary alloy tested was given a T6 aging heat treatment (24 hours at 120°C.) an average grain boundary precipitate size of 200 Å resulted and occasional bubbles were observed. Therefore, material aged beyond the T6 condition should have an average grain boundary precipitate size greater than 200 Å and the more innocuous hydrogen bubbles would form when exposed to water vapour.

The superior stress corrosion resistance of T73 material

compared to the T6 temper may be explained by the hydrogen bubble phenomena outlined. As a stress corrosion crack approaches a large (greater than 200 Å) grain boundary precipitate, crack propagation will be slow, since the hydrogen diffusing into the material should form an innocuous bubble adjacent to the precipitate and the concentration of hydrogen in solution should be kept low, figure 26. Eventually the crack grows to the precipitate particle and collapses the bubble of molecular hydrogen adjacent to it, releasing the molecular hydrogen to the atmosphere. In the T73 condition, the large precipitate particles are close together and "fresh" atomic hydrogen, formed through continuing hydrolysis at the crack tip, will enter the metal and diffuse to the next large precipitate and form another innocuous bubble. Hydrogen may not diffuse into the crack "walls" because of the formation of a protective passive film, leaving only the freshly exposed crack tip unprotected. In the T6 condition before hydrogen can diffuse to the next large precipitate (which would be far removed) hydrogen in solution ahead of the crack tip lowers the strength of the boundary sufficiently to cause fast crack growth to a point where the concentration of hydrogen in solution is lower and the cohesive strength of the boundary higher. Eventually, the crack will approach another large precipitate and crack growth will again be slowed as the hydrogen forms a bubble around the precipitate. By this process there is discontinuous crack growth by a hydrogen embrittlement mechanism. Temporary crack arrest may occur

each time a hydrogen bubble is reached because of crack tip blunting. This would be a larger factor in the T73 condition, slowing crack propagation in this condition even further.

It is clear from the preceeding discussion that for 7075 the stress corrosion resistance is greatly affected by the grain boundary precipitate structure. By studying material which has been heat treated to the standard T6 and T73 tempers the precipitate morphology leading to good stress corrosion resistance cannot be definitively determined, because of concurrent changes in tensile strength caused by changes in precipitate structure. A novel heat treatment, called retrogression and reaging, which has been developed by Cina at the Israel Aircraft Industries Ltd. offers another approach to the heat treatment of 7075⁵⁹. Retrogression and reaging (RRA) treatments, applied to 7075, are claimed to achieve strength levels equivalent to the T6 temper and stress corrosion resistance equivalent to the T73 temper. Thus, the strength - stress corrosion trade-off is eliminated. More importantly, by comparing T6 and RRA material the microstructural factors required for good stress corrosion resistance can be isolated, since T6 and RRA should have equivalent tensile strengths, and differences in precipitate structure between T6 and RRA material could be correlated to stress corrosion resistance without the tensile strength influence.

5. RETROGRESSION AND REAGING

Retrogression and reaging is a two stage heat treatment applied to 7075-T6. The first stage, retrogression, involves a short-time partial solution treatment in the temperature range 200 to 280°C. During retrogression the hardness (or tensile strength) changes radically, figure 27. Initially the hardness decreases rapidly with increasing retrogression time, until a minimum hardness is reached. After the minimum, some process of secondary hardening occurs, until finally at long retrogression times the hardness falls continually as the material becomes overaged. The second stage, reaging, is a long age at 120°C, and essentially, at short retrogression times, reaging restores the hardness to T6 levels, figure 27. According to Cina^{59,60}, the optimum combination of tensile and stress corrosion resistance occurs when the retrogression time corresponds to the minimum hardness on the retrogression curve, figure 27, and reaging is applied. Apparently this RRA treatment will result in T6 strength and T73 stress corrosion resistance.

Figure 28 shows Cina's hardness results for various retrogression times and temperatures. For each retrogression temperature the hardness curves have the general shape of the schematic curve of figure 27. Figure 28 illustrates

several points of interest. Firstly, the initial hardness minimum in the retrogression curves decreases with increasing retrogression temperature. Also, the retrogression time to the hardness minimum is shorter for higher retrogression temperatures. These two trends indicate that the microstructural processes taking place during retrogression occur faster and to a greater extent at higher retrogression temperatures.

An important aspect of Cina's results is the short retrogression times obtained. In figure 28 the longest retrogression time illustrated is 120 seconds, while the longest retrogression time corresponding to the first minimum in the hardness curve (which apparently gives the best combination of tensile and stress corrosion properties), is approximately 30 seconds. Since Cina tested the RRA treatments on thin sheet (less than 1 mm thick) the complete sheet cross section would receive the full heat treatment. However, if these short retrogression times were applied to thick section components (e.g. plate 25 mm to 80 mm thick, or more), the center section of the component would receive essentially no treatment, because of the time required for the conduction of heat from the component surfaces to its center section. Alloy 7075 is often used for thick section components, and consequently for RRA to be applied to thick

sections longer retrogression times are required.

Some of the stress corrosion results obtained by Cina are given in Table IV ⁶⁰. Time - to - failure stress corrosion tests were performed and it is clear from Table IV that the RRA material has superior stress corrosion times - to - failures compared to T6 material. Also, increasing the retrogression temperature increased the stress corrosion life, however, the retrogression times become extremely short (i.e. 15 seconds at 240°C). Although the reaging times shown in Table IV vary, this effect was determined to be minor ⁵⁹. Improved time - to - failure stress corrosion lives, as discussed previously, could indicate either an increased resistance to pitting corrosion, and hence stress corrosion crack initiation, an improved stress corrosion crack growth rate or an improved fracture toughness.

As was indicated, by studying RRA treatments, there is potential to determine the microstructural factors required for good stress corrosion resistance. Also, by varying the retrogression heat treatment parameters carefully, materials with a range of stress corrosion resistance, but the same tensile strengths, may be obtained. By collecting quantitative stress corrosion and corrosion fatigue crack growth rate data for the variously retrogressed and reaged material

new insight into the interaction between corrosion fatigue and stress corrosion may be possible.

In addition to the potential of RRA outlined above, the practical benefits of the process are also important. That is, the achievement of T6 tensile strength and T73 stress corrosion resistance in the same material, which would be of great value in high strength stress corrosion resistant structures. It is on this aspect of the RRA process that the remainder of this thesis will concentrate.

6. EXPERIMENTAL OUTLINE AND TECHNIQUE

6.1 Outline of Study

To verify the claims made about the RRA process and to determine the effects of RRA in more detail, the study described in this thesis had four goals.

- A. To determine the physical properties of retrogressed as well as retrogressed and reaged material using yield strength and electrical conductivity data. The yield strength data will determine whether in fact 7075-T6 strength levels can be achieved with material which has been retrogressed and reaged. Electrical conductivity measurements can be used to give an indication of over-aging in 7075. As explained previously, the stress corrosion resistance of 7075 increases as it is aged beyond the T6 condition, and since RRA is a further aging process applied to T6 material, it should be possible to use electrical conductivity to indicate the stress corrosion resistance of particular RRA treatments.
- B. Secondly, it was aimed to explore the use of lower retrogression treatments. In this work four retrogression temperatures have been investigated; 220, 200, 180, and

160°C, retrogression was followed by water quenching. The higher two temperatures fall in the range initially investigated by Cina and thus will serve as a comparison to the data already discussed. At the lower retrogression temperatures (160 and 180°C) the kinetics of the reaction occurring during retrogression may be slowed down. This would result in longer times to the minimum hardness on the retrogression curve, figure 27, thus increasing the process' practicality. Also the 160 and 180°C retrogression temperatures fall above and below the standard T73 overaging temperature of 177°C. Consequently, by studying long retrogression times at these temperatures, the influence of temperature variations and reaging on the standard T73 temper will be discerned. The reaging (RA) treatment applied was 29 hours at 121°C.

- C. Determine the stress corrosion resistance resulting from RRA treatments using fracture mechanics data, i.e. stress corrosion crack growth rate (da/dt) versus crack tip stress intensity (K). Figure 29 shows a typical stress corrosion crack growth rate curve for 7075-T6, generated during this study, in which the stage I and II regions of crack growth, discussed in chapter 2 are clear. As 7075 is overaged beyond the T6 condition and the stress corrosion resistance improves, the plateau growth rate

is decreased and the stage I crack growth may shift to higher stress intensities, as shown in figure 29 by the arrows.

- D. Using electron microscopy determine the microstructural changes occurring during retrogression and correlate the changes with mechanical properties. With regard to the model for stress corrosion presented in the previous chapter, the grain boundary precipitate structure is of particular interest.

6.2 Experimental Technique and Materials

6.2.1 Tensile Tests.

The geometry and specifications of the tensile specimen used are shown in figure 30. Tensile tests were carried out in accordance with ASTM standard E8 ⁶¹, using a computer controlled servo-hydraulic test system. The tests were performed at a constant loading rate (690 MPa/min) until the yield point was reached, and then at a constant crosshead speed (0.5 cm/min) to fracture. Tensile specimens were machined from 1.9 cm (0.75 in) diameter extruded bar or 2.5 cm (1 in.) thick 7075-T651 plate. Heat treatments were always applied prior to machining. Ductility measurements were accomplished

by scribing lines on each specimen 50.8 mm (2 in) apart prior to testing and then measuring the scribe line spacing after testing.

6.2.2 Electrical Conductivity Measurements.

The electrical conductivity of all the "as received" material and each individual tensile and stress corrosion specimen was measured using a Magnaflux FM-100 conductivity meter. This instrument uses an eddy current probe to measure electrical conductivity as a percentage of the International Annealed Copper Standard (% IACS).

6.2.3 Stress Corrosion Tests.

Stress corrosion tests were performed using double cantilever beam specimens, figure 31, which have been used extensively by the Boeing Company^{18,20}. Double cantilever beam specimens were 2.5 cm X 2.5 cm X 12.5 cm (1" X 1" X 5") and machined from 7075-T651, 2.5 cm thick plate, which had a microstructure typical of rolled material, figure 32. The T651 temper is a stress relieved (by stretching 1.5 to 3.0%) version of T6. The length of the specimen corresponds to the rolling direction (longitudinal) of the plate. The RRA treatments were applied to specimen blanks immersed in silicone

oil to prevent oxidation, and then the starter notch and bolt hole introduced. Before the retrogression treatment was applied, the specimen blanks were preheated to allow the center section of the blanks to reach the retrogression temperature. The preheating time was determined as shown in figure 33. The time for the thermometer bulb to reach the retrogression temperature was used as the preheating time. These times were 1.75 minutes and 3 minutes for retrogression at 180°C and 220°C respectively.

The specimens are loaded through the bolt in the short-longitudinal direction until a mechanical "pop-in" crack forms at the root of the machined starter notch. The initial load line displacement to cause "pop-in" is left constant for the duration of the test. After the steel loading bolt has been coated with a protective lacquer to prevent galvanic corrosion with the aluminum specimen, a standard 3.5% NaCl seawater solution ⁶² is dropped into the crack tip three times daily. This procedure apparently gives similar results compared to total immersion in seawater ¹⁸, but does not cause general corrosion attack on all surfaces which would hinder crack measurement.

* The load line displacement induced by the bolt load is kept constant for the duration of the test. Consequently as

the crack grows by stress corrosion, the crack tip stress intensity decreases according to the compliance calibration equation ⁶³:

$$K = \frac{vEh [3h(a + 0.6h)^2 + h^3]^{1/2}}{4 [(a + 0.6h)^3 + h^2a]} \quad \dots(14)$$

Where: K - crack tip stress intensity

a - crack length

E - elastic modulus

v - load line displacement

h - $\frac{1}{2}$ specimen height

At short crack lengths the stress intensity decreases quickly with crack length, while at longer crack lengths the decrease is much slower, as shown in figure 34. Due to the decreasing stress intensity, by measuring the crack length with time, the crack growth rate for a range of stress intensities can be determined as illustrated in figure 29.

As the bolt load is applied the stress intensity at the tip of the machined notch will increase until it reaches the fracture toughness (K_C) of the material. At this load the "pop-in" crack will grow until its length corresponds to a stress intensity just below the fracture toughness. Hence, by measuring the "pop-in" crack length the fracture toughness can be calculated from equation (14). However, since double cantilever beam samples do not meet the geometry

specifications of ASTM E399 ⁶⁴ (Plane Strain Fracture Toughness of Metallic Materials) the values of toughness obtained cannot be considered as K_{1C} values. Nevertheless, relative values of toughness can be obtained, allowing comparison of the variously heat treated conditions.

Crack lengths were measured on both sides of the specimen using a travelling microscope, with a vernier scale attachment. A scribe line of known distance from the load line, was marked on the specimen close to the machined notch. The distance from the scribe line to the crack tip was measured after various time intervals, and added to the distance between the scribe line and the load line to give the total crack length. A series of computer programs, described in Appendix A, were used to calculate crack growth rates and stress intensities.

The stress intensity values obtained by measuring the crack surface length will always be high because of crack tunnelling (midsection crack longer than surface crack). Tunnelling, even if only minor, would make a large difference to the stress intensity for short crack lengths because of the sharp drop in stress intensity with crack length, figure 34. The error in stress intensity introduced by crack tunnelling at longer crack lengths would be considerably

smaller since the slope of the stress intensity versus crack length curve decreases sharply. A correction for crack tunnelling could only be applied after the stress corrosion tests on the double cantilever beam specimens were completed and the specimens were broken open. The boundary between the "pop-in" crack and the stress corrosion crack was visible on the fracture surface, figure 35. By measuring the "pop-in" crack at the specimen midsection (using a low power travelling microscope) the difference between the midsection and surface crack lengths was determined. Half the difference was then added to all the measured surface crack lengths so that a crack growth rate versus stress intensity curve, corrected for tunnelling, could be obtained. A corrected crack growth rate curve is compared to an uncorrected curve in figure 36. Applying the tunnelling correction only affects the stress intensity values. Also, as explained, it is only at high stress intensities, that is short crack lengths, that significant changes in stress intensity due to tunnelling occur. The tunnelling correction was applied to the fracture toughness values also.

6.2.4 Tracker Tests.

Before testing the double cantilever beam samples just described an initial series of sub-size double cantilever

beam samples were machined from Tracker wing-lock fittings and tested in a similar manner as just outlined, however no allowance was made for heat-up times. For these tests grid lines spaced at 1 mm were photoetched onto the specimens (by a procedure outlined in Appendix B), figure 37. Consequently the distance from the crack tip to the nearest grid line only needed to be measured. Crack length could be measured faster with the grid lines, however the surfaces with grid lines were stained by the seawater solution quickly, making the crack tips hard to discern.

6.2.5 Electron Microscopy

The small size of the precipitate particles that are affected by heat treatment necessitated the examination of thin-foils of material at high magnification. The thin-foil samples were machined from the threaded section of extruded bar tensile specimens and subsequently electrolytically polished to a thickness that would be transparent to the microscope electron beam. The details of the thin-foil specimen preparation are given in Appendix C.

7

7. RESULTS AND DISCUSSION

7.1 Tracker Tests

The double cantilever beam stress corrosion tests on material from Tracker wing-lock fittings resulted in similar stress corrosion crack growth rates for RRA heat treated material and T6 material, a typical curve is shown in figure 29. The similar stress corrosion resistance of T6 and RRA heat treated wing-lock fittings was confirmed with electrical conductivity measurements which were typical of T6 material, (see Appendix D). The Tracker wing-lock fittings which had been overaged to the T73 condition exhibited very slow crack growth rates and higher electrical conductivities. The disappointing response of the material to RRA treatments was attributed to the heat treatment procedure. In particular, since retrogression times can be short (e.g. 1 minute at 220°C), the double cantilever beam billet center sections would not receive the full retrogression treatment, because of insufficient through heating. Consequently, before the rest of the RRA treatments described in this thesis were performed the heat-up time at the center section of the double cantilever beam specimens, was determined as outlined previously. Henceforth, before the desired retrogression times were applied, the specimens were preheated for the

measured heat-up time, to ensure the center section received the full heat treatment.

7.2 Extruded Bar Material

7.2.1 Tensile Results.

The effect of retrogression treatments on the yield strength of extruded 7075-T6 bar was determined at each retrogression temperature tested (160, 180, 200 and 220°C). The results for retrogression times up to 60 minutes are shown in figure 38, along with the 7075-T6 and T73 minimum yield strengths for extruded bar. The 7075-T6 extruded bar used for retrogression had an "as received" yield stress of 496 MPa, which is very close to the T6 minimum. It is clear from figure 38 that the general shape of the retrogression curves obtained are similar to those of Cina⁵⁵, figure 28. The trends that were evident in figure 28 are repeated in figure 38. They are : higher retrogression temperatures cause the initial yield strength (or hardness) minimum to be lowered, and secondly, longer retrogression times to the yield strength minimum at lower temperatures. The important difference between the results of figure 38 and Cina's results (figure 28) are the longer times to the minimum in the retrogression curves. For example: for retrogression at 200°C

the minimum yield strength occurs at a retrogression time of 5 minutes (figure 38), whereas previously the minimum occurred only after 35 seconds (figure 28). Furthermore, at the lower retrogression temperature of 160°C the yield strength minimum occurs after 15 minutes. This indicates that the kinetics of the reaction occurring during retrogression are slowed down at lower retrogression temperatures. With longer retrogression times the process can be applied to thick section parts more easily, since the center section heat-up time is a smaller portion of the total retrogression time, hence the heat treatment applied to the material cross-section is more uniform.

The reaging response of retrogressed material is illustrated in figure 39, from which several points should be noted. Firstly, the retrogressed and reaged material has yield strengths well above the minimum T6 value and the "as received" T6 value for the particular extruded rod tested. This is due to the fact that, as mentioned, the T6 condition is a slight underage and further aging can cause an increase in strength before overaging. The second feature to note from figure 39 is that even after retrogression for 1 hour at 200°C , reaging, returns strength levels to above the original T6 values. This retrogression time is well past the time corresponding to the initial minimum in the retrogressed curve, and is even beyond

the secondary hardening peak in the curve. Indeed, all of the retrogressed data points of figure 38 (except 30 minutes and 60 minutes at 220°C) had reaged strength levels in excess of the T6 minimum.

RRA treatments did not affect the tensile ductility. All of the samples tested had elongations of between 8.5 and 10% (see Appendix D) which is above the T6 and T73 minimum of 7% ²¹. Ultimate tensile strengths (given in Appendix D) followed the same trends as the yield strength.

7.2.2 Electrical Conductivity Results.

Electrical conductivity measurements on the tensile samples indicated that during retrogression the conductivity continually increases, figure 40. An important point is that reaging causes a further increase in conductivity of about 0.5% IACS, hence stress corrosion resistance should continually increase during RRA. In contrast, the tensile strength decreases during retrogression and is then restored during reaging. Reaging then, appears to increase both the yield strength and stress corrosion resistance.

The extruded bar from which the tensile samples were machined had an "as received" T6 electrical conductivity

of 35.5% IACS; above the T6 minimum of 32% IACS. Retrogression for 31 minutes or longer at 180°C caused the conductivity to increase to at least the T73 minimum, figure 40. As mentioned, in reference to figure 38, retrogression times of up to 60 minutes at 180°C resulted in T6 yield strengths after reaging. Consequently, retrogression between 31 and 60 minutes results in T6 strength and T73 stress corrosion resistance. Similar ranges for retrogression times can be determined for the other retrogression temperatures tested, from the curves shown in figure 41. It should be noted that the retrogression times required to reach the minimum T73 conductivity (e.g. 31 minutes at 180°C or 5 minutes at 220°C) are longer than the retrogression times corresponding to the minimum in the retrogressed yield strength curves, figure 38. It would seem, then, that the retrogressed strength minimum may not represent a critical point with respect to mechanical properties, as was concluded by Cina⁵⁹.

It is evident from figure 41, that the electrical conductivity increases faster at higher retrogression temperatures. This supports the conclusion of the previous section, that the retrogression reaction is slowed down at lower retrogression temperatures.

7.3 Plate Material

From the extruded bar tensile and electrical conductivity results obtained particular RRA treatments were selected for application to 2.5 cm plate material. Additionally, T651 and T7351 material was tested for comparative purposes. The retrogression treatments chosen fall into three groups, short times, intermediate times and long times, which are shown schematically in figure 42. In "short time" treatments the retrogression time corresponds to the initial yield strength minimum in the retrogression curve. These treatments should verify whether in fact the retrogressed yield strength minimums represent a critical point with respect to mechanical properties. The second group of treatments, "intermediate time", have longer retrogression times, but should still result in T6 strength after reaging. The longer retrogression times are more practical for thick section parts and since they should result in T6 strengths, the stress corrosion crack growth rates that result are of interest. The last group of treatments, "long times", have retrogression times equivalent to the T73 high temperature age. The "long time" retrogression temperatures of 160°C and 180°C are slightly below and above the T73 overaging temperature (177°C). These treatments should determine the influence of temperature variations and reaging on the T73 temper.

7.3.1 Tensile Results.

The results of tensile tests on the 2.5 cm thick plate are summarized in Tables V and VI. The results of Table V are for tensile samples machined from the largest half of a used double cantilever beam stress corrosion specimen. Table VI lists results for tensile specimens machined from the center of blanks 2.5 cm X 2.5 cm X 12.5 cm cut from the plate. The samples of Table VI were heat treated before machining and since they had the same cross-section as ~~double~~ cantilever beam specimen billets, the same retrogression heat-up times were applied (1.75 minutes and 3 minutes for 180°C and 220°C respectively).

The results of Tables V and VI have several general points of interest. Firstly, the T651 and T7351 tensile properties are all above the minimum requirements for these tempers (see Table II), except for the elongation of the transversely loaded T7351 material, which is slightly below the minimum requirement. Secondly, unlike the extruded bar, which could be retrogressed and reaged to strengths above the "as received" T6 strength, none of the retrogressed and reaged plate material had tensile properties (strength or percent elongation) superior to the T651 starting condition. However, several retrogressed and reaged conditions had

tensile properties well above the T651 minimum requirements.

Comparing the results of Table V and VI there are some notable differences. The largest differences are the higher tensile strengths in Table VI for material given the "intermediate time" heat treatment of 5 minutes at 220°C plus reaging and the "long time" treatment of 8 hours at 180°C plus reaging. The lower tensile strengths in Table V for these heat treatments could be due to several factors associated with the prior double cantilever beam stress corrosion testing of the material. Some of these factors are; 1) the long time period over which the material had been stressed by the double cantilever beam loading bolt; 2) the long time exposure to a corrosive solution (the seawater mixture applied during double cantilever beam testing); 3) the samples of Table V were machined from areas near the original surface of the plate - done out of necessity, because of the stress corrosion crack in the tested double cantilever beam samples -, whereas samples of Table VI were machined from the center of the plate stock, and the grain size in the short-transverse direction was larger at the plate centers compared to the surfaces; 4) experimental scatter. None of these factors can explain, by itself, the differences between Table V and VI. However, the factors listed may in some instances, combine to give erroneous results.

From the preceding discussion, it would seem that the results of Table VI show the effects of RRA treatments on tensile properties of plate material more accurately than the results of Table V.

It should be noted from Table VI that the "short and intermediate time" RRA treatments result in yield and ultimate strengths above the minimum T651 requirements, regardless of whether the tensile load is applied in the longitudinal or transverse direction. The percent elongation for "short and intermediate time" RRA treatments was either the minimum required for T651 material or just below. However, as mentioned, none of the RRA treatments resulted in strengths above the T651 material tested. It seems, then, that the reaging response, that is the ability to regain T6 strength during reaging, of plate material is not as good as for the extruded bar.

7.3.2 Electrical Conductivity Results.

Listed in Table VII are the electrical conductivities of the variously heat treated double cantilever beam specimens. The conductivity of the "as received" T651 plate was 34.5% IACS and overaging to the T73 temper (8 hours at 177°C) increased the conductivity to 42.4% IACS, which is well above the

minimum for T73 material (38% IACS). The conductivity of "short time" RRA treatments is below the T73 minimum, but 2 to 3% IACS above the "as received" values. Consequently, "short time" RRA material should have improved stress corrosion crack growth rates compared to T651 material. However, stress corrosion crack growth rates are probably not equivalent to T7351 material. The "intermediate time" RRA conductivities are above the T73 minimum (38% IACS), but not as high as the T73 sample tested. For both the "short and intermediate time" RRA treatments, the 220°C retrogression temperature results in higher conductivities than the 180°C retrogression temperature, even though the retrogression times are much shorter. The smaller rise in conductivity at lower retrogression temperatures, once again, indicates that the kinetics of the reaction occurring during retrogression is slowed down at lower temperatures. This effect is repeated with the "long time" RRA treatments. The 8 hours at 180°C plus reaging treatment resulted in a conductivity equivalent to the T73 specimen tested, whereas 8 hours at 160°C plus reaging has a conductivity only slightly above the T73 minimum. The conductivities of the RRA double cantilever beam specimens tested span the range from the "as received" T651 value to the T7351 value. Hence, a range of stress corrosion crack growth rates should result.

7.3.3 Stress Corrosion Results

In figure 43 the stress corrosion crack growth rate scatterbands resulting from "short time" RRA treatments are compared to the "as received" T651 and the T7351 crack growth rates. The stage II plateau crack growth rates of the "short time" treatments are lower than the T651 condition and stage I stress intensities are higher. However, as expected from the conductivity results, the stress corrosion crack growth rates of "short time" RRA treatments are not comparable to those of the T7351 condition. The stress corrosion crack growth rates resulting from "intermediate and long time" RRA treatments are shown in figure 44. Some of the RRA treatments illustrated had considerably improved stress corrosion crack growth rates compared to the T651 condition. Although all the RRA treatments tested had improved stress corrosion crack growth rates compared to T651 material, only the treatments that resulted in conductivities comparable to the T7351 condition, had crack growth rates comparable to the T7351 condition: for example, the "intermediate time" treatment of 5 minutes at 220°C plus reaging, and the "long time" treatment of 8 hours at 180°C plus reaging. Both of these treatments have retrogression times much longer than that required to reach the initial minimum on the extruded bar retrogressed yield strength curves, figure 39. In fact, the stress corrosion crack growth rates resulting from retro-

gression to the yield strength minimums ("short time" treatments, figure 43), had only slightly improved stress corrosion crack growth rates and conductivities compared to the T651 condition. From these results a number of things are apparent. Firstly, the initial minimum in the retrogressed yield strength curve does not result in the optimum combination of stress corrosion and tensile properties. As well, stress corrosion resistance increases with increasing electrical conductivity. Of particular significance are the properties resulting from the "intermediate time" treatment of 5 minutes at 220°C plus reaging. This condition had stress corrosion crack growth rates comparable to the T7351 temper, figure 44, and tensile properties that meet the T651 minimum requirements, Table VI.

Retrogression for 8 hours at 180°C should result in a material condition very similar to the T73 temper, since the recommended treatment to overage T6 material to the T73 temper is 8 hours at 127°C ²⁹. Thus the RRA treatment of 8 hours at 180°C plus reaging is essentially the T73 temper reaged (29 hours at 121°C). The electrical conductivity of the T73 temper and the treatment of 8 hours at 180°C plus reaging are identical (Table VIII), as are their stress corrosion crack growth rates, figure 44. However, reaging the T73 condition did not cause an increase or decrease in

tensile yield or ultimate strength (see Table VI). Hence, there is nothing to be gained by reaging T7351 material.

7.3.4 Fracture Toughness Results.

The short-longitudinal fracture toughness values obtained by measuring the initial "pop-in" crack lengths of double cantilever beam specimens are given in Table VIII, along with the electrical conductivity of each condition. As mentioned, these toughness values cannot be considered K_{IC} values, as defined by ASTM E399⁶, however they do provide an indication of the relative toughness of each heat treated condition. It is clear from Table VIII that RRA treatments can improve the fracture toughness. Plotting fracture toughness against electrical conductivity, which has been shown to increase during RRA treatments, shows the trend of fracture toughness properties clearly, figure 45. The fracture toughness steadily increases with conductivity up to approximately 40% IACS, after which a sharp rise occurs.

The sudden increase in toughness can be explained by considering the stress state at the crack tip. Material at the midsection of the specimen is constrained in the direction of the width of the specimen, by the material around it, while material near the surfaces of the specimen

can deform in the width direction. Consequently, the plastic zone size at the midsection is smaller than at the surfaces, as illustrated in figure 46⁸, and because of the constraints the midsection is in plane strain while the surfaces are in plane stress. In figure 45 the calculated fracture toughness values from the "pop-in" cracks are plotted, regardless of whether the crack tip plastic zone is in a plane stress or plane strain stress state. However, as the fracture toughness increases the load line displacement to cause "pop-in" cracking also increases, which results in a larger proportion of the crack front in plane stress rather than plane strain. Hence, in order to obtain valid plane strain fracture toughness values for high toughness materials, a wide specimen is required. Also, as the load line displacement increases there is a larger tendency toward a "hinging" action at the notch tip, and, in this case the crack tip will not be in plane strain. To prevent this a specimen with sufficient height to prevent "hinging" is required.

For compact tension specimens valid plane strain fracture toughness values are obtained when the following criteria is met⁶⁴:

$$a \text{ \& } B \geq 2.5 \left(\frac{K_C}{Y.S.} \right)^2 \quad \dots (15)$$

where: a - crack length
B - specimen width
 K_C - plane strain fracture toughness
Y.S. - yield strength

For the double cantilever beam specimens tested the width "B" was 25.4 mm and the crack length "a" (the distance from the bolt tip to the end of the machined notch) 8.9 mm. The yield strengths are known and hence the maximum values of K_C that can be validly determined with the double cantilever beam specimen geometry can be calculated. These maximum values of K_C are $29.8 \text{ MPa}\sqrt{\text{m}}$ and $47.3 \text{ MPa}\sqrt{\text{m}}$ for the crack length and specimen width criteria respectively. The lower of these two values is the limiting value in practice. In figure 45 the fracture toughness values exceeding the valid K_C values obtainable with the double cantilever beam specimen geometry are indicated.

The "invalid" data points of figure 45 are essentially plane stress fracture toughness values or mixed mode values rather than plane strain values. Since a plane stress condition is not as constrained as plane strain, more deformation and yielding can occur. In figure 47 the "pop-in" crack profiles of two double cantilever beam specimens are shown. The T651 specimen of figure 47a met the crack length and

specimen width requirements of equation (15), while the RRA specimen of figure 47b did not. Thus figure 47a illustrates a predominantly plane strain crack with no deformation at the specimen surfaces present; while in contrast figure 47b shows a plane stress or mixed mode crack profile and the resulting surface yielding is apparent. It should also be noted that the crack in the T651 sample of figure 47a, occurred suddenly while the bolt load was applied and the appearance of the "pop-in" crack could be associated with a definite load line displacement. While loading the specimen of figure 47b audible clicks were heard as the crack formed, however no crack could be seen at the specimen surfaces. Consequently, the appearance of the crack could not unequivocally be associated with a particular load line displacement. The crack in figure 47b, then, cannot be considered a true "pop-in" crack.

Notwithstanding the factors just outlined, the results of figure 45 clearly indicate that fracture toughness increases with electrical conductivity. This implies that RRA treatments increase fracture toughness. Even considering only the valid data points of figure 45, a 21% increase in toughness occurred during RRA. If valid plane strain fracture toughness values were obtained for the RRA conditions that gave invalid fracture toughness data from the double cantilever beam specimens, a larger percentage increase in toughness would likely be observed.

7.4 Electron Microscopy Results

The transmission electron microscopy was carried out with the aim of establishing the microstructural changes occurring during retrogression and reaging. Of particular interest were microstructural changes that would lend support to the proposed mechanism for stress corrosion cracking presented in chapter 4. The essence of the mechanism outlined was that at large grain boundary precipitates bubbles of molecular hydrogen form. The formation of molecular hydrogen bubbles would decrease the concentration of hydrogen in interstitial solid solution which can lower the cohesive atomic bond strength, and thus accelerate stress corrosion crack growth. If such a mechanism for stress corrosion cracking prevails, then the more stress corrosion resistant material conditions, should have larger grain boundary precipitate sizes. In addition to interest in the grain boundary precipitate size, the intragranular precipitate morphology was examined so that an explanation for the loss of tensile strength during retrogression and subsequent increase in tensile strength during reaging could be obtained.

Only four material conditions were selected for electron microscopy, because of the involved specimen preparation required (see Appendix C). Two of these material conditions were the standard T6 and T73 tempers, which were examined so

as to provide a basis for comparison with RRA treatments. The retrogression and reaging heat treatment of 5 minutes at 220°C plus 29 hours at 121°C was examined after retrogression as well as after reaging. This particular RRA treatment was chosen because both plate and extruded bar in this condition had tensile properties meeting the T6 minimum requirements (Table VI and Appendix D), as well as electrical conductivities above the T73 minimum (Table VII and figure 41). In addition, plate material retrogressed for 5 minutes at 220°C and reaged exhibited stress corrosion crack growth rates very similar to T7351 material (figure 44).

As mentioned, an important objective of the electron microscopy was the determination of the grain boundary precipitate size of each of the four samples examined. In order to do this, grain boundaries must be viewed tilted rather than 'straight on'. In figure 48, approximately the same grain boundary region is shown 'straight on', figure 48a, and tilted, figure 48b. By viewing the grain boundary as shown in figure 48a, individual grain boundary precipitates appear superimposed on one another, or are not visible at all. In figure 48b, however, individual precipitates lying on the grain boundary are discernable, and their size and spacing can be distinguished. Measurement, using an eyepiece, of approximately 20 to 30 typical grain boundary precipitates of T6 samples, such as shown in figure 48, resulted in an average grain

boundary precipitate size of approximately 140 Å.

Figure 49 illustrates the grain boundary structures resulting from the retrogression treatment of 5 minutes at 220°C, figure 49a, and this same retrogression treatment plus reaging, figure 49b. Comparing these conditions to the T6 sample of figure 48b, it is seen that retrogression and reaging treatments definitely result in more closely spaced grain boundary precipitates. Measurement of the grain boundary precipitates yielded the sizes of 230 Å for the retrogressed condition and 240 Å for the retrogressed and reaged condition. It should be recalled that the two material conditions of figure 49 displayed higher electrical conductivities and far superior stress corrosion crack growth rates than the T6 sample of figure 48.

Figure 50 shows the grain boundary structure of the T73 condition; the most stress corrosion resistant condition. Measurement of the grain boundary precipitates of the T73 condition gave an average size of 300 Å. The grain boundary precipitates of figure 50 are visibly larger than those of figure 48 or 49.

It is apparent that during retrogression for 5 minutes at 220°C the grain boundary precipitate size increases from

approximately 140 Å to 230 Å, with reaging causing a further increase to 240 Å. This increasing precipitate size corresponds to increases in electrical conductivity and stress corrosion resistance. It is possible, then, that the improved stress corrosion resistance of T73 and RRA material is due to the formation of hydrogen bubbles at grain boundary precipitates larger than some critical size, as previously outlined. It will be recalled from chapter 4 that the critical grain boundary precipitate size for hydrogen bubble formation in Al-6%Zn-3%Mg was established to be about 200 Å⁵⁸. If the present stress corrosion cracking results in 7075 are due to a similar hydrogen-precipitate interaction, it would appear that the critical precipitate size for hydrogen bubble formation would be in the range of 140 Å to 230 Å.

Further evidence of hydrogen bubble formation was sought by examining thin-foils that had been exposed to moist laboratory air for various periods of time. Evidence supporting the hydrogen bubble phenomena was found in retrogressed and reaged samples exposed to moist air for 14 to 28 days, figure 51. As reported by Alani and Swann⁵⁶ and Scamans⁵⁷, the hydrogen bubbles themselves are not visible, but are revealed by the fringes around them resulting from the strong elastic strain field that surrounds them (caused by pressure within the bubble⁵⁶). The strain fringes shown in figure 51 are nearly identical to those observed by Scamans⁵⁷, figure 52,

which were attributed to hydrogen bubbles. The strain fringes cannot be a result of grain boundary precipitates, since none are associated with the grain boundary precipitates of the retrogressed and reaged sample of figure 49b. Hydrogen bubbles were not observed in any of the other material conditions examined, but this does not necessarily preclude their formation in these conditions in other corrosive environments.

In figures 48 to 51 the intragranular precipitate structure is apparent to varying extents. The Guinier-Preston zones present in the T6 condition (giving the T6 condition its higher tensile strength) are seen as the fine background matrix mottling, evident in figure 48b. The varying brightness of the background is caused by curvature of the thin-foil specimen surfaces. After retrogression for 5 minutes at 220°C the matrix appears more homogeneous indicating a lower density and/or size of the GP zones, figure 49a. The structure shown in figure 49a is typical of the retrogressed samples examined. Since GP zones do not form during aging treatments above 130°C ²⁴, it would be expected that during retrogression the GP zones would be partially solutionized, resulting in the lower tensile strength for retrogressed material and the lower density and/or size of the GP zones observed in figure 49a.

After reaging at 121°C (the same temperature as the T6 age) GP zones are once again present, as evidenced by the background mottling in figure 49b and 51. As a result of the reformation of GP zones during reaging the tensile strength is restored to values similar to the T6 temper. In the T73 temper, figure 50, the GP zones have been overaged to form metastable MgZn_2 precipitates, causing the loss in strength. The precipitate in the T73 condition is clearly coarser than in the RRA or T6 conditions. The precise microstructural changes occurring during retrogression will need to be established by more detailed electron microscopy. However, the explanation given here must be consistent with the fact that the changes are, at least in part, reversible. Hence a model of partial GP zone solutioning followed by reprecipitation during reaging is plausible.

In summary, retrogression for 5 minutes at 220°C partially solutionizes the intragranular GP zones, present in the T6 temper, which results in the loss of strength. Concurrently, the grain boundary precipitates coarsen, giving increased stress corrosion resistance. Reaging allows the intragranular GP zones to reform, restoring strength, as well as producing a further small increase in grain boundary precipitate size. Overaging to the T73 temper causes coarsening of grain boundary and intragranular precipitates, which lowers strength and

increases stress corrosion resistance. Increasing the grain boundary precipitate size above a critical value improves stress corrosion resistance, since the precipitates can act as nucleation sites for hydrogen bubbles, substantially reducing the hydrogen embrittlement of grain boundaries, that would occur during stress corrosion.

The increase in hardness, after the initial minimum, in the retrogressed hardness curve, figure 27, could be due to coarsening of the undissolved GP zones. At the retrogression temperatures these undissolved GP zones would quickly nucleate into precipitate particles, which, with further retrogression time, would become overaged, resulting in the decreasing hardness at the longer retrogression times. Once overaging occurs, reaging can no longer result in the reformation of GP zones, as was the case for short retrogression times. Consequently, after long retrogression times reaging increases the hardness only a small amount, or not at all.

8.0. CONCLUSIONS

1. There is no relationship between stress corrosion resistance and yield strength. This implies that the traditional trade-off between stress corrosion resistance and yield strength, in choosing between the T73 and T6 tempers, is unnecessary.
2. The initial minimum in the extruded bar retrogressed yield strength curve does not correspond to the retrogression time giving the optimum combination of strength and stress corrosion resistance, as was concluded by Cina⁵⁹.
3. Lower retrogression temperatures cause the kinetics of the reaction occurring during retrogression to be slowed down, leading to longer times to the minimum in the retrogressed yield strength curve.
4. Retrogression and reaging treatments on the 1.9 cm diameter extruded bar material used in this thesis resulted in longer times to the minimum yield strength on the retrogression yield strength curves, compared to those reported by Cina⁵⁹.

TABLE IV

Stress Corrosion Results - Ciná 59

Material Condition	Number of Tests	Time to Failure loaded to 75% of yield stress (hours)
T6	4	0.68
T6 + 120 sec/200°C, W.Q. + 16 h/121°C	5	30.7
T6 + 60 sec/220°C, A.C. + 24 h/121°C	2	37.0
T6 + 15 sec/240°C, A.C. + 48 h/121°C	4	38.0

5. During retrogression and reaging the electrical conductivity increases and is proportional to the stress corrosion resistance.
6. Extruded bar and plate material can be retrogressed and reaged to electrical conductivities above the T73 minimum, and to tensile strengths above the T6 minimum; for example the treatment of 5 minutes at 220°C plus 29 hours at 121°C.
7. Retrogression and reaging treatments increase fracture toughness. Fracture toughness is proportional to electrical conductivity.
8. Plate material retrogressed for 5 minutes at 220°C and reaged had stress corrosion crack growth rates similar to the T7351 condition, while still meeting the T651 minimum tensile strength requirements in the longitudinal and transverse directions.
9. Plate material could not be reaged to above the "as received" T651 tensile strength - although it could be reaged to above the T651 minimum tensile strength requirements. Extruded bar material could be reaged above the "as received" tensile strength, however the

extruded bar "as received" T6 strength was only 1 MPa higher than the minimum requirement.

10. During "short and intermediate time" retrogression treatments the T6 Gunier-Preston zones are partially dissolved causing the loss in tensile strength observed. Subsequent reaging allows Gunier-Preston zones to reform restoring strength to T6 levels.
11. During retrogression and reaging treatments the grain boundary precipitate size increases and this corresponds to the increasing stress corrosion resistance.
12. Bubbles of molecular hydrogen form at grain boundary precipitates in the retrogressed and reaged condition. Their formation lowers the concentration of atomic hydrogen in interstitial solid solution. This, in turn, increases the grain boundary cohesive strength, resulting in decreased stress corrosion crack growth rates.

REFERENCES

1. Cohen, B.A.; "Corrosion Fatigue in the Aerospace Industry", NACE-2, Houston, 1972, p. 65.
2. Ritchie, R.O.; "Near-Threshold Fatigue Crack Propagation in Steels", Int. Met. Rev., No. 5,6, 1979, p. 205.
3. Beevers, C.J.; "Some Aspects of Fatigue Crack Growth in Metals and Alloys", Fracture 1977, Vol. 1, ICF4, Waterloo, Canada, June 19-24, 1977, p. 239.
4. Hudak, S.J.; Saxena, A.; Bucci, R.J.; Malcolm, R.C.; "Development of Standard Methods for Testing and Analyzing Fatigue Crack Growth Data", AFML-TR-78-40.
5. Wanhill, R.J.H.; "Microstructural Influences on Fatigue and Fracture Resistance in High Strength Structural Materials", Eng. Fract. Mech., Vol. 10, 1978, p. 337.
6. Speidel, M.O.; "Corrosion Fatigue Crack Growth in High Strength Aluminum Alloys", ICAF Symposium, June 2-5, 1975, Lausanne, Switzerland.
7. Plumbridge, W.J.; Ryder, D.A.; "The Metallography of Fatigue", Met. & Mat. and Metall. Revs., Vol. 3, No. 8, 1969, p. 119.
8. Plumbridge, W.J.; "Review: Fatigue Crack Propagation in Metallic and Polymeric Materials", J. Mat. Sci., No. 7, 1972, p. 939.
9. Broek, D.; "Elementary Engineering Fracture Mechanics", Sijthoff & Noordhoff, 1978.

10. Broek, D.; "The Effect of Intermetallic Particles on Fatigue Crack Propagation in Aluminum Alloys", Fracture 1969, Proc. of 2nd Int. Conf. on Fracture, Brighton, England, p. 754.
11. Ryan, N.E.; "Fatigue Crack Growth in D6AC Steel Heat Treated to Different Values of Fracture Toughness", Dept. of Defense, Aero. Res. Labs., Melbourne, AR-001-734.
12. Wanhill, R.J.H.; "Environmental Effects on Fatigue of Aluminum and Titanium Alloys", AGARD-R-659, Apr. 1977, p.2-1.
13. Selines, R.J.; Pelloux, R.M.N.; "Effect of Cyclic Stress Wave Form on Corrosion Fatigue Crack Propagation in Al-Zn-Mg Alloys", Met. Trans., Vol. 3, Sept. 1972, p. 2525.
14. Barsom, J.M.; "Effect of Cyclic Stress Form on Corrosion Fatigue Crack Propagation Below K_{ISCC} in a High Yield Strength Steel", NACE-2, Houston, 1972, p. 424.
15. Pelloux, R.M.N.; "Corrosion-Fatigue Crack Growth", Fracture 1969, Proc. of 2nd Int. Conf. on Fracture, Brighton, England, p. 731.
16. Wanhill, R.J.H.; "Formation of Brittle Fatigue Striations", Corrosion-NACE, Vol. 31, No. 2, 1975, p. 66.
17. Hyatt, M.V.; Speidel, M.O.; "High Strength Aluminum Alloys", chap. 4 from "Stress Corrosion Cracking in High Strength Steels and in Titanium and Aluminum Alloys", ed. B.F. Brown, Naval Research Lab.
18. Hyatt, M.V.; "Use of Precracked Specimens in Stress Corrosion Testing of High Strength Aluminum Alloys", Corrosion-NACE, Vol. 26., No. 11, 1970, p. 487.

19. Smith, H.R.; Piper, D.E.; "Stress Corrosion Testing with Precracked Specimens", chap. 2, from, "Stress Corrosion Cracking in High Strength Steels and in Titanium and Aluminum Alloys", ed. B.F. Brown, Naval Research Laboratory, United States.
20. Speidel, M.O.; "Current Understanding of Stress Corrosion Crack Growth in Aluminum Alloys", from "The Theory of Stress Corrosion Cracking in Alloys", ed. J.C. Scully, NATO-Scientific Affairs Division, Brussels, 1971.
21. "Aluminum Standards and Data", the Aluminum Association Incorp. sixth edition, 1979.
22. Griffith, W.M.; "Recent Developments in High Strength Aluminum Alloys", Wright Patterson AFB, Ohio, 45433.
23. Staley, J.T.; "How Microstructure Affects Fatigue and Fracture of Aluminum Alloys", Int. Sym. on Fracture Mechanics, 1978, p. 671.
24. Hunsicker, H.Y.; "The Metallurgy of Heat Treatment", chap. 5 from, "Aluminum", Vol. 1, ed. K. Van Horn, ASM, Metals Park, Ohio, 1967.
25. United States Military Specifications; "Heat Treatment of Aluminum Alloys", MIL-H-6088E, Feb. 5, 1971.
26. ASM Metals Handbook, ninth edition; "Volume 2, Properties and Selection: Nonferrous Alloys and Pure Metals", ASM, Metals Park, Ohio, 1979.
27. Koch, G.H.; Kolijn, D.T.; "The Heat Treatment of Commercial Aluminum Alloy 7075", J. of Heat Treating, Vol. 1, No. 2, 1979, p. 3.

28. Lifka, B.W.; Sprawls, D.O.; Kaufman, J.G.; "Exfoliation and Stress-Corrosion Resistance of High Strength Heat Treatable Aluminum Alloy Plate", Corrosion-NACE, Nov. 1967, p. 335.
29. Alcoa Green Letter, "Alcoa Alloy 7075-T73", Sept. 1971.
30. Speidel, M.O.; "Interaction of Dislocations with Precipitates in High Strength Aluminum Alloys and Susceptibility to Stress Corrosion Cracking", NACE, Houston, 1969, p. 561.
31. Coyne, Jr., E.J.; Starke, Jr., E.A.; "The Effect of Microstructure on the Fatigue Crack Growth Behaviour of An Al-Zn-Mg-(Zr) Alloy", Int. J. of Fracture, Vol. 15, No. 5, 1979, p. 405.
32. Fu-Shiong Lin; Starke, Jr., E.A.; "The Effect of Copper Content and Degree of Recrystallization on Fatigue Resistance of 7XXX Type Aluminum Alloys; I, Low Cycle Corrosion Fatigue.", Met. Sci. and Eng., Vol. 39, 1979, p. 27.
33. Knot, J.F.; Pickard, A.C.; "Effects of Overload on Fatigue Crack Propagation: Aluminum Alloys", Met. Sci., Aug./Sept., 1979, p. 399.
34. Wei, R.P.; Landes, J.D.; "Correlation Between Sustained-Load and Fatigue Crack Growth in High Strength Steels", Mat. Res. & Stds, Vol. 9. No. 7, 1969, p. 25.
35. Gerberich, W.W.; Birat, J.P.; Zackay, V.F.; "On The Superposition Model For Environmentally Assisted Fatigue Crack Growth", NACE-2, Houston, 1972, p. 396.

36. Atkinson, J.D.; Lindley, T.C.; "The Effect of Frequency and Temperature on Environmentally Assisted Fatigue Crack Growth Below K_{Isc} in Steels", I. Mech. E., Vol. 4, 1977, p. 65.
37. Parkins, R.N.; Greenwell, B.S.; "The Interface Between Corrosion Fatigue and Stress-Corrosion", Met. Sci., No. 11, 1977, p. 405.
38. Dawson, P.B.; Pelloux, R.M.; "Corrosion Fatigue of Titanium Alloys in Aqueous Environments", Met. Trans., Vol. 5, 1974, p. 723.
39. Austen, I.M.; Walker, E.F.; "Quantitative Understanding of the Effects of Mechanical and Environmental Variables on Corrosion Fatigue Crack Growth Behaviour", I. Mech, E., Vol. 4, 1977, p.1.
40. Endo, K.; Kamai, K.; Yamato, J.; "Threshold Stress-Intensity Under Cyclic Stress Corrosion Cracking", Proc. of 22nd Japan Congress on Met. Res., 1979, p. 199.
41. Wei, B.P.; "Some Aspects of Environmentally-Enhanced Fatigue Crack Growth", Eng. Fract. Mech., Vol. 1, 1970, p. 633.
42. Scamans, G.M.; "Evidence for Crack Arrest Markings On Intergranular Stress Corrosion Fracture Surfaces of Al-Zn-Mg Alloys", Met. Trans. Vol. 11A, 1980, p. 846.
43. Austen, I.M.; McIntyre, P.; "Corrosion Fatigue of High Strength Steels in Low Pressure Hydrogen Gas", Met. Sci., Vol. 13, No. 7, 1979, p. 420.

44. Speidel, M.O.; Blackburn, M.J.; Beck, T.R.; Feeney, J.A.; "Corrosion Fatigue and Stress Corrosion Crack Growth in High Strength Aluminum Alloys, Magnesium Alloys, and Titanium Alloys Exposed To Aqueous Solutions", NACE-2, Houston, 1972, p. 324.
45. Tomkins, B.; "Role of Mechanics in Corrosion Fatigue", Met. Sci., Vol. 13, No. 7, 1979, p. 387.
46. Meyn, D.A.; "An Analysis of Frequency and Amplitude Effects on Corrosion Fatigue Crack Propagation in Ti-8Al-1Mo-IV", Met. Trans., Vol. 2, No. 3, 1971, p. 853.
47. Procter, R.P.M.; Moss, K.; "Corrosion-Fatigue of a High Strength Aluminum Alloy", 6th I.C.M.C., Sydney, 1975, p. 2-24.
48. Damage Tolerant Design Handbook, AFML, Wright Patterson AFB, MCIC-HB-01, Dec. 1972.
49. Parkins, R.N.; "Stress Corrosion Spectrum", British Corrosion Journal, Vol. 7, 1972, p. 15.
50. Thompson, A.W.; "Current Status of the Role of Hydrogen in Stress Corrosion Cracking", Mat. Sci. and Eng., Vol. 43, 1980, p. 41.
51. Thompson, A.W.; Bernstein, I.M.; "Environmental Fracture of Aluminum Alloys and Stainless Steels as a Function of Composition and Microstructure", from "Stress Corrosion Cracking", ed. J. Yakalom, A. Aladjem, Freund Publishing House, Tel-Aviv, 1980.

52. Jacobs, A.J.; "The Role of Dislocations in Stress Corrosion Cracking of 7075 Aluminum Alloy", Trans. of the ASM, Vol. 58, 1965, p. 579.
53. Helfrich, W.J.; "Fractography of Stress Corrosion Cracks in Aluminum Alloy 7075", Corrosion-NACE, Vol. 29, No. 8, 1973, p. 316.
54. Speidel, M.O.; "Hydrogen Embrittlement of Aluminum Alloy?", Proc. of an Int. Conf. on the Effects of Hydrogen on Materials Properties and Selection and Structural Design, 23-27 Sept. 1973, Seven Springs, Pa., p. 248.
55. Scamans, G.M.; Alani, R.; Swann, P.R.; "Pre-Exposure Embrittlement and Stress Corrosion Failure in Al-Zn-Mg Alloys", Corrosion Science, Vol. 16, 1976, p. 443.
56. Alani, R.; Swann, P.R.; "Water Vapour Embrittlement and Hydrogen Bubble Formation in Al-Zn-Mg Alloys", British Corrosion Journal, Vol. 12; No. 2, 1977, p. 80.
57. Scamans, G.M.; "Hydrogen Bubbles in Embrittled Al-Zn-Mg Alloys", J. of Materials Science, Vol. 13, 1978, p. 27.
58. Christodoulou, L.; Flower, H.M.; "Hydrogen Embrittlement and Trapping in Al-6%Zn-3%Mg", Acta Metallurgica, Vol. 28, 1980, p. 481.
59. Cina, B.; Ranish, B.; "New Technique for Reducing Susceptibility to Stress Corrosion of High Strength Aluminum Alloys", in "Aluminum Industrial Products", Pittsburgh Chapter, American Society for Metals, 1974.

60. United States Patent, 3856584, 24 Dec. 1974.
61. ASTM E8-79a;"Standard Methods of Tension Testing of Metallic Materials", 1980 Annual Book of ASTM Standards, Part 10, p. 197.
62. ASTM-D-1141-75;"Standard Specification for Substitute Ocean Water", 1980 Annual Book of ASTM Standards, Part 31, p. 1044.
63. Mostovoy, S.; Crosley, P.B.; Ropling, E.J.;"Use of Crack Line Loaded Specimens for Measuring Plane Strain Fracture Toughness", J. of Materials, Vol. 2, No. 3, 1967, p. 661.
64. ASTM E399-78a; "Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials", 1980 Annual Book of Standards, Part 10, p. 580.
65. Hunsicker, H.Y.;"Development of Al-Zn-Mg-Cu Alloys for Aircraft", Aluminum Company of America, Alcoa Center, Penns. 15069.

TABLE I

Composition of Some 7000 Series Aluminum Alloys

	7075	7475	7079
Zn	5.1 - 6.1	5.2 - 6.2	4.3
Mg	2.1 - 2.9	1.9 - 2.6	3.3
Cu	1.2 - 2.0	1.2 - 2.0	0.60
Fe	0.50	0.12	0.50
Si	0.40	0.10	0.40
Mn	0.30	0.06	0.20
Cr	0.18-0.35	0.18-0.25	0.20
Al	bal.	bal.	bal.

TABLE II

Mechanical Properties of Some 7000 Series
Aluminum Alloys.

Alloy (Plate)	Yield Stress (MPa) L†	Ultimate Stress (MPa) L	% Elongation L	K _{Ic} (MPa√m) ST§	SCC Threshold (MPa) ST	Electrical Conductivity % IACS
7075-T651	469 (495)ζ	537	7	19.8	<55	32
7075-T7351	393 (421)	476	7	22.0	>296	38
7475-T651	469	537	8	34.1	>276	
7475-T7351	393	476	8	36.3	>276	
7079-T651	470	540	7		<55	

†- L loaded in longitudinal direction

§- ST loaded in short transverse direction

ζ- numbers in brackets are minimum yield strengths
for extruded bar.

TABLE III

Heat Treatment Options For 7075

Temper	Solution Treatment	First Age	Second Age	Comments
T6	1.5 hours at 460°C	24 hours at 121°C		-high strength -poor corrosion resistance
T73	1.5 hours at 460°C	8 hours at 110°C	8 hours at 177°C	-lower strength -good corrosion resistance

TABLE IV

Stress Corrosion Results - Ciná 59

Material Condition	Number of Tests	Time to Failure loaded to 75% of yield stress (hours)
T6	4	0.68
T6 + 120 sec/200°C, W.Q. + 16 h/121°C	5	30.7
T6 + 60 sec/220°C, A.C. + 24 h/121°C	2	37.0
T6 + 15 sec/240°C, A.C. + 48 h/121°C	4	38.0

TABLE V

Tensile Results of Samples Machined from Used
Double Cantilever Beam Specimens

loaded in the longitudinal direction

Material Condition	Number of Tests	0.2% Yield Stress (MPa)	Ultimate Tensile Stress (MPa)	% Elongation
T651	4	543	588	8.6
T7351	1	507	566	7.3
"short time"				
1 m/220°C + RA *	2	521	572	7.5
15 m/180°C + RA	2	523	575	7.9
"intermediate time"				
5 m/220°C + RA	1	515	570	7.7
50 m/180°C + RA	2	459	527	7.4
"long time"				
8h/160°C + RA	2	479	547	7.9
8h/180°C + RA.	2	401	487	7.7

* all RRA treatments applied to T6 material

TABLE VI

Tensile Results of Samples Machined from
2.5 cm 7075 Plate

Material Condition	Number of Tests	0.2% Yield Stress (MPa)	Ultimate Tensile Stress (MPa)	% Elongation
loaded in the longitudinal direction				
T651	1	553	608	8.1
T7351	2	458	526	7.2
"short time" 1m/220°C + RA	2	537	589	6.5
15m/180°C + RA	2	543	593	7.0
"intermediate time" 5m/220°C + RA	3	501	555	7.0
"long time" 8h/180°C + RA	2	458	524	7.0
loaded in the transverse direction				
T651	2	548	606	8.1
T7351	1	453	521	6.9
"intermediate time" 5m/220°C + RA	2	502	554	6.6

TABLE VII

Electrical Conductivity of Heat Treated
2.5 cm 7075 Plate

Material Condition	Electrical Conductivity % IACS
T651	34.3
T7351	42.4
"short time" 1 m/220°C + RA 15 m/180°C + RA	37.5 36.5
"intermediate time" 5 m/220°C + RA 50 m/180°C + RA	40.4 38.3
"long time" 8 h/160°C + RA 8 h/180°C + RA	38.2 42.4

TABLE VIII

Short Transverse Fracture Toughness Results

Material Condition	Number of Tests	Fracture Toughness from Pop-in (MPa $\sqrt{m}$)	K _{1c} (MPa $\sqrt{m}$) ref. 65	Electrical Conductivity
T651	3	24.8	19.8	34.9
T7351	1	44.0	22.0	42.4
"short time"				
1 m/220°C + RA	2	25.1		37.5
15 m/180°C + RA	2	26.8		36.5
"intermediate time"				
5 m/220°C	2	36.5		39.3
5 m/220°C + RA	2	30.9		40.4
50 m/180°C + RA	2	27.6		38.3
"long time"				
8 h/160°C + RA	2	26.9		38.2
8 h/180°C + RA	2	47.6		42.4

Figure 1. The Grumman CS2F Tracker Aircraft, used in Canada for coastal surveillance.

Figure 2. a) Typical Tracker wing-lock attachment. Note stress corrosion cracks perpendicular to short-transverse direction.
b) Profile of stress corrosion cracking in wing-lock fitting shown in a.

Figure 3. Microstructure of the Tracker 7075-T6 plate wing-lock attachment fittings. Magnification X80, etched in Keller's Reagent.

Figure 4. Fracture surface of Tracker wing-lock attachment fitting. Arrows enclose stress corrosion corroded area. Magnification approximately X6.

Figure 5. Standard compact-tension specimen for fatigue crack growth rate testing. Shown approximately actual size.

POOR COPY / /
COPIE DE QUALITÉ INFERIEURE

Figure 6. Schematic plot of a typical fatigue crack growth rate versus stress intensity plot for high strength materials, showing the three stages of crack growth.

Figure 7. Diagrammatic representation of plane strain and plane stress crack growth in a compact-tension specimen.

Figure 8. Schematic showing the effects of corrosive environments to the position of a fatigue crack growth rate versus stress intensity curve.

Figure 9. Fatigue crack growth rate versus stress intensity range curve for 7075-T6 and T73 sheet in air and salt environments. Note displacement of curves to lower stress intensities and higher crack growth rates. Stress ratio, 0.1; frequency, 2.5 Hz. (from Wanhill, ref. 12).

Figure 10. Fractograph showing various fatigue striation morphologies for 7075-T6. Note transition from brittle striations in air, to no striations in vacuum, to ductile striations in air. Frequency; 15 Hz. (from Wankill, ref. 16).

Figure 11. Time-to-failure stress corrosion curve for 7075-T6 plate, loaded in the short-transverse direction, alternately immersed in 3.5% NaCl. Stress corrosion threshold (σ_{SCC}) of approximately 50 MPa is evident. (from Hyatt and Speidel, ref. 17).

Figure 12. Schematic representation of a typical stress corrosion crack growth rate versus stress intensity plot for high strength aluminum alloys.

Figure 13. The effect of aging time or higher aging temperatures on the strength and stress corrosion resistance of 7075.

Figure 14. Computation procedure to obtain total crack growth. (a) (top left) fatigue stress intensity spectrum, (b) (top right) crack growth rate under sustained load in aggressive environment, (c) (bottom left) environmental contribution to crack growth in fatigue, (d) (bottom right) integrated effects of environment and $K_{\max}$ on fatigue crack growth rate. (from Wei and Landes, ref. 34).

Figure 15. Schematic of fatigue load cycles where (a) stress corrosion can occur throughout the whole load cycle, (b) stress corrosion occurs during a portion of the load cycle. (from Gerberich et. al., ref. 35).

Figure 16. Schematic comparison of the crack growth curves for the three models for environmentally enhanced fatigue crack growth.

Figure 17. The effect of frequency on stress corrosion fatigue. The transition from true corrosion fatigue to stress corrosion fatigue occurs at $6 \text{ MPa}\sqrt{\text{m}}$, stress ratio, 0.5, steel 835M30. (from Austen and Walker, ref. 39).

Figure 18. Results for Ti-8Al-1Mo-1V showing similar effects as figure 17. Transition from true corrosion fatigue to stress corrosion fatigue occurs at 18.7 $\text{MPa}\sqrt{\text{m}}$. (from Meyn, ref. 46).

Figure 19. a) Results similar to those of figures 17 and 18 for an aluminum alloy (Al-Cu-Mg) under square wave loads.
 b) Results for the same material as (a) indicating a strong frequency effect at low stress intensities. (from Proctor and Moss, ref. 47).

Figure 20. Electron micrograph of Ti-8Al-1Mo-1V, showing cleavage typical of cracking at 0.5 Hz in 3.5% NaCl solution at stress intensities above K_{Isc} . Arrow indicates direction of crack growth. (from Meyn, ref. 46).

POOR COPY
COPIE DE QUALITÉ INFERIEURE

Figure 21. Electron micrograph of Ti-8Al-1Mo-1V at stress intensities below K_{Isc} in 3.5% NaCl tested at 30 Hz. Note fatigue striations in upper left corner. Arrow indicates direction of crack growth. (from Meyn, ref. 46).

POOR COPY / /
COPIE DE QUALITEE INFERIEURE

Figure 22. Corrosion fatigue crack growth rates of 12Ni-5Cr-3Mo steel tested in 3% NaCl under various load profiles. Triangular and positive sawtooth waves have the same load rise times. (from Barsom, ref. 14).

Figure 23. The effect of rise time on fatigue crack growth rates for A533B-1 steel tested at ΔK of 50 MPa $\sqrt{m}$ and a stress ratio of 0.05. (from Atkinson and Lindley, ref., 36).

Figure 24. Effect of strain rate on susceptibility to stress corrosion cracking. (from Parkins, ref. 49).

Figure 25. The effect of exposing Al-6%Zn-3%Mg to water vapour saturated air at 70°C for 72 hours after aging at various temperatures for 24 hours. (from Chistodoulou and Flower, ref. 58).

Figure 26. A model for stress corrosion crack growth:

a) Initial slow crack growth as hydrogen diffuses into a bubble; very little hydrogen in solid solution.

T73 Condition

b) Crack reaches first bubble and hydrogen starts to diffuse to next bubble.

c) Slow crack growth, since there is little hydrogen in solid solution.

T6 Condition

d) Crack reaches first bubble, hydrogen goes into solid solution ahead of crack tip.

e) Fast crack growth until hydrogen starts to diffuse to next large precipitate.

f) Slow crack growth since hydrogen diffusing to next large precipitate and very little hydrogen in solid solution.

Figure 27. Schematic representation of the changes in hardness during retrogression and reaging. The optimum treatment, according to Cina⁵⁹, is shown by the dotted line, that is retrogression to the minimum hardness plus reaging.

Figure 28. Hardness results of retrogressed 7075-T6 sheet obtained by Cina⁵⁹. Note very short times to minimum hardness.

Figure 29. Typical stress corrosion crack growth rate curve. Arrows indicate the effect of overaging on the curve position. Data points from one double cantilever beam test of 7075-T6 Tracker wing-lock fitting.

Figure 30. Geometry of tensile specimens used for this thesis. Specimens machined and tested in accordance with ASTM E8⁶¹. (Dimensions in inches).

Figure 31. Geometry of double cantilever beam stress corrosion samples machined from 2.5 cm (1") 7075 plate. (Dimensions in inches). Photo taken after approximately 500 hours of testing and a crack of about 2 cm long is visible.

Figure 32. Microstructure of 2.5 cm plate (7075-T651) used in this thesis. Magnification X200, etched in Keller's Reagent.

Figure 33. Schematic showing the set-up used to determine the heat-up times to the retrogression temperatures of 180°C and 220°C of double cantilever beam midsections. The time for the centre of the DCB sample (where the thermometer bulb is placed) to reach temperature was recorded.

Figure 34. Plot showing the decrease in stress intensity with increasing crack length for a double cantilever beam specimen.

Figure 35. Fracture surface of a broken open double cantilever beam stress corrosion sample. Open arrows indicate the extent of the pop-in crack and solid arrows the extent of the stress corrosion crack. Approximately actual size.

POOR COPY
COPIE DE QUALITEE INFERIEURE

Figure 36. Plot showing the effect of correcting stress corrosion crack growth rate results for crack tunnelling. * show results corrected for crack tunnelling, + show results uncorrected for crack tunnelling.

Figure 37. Photo showing 1 mm grid lines photoetched onto a double cantilever beam specimen.

Figure 38. Tensile yield strength of retrogressed 7075-T6 for retrogression times up to 60 minutes.

Figure 39. Yield strength of 7075-T6 retrogressed at 200°C for up to 5 hours and reaged.

Figure 40. Electrical-conductivity of 7075-T6 retrogressed at 180°C for up to one hour and reaged.

Figure 41. Electrical conductivity of 7075-T6 retrogressed at 160°C, 180°C, 200°C, and 220°C for up to one hour.

Figure 42. The relative positions of short, intermediate and long time retrogression and reaging heat treatments, on the hardness or yield strength retrogression and reaging curves.

Figure 43. Stress corrosion crack growth rate versus stress intensity plots, for "short time" RRA treated double cantilever beam specimens. Numbers in brackets are electrical conductivity values.

Figure 44. Stress corrosion crack growth rate versus stress intensity plots, for "intermediate and long time" RRA treated double cantilever beam specimens. Numbers in brackets are electrical conductivity values.

Figure 45. Plot of fracture toughness of heat treated 7075 plate versus electrical conductivity. Fracture toughness calculated from "pop-in" crack lengths on double cantilever beam specimens.

Figure .46.a) Schematic showing variation of plastic zone size across a compact-tension or double cantilever beam specimen. Plane stress exists at specimen surfaces and plane strain at midsection.
 b) Drawing showing relative sizes of plane strain and plane stress plastic zones.

a)

b)

Figure 47.a) Profile of "pop-in" crack in a 7075-T6 double cantilever beam specimen. Crack tip is predominantly in plane strain and no plastic deformation is visible.

b) Profile of a "pop-in" crack in a double cantilever beam specimen heat treated (T6+8h/180°C+RA) to a high toughness, such that crack tip at specimen surfaces is in a plane strain-plane stress mixed mode stress state. Note evidence of plastic deformation at crack tip.

Figure 48. Electron micrograph of extruded bar 7075-T6 showing,
a) grain boundary oriented 'straight on' to electron beam,
b) approximately the same grain boundary as a, but tilted
with respect to the electron beam.
Magnification, X57,000.

Figure 49. a) Electron micrograph of 7075-T6 extruded bar retrogressed for 5 minutes at 220°C. Magnification, X52,000.
b) Electron micrograph of 7075-T6 extruded bar retrogressed as in a, plus 29 hours at 121°C. Magnification, X64,000.

Figure 50. Electron micrograph showing structure of
7075-T73 extruded bar. Magnification, X64,000.

POOR COPY
COPIE DE QUALITEE INFERIEURE

Figure 51. Two examples of molecular hydrogen along grain boundaries in 7075-T6 extruded bar retrogressed for 5 minutes at 220°C and reaged. Arrow indicates a hydrogen bubble with particularly clear fringes, caused by the elastic strain field around the bubble. Magnification, X115,000.

Figure 52. Elastic strain field fringes along grain boundaries of artificially aged (48 hours at room temperature + 4 hours at 90°C + 4 hours at 150°C) Al-4.4%Zn-1.5%Mg that have been attributed to hydrogen bubbles.
 a) Grain boundary viewed straight on with respect to electron beam.
 b) Grain boundary viewed tilted to electron beam.
 (from Scamans, ref. 57).

APPENDIX A

Double Cantilever Beam Data Reduction

Introduction

The programs described in this appendix are designed to take crack length versus time data from double cantilever beam specimens and produce crack growth rate versus stress intensity plots. For convenience, the data reduction process consists of running three separate programs to perform the following tasks;

- 1) Input and store crack length versus time data,
- 2) Calculate crack growth rate and stress intensity at each valid data point,
- 3) Comparison of crack growth rates obtained from the second program.

The programs are designed to run on a Digital PDP-11/04 computer and are written in MTS Basic-11. However, in order to execute the programs neither a knowledge of the Basic language or the analytical procedure used is required. The next section describes each program sufficiently to allow successful execution, while the last section describes the analytical procedure used in each program.

Summary of Programs.

The first program, called 'IRD' (Input Raw Data), collects all the relevant specimen dimensions and the double cantilever beam time-crack length data. This information is stored for further analysis. All dimensions are input in millimeters and time in hours.

The program can be used in two different ways. Firstly, for input and storage of data for a new specimen, that is, for a specimen not previously analyzed. In this case the specimen dimensions and identification required for analysis is requested. This is followed by the input of the time-crack length measurements (crack lengths for both sides of double cantilever beam specimen), with the first time input being zero hours, or the initial crack length. All the data input is stored on floppy disc. All inputs are preceded by messages explaining the information being requested.

The second role of the program is the updating of previously stored data. In this case the double cantilever beam specimen to be updated is identified, the previously stored data is listed, and then additional data can be input. This second role allows data from a test in progress to be analyzed and then as further test data is accumulated

it can be added to the existing stored data.

The second program, 'SCCDAT', converts the time-crack length data to crack growth rate versus stress intensity results. The only input the program requires is identification of the specimen to be analyzed and whether a copy of the results is required. The program reads all other information from the data stored by program 'IRD'. This program outputs the original data in the form of crack length versus time plots, see figure A1. After the analysis is complete a table giving the time, crack length, crack tip stress intensity and crack growth rate is produced as shown in figure A2. Lastly a plot of the crack growth rate (on a logarithmic scale) is plotted against the crack tip stress intensity, figure A3. This last plot is stored on floppy disc for comparison with other specimens.

The last program of the analysis, 'CAD' (Compare Analyzed Data) can make a comparison of crack growth rate versus stress intensity plot for up to six specimens. The input required is the number of specimens to be compared and the specimen identification. The information for the comparison plot, figure A4, is obtained from the results stored on floppy disc by 'SCCDAT'.

2

Program Details.

The flowcharts and MTS Basic code for the three programs are given in figures A5 to A10. The location of each flowchart step within the relevant program is indicated. By following the flowcharts and accompanying programs the analytical technique used should be readily apparent. However there are some aspects of the program that require further explanation.

The essential part of the analysis is the calculation of the slope of the crack length versus time plot to obtain the crack growth rate. This is accomplished by fitting a second order polynomial to seven successive crack length versus time data points and calculating its derivative to give the crack growth rate. One of the requirements of this technique, is that the increment in crack length between data points should be ten times the error in the measurement of the crack length. The crack length measurement error depends on several factors including, user experience and specimen surface condition. However, an increment in crack length between data points of 0.3 mm was found to be adequate to give satisfactory results. Consequently the data from double cantilever beam specimens is scanned, lines 123-155 of 'SCCDAT', and data eliminated so there is

at least 0.3 mm crack growth between successive data points.

The crack growth rate calculation starts by fitting a second order polynomial to the first seven crack length versus time data points shown in figure A1. The polynomial coefficients are calculated using a least squares criterion, lines 610-680 in 'SCCDAT'. The derivative of the polynomial (or the crack growth rate) is calculated for the fourth data point, lines 690-730 in 'SCCDAT'. The fourth data point is the 'middle' point of the seven points used to obtain the fitting polynomial, and represents the data point at which the polynomial fit has the smallest error. The procedure is then repeated for the second to eighth crack length versus time data points to obtain the crack growth rate at the fifth data point. This process is continued until the crack growth rate has been calculated for each data point. The accuracy of this curve fitting procedure is reflected in the high values of the multiple correlation coefficient (M.C.C.), see figure A2, which is calculated in lines 740-800 of 'SCCDAT'. The multiple correlation coefficient equals one for a perfect fit. The major drawback of this curve fitting technique is that an accurate crack growth rate cannot be calculated for the first or last three crack length versus time data points. This is because these data points can never be the middle points of seven

successive ~~data~~ points. Nevertheless, it is the recommended method for calculation of fatigue crack growth rates in ASTM standard ("Tensile Test Methods for Constant Amplitude Fatigue Crack Growth Rates Above 10^{-8} m/cycle").

Figure A1. Example of crack length versus time plot, from program 'SCCDAT'.

SCCOCB - 15M/180+RA 21 T.HRS A.MM	K	DA/DT	FIT (MCC)
0	33.5353	0466586	992885
6	33.5353	0358474	994612
1.2	32.318	0281215	994037
23.3333	28.526	0288527	991816
59.3	23.711	0314212	990965
96.925	23.1245	0335108	997288
148.5	21.2015	0459592	992793
168.267	20.5641	0451905	995167
191.8	19.3656	0471439	995869
262	15.9594	0499053	996439
286	14.1275	0448462	996873
314	12.885	0400627	994951
336	12.4104	0329259	996384
430	9.58031	0310085	977378
441	9.15174	0246936	989151
465	8.71227	0227046	992757
481	8.55613	0199156	988375
502	8.30082	0193043	986745
558	7.93766	0183902	985114
601	7.43667	0145801	995124
622	7.24579	0135963	994967
659.5	7.04558	0122155	994933
729	6.80551		
766	6.57646		
871.75	6.15591		

Figure A2. Example of the tabulated results from program 'SCCDAT'.

SCDCB - 15M/180+RA 21 T, HRS	A, MM	K	DA/DT	FIT
986	37.113	5	0107092	994608
1006	37.617	5	0103272	994992
1078	38.1835	5	9.92803E-03	993933
1126	38.7465	5	9.87871E-03	994686
1174	39.0585	5	9.10196E-03	997222
1257	39.97	5	8.84565E-03	998131
1381	40.9847	5	7.84002E-03	998171
1492	41.68	4	6.95237E-03	998493
1584	42.436	4	6.70302E-03	995227
1656	42.8015	4	6.27539E-03	993801
1776	43.388	4	5.87144E-03	994027
1884	44.3445	4	5.43310E-03	992785
2052	44.9688	4	5.33883E-03	993285
2184	45.547	4	5.65775E-03	990753
2304	46.3065	4		
2496	47.361	3		
2648	48.704	3		

Figure A2. (continued)

Figure A3. Example plot of stress corrosion crack growth rate versus stress intensity, from program 'SCCDAT'.

Figure A4. Comparison plot of various stress corrosion crack growth rates versus stress intensity, from program 'SCCDAT'.

Figure A5. Flowchart for computer program "IRD" (Input Raw Data).

```

IRD      23-DEC-80  MTS BASIC V01B-02C

80 DIM T(100),A8(100),A9(100)
100 PRINT "DO YOU WANT TO INPUT FROM TIME ZERO OR"
110 PRINT "JUST ADD TO AN EXISTING FILE (TZERO OR ADD)?"
120 INPUT Q$
130 IF Q$="ADD" GO TO 600
140 IF Q$<>"TZERO" GO TO 100
300 PRINT "INPUT SPECIMAN ID (RD#)"
310 INPUT I$
320 PRINT "NO. OF TIME READINGS,COD,HALF SPECIMAN HEIGHT"
330 INPUT N,B,H
340 PRINT "INPUT ANY OTHER ID"
350 INPUT I1$PRINT \PRINT
351 PRINT "INPUT TIME AND CRACK LENGTH DATA POINTS"
360 FOR J=1 TO N
370 INPUT T(J),A8(J),A9(J)
380 NEXT J
390 A1$="DX1."&I$&".DAT"
400 OPEN A1$ FOR OUTPUT AS FILE #1
410 PRINT #1:N","B","H
420 PRINT #1:I1$
430 FOR J=1 TO N
440 PRINT #1:T(J),"A8(J)","A9(J)
450 NEXT J
460 CLOSE #1
470 STOP
600 PRINT "INPUT SPECIMAN FOR WHICH YOU HAVE MORE DATA(RD#)"
610 INPUT I$
630 A1$="DX1."&I$&".DAT"
640 OPEN A1$ FOR INPUT AS FILE #2
650 INPUT #2:N,B,H:INPUT #2:S$
655 PRINT "PREVIOUS NO. OF DATA POINTS "N

```

Figure A6. Listing of code for program 'IRD'.

```

636 PRINT TAB(2), "T, HRS", TAB(17), "A, MM"
660 FOR I=1 TO N
670 INPUT #2, T(I), A8(I), A9(I)
680 PRINT T(I), A8(I), A9(I)
690 NEXT I
695 PRINT "INPUT NO. OF ADDITIONAL DATA POINTS "\INPUT D
700 N1=N+D
710 C=N+1
720 PRINT "INPUT ADDITIONAL DATA"
721 FOR K=C TO N1
730 INPUT T(K), A8(K), A9(K)
740 NEXT K
760 PRINT "NEW DATA POINT TOTAL ", N1
770 OPEN A$ FOR OUTPUT AS FILE #3
780 PRINT #3, N1, "B", "H"
790 PRINT #3, S$
795 PRINT TAB(2), "T, HRS", TAB(17), "A, MM"
800 FOR I=1 TO N1
810 PRINT T(I), A8(I), A9(I)
820 PRINT #3, T(I), "A8(I)", "A9(I)"
830 NEXT I
840 CLOSE #3
850 STOP

READY

```

Figure A6. (continued).

Figure A7. Flowchart for computer program "SCCDAT".

Figure A7. (continued)

Figure A7. (continued).

Q\$ - specimen number
 N - number of data points
 B - load line displacement (mm)
 H - $\frac{1}{2}$ height of double cantilever specimen (mm)
 S\$ - specimen ID
 S0\$ - specimen number and heaving
 S1\$ - half height and title
 T() - array for time data
 A8() - array for left side crack length data
 A9() - array for right side crack length data
 A() - average of A8() & A9()
 A1() - scanned crack length array
 T1() - scanned time array
 M1 - crack length in meters
 L - length of scanned array
 C1 - 7 point incremental polynomial scalars
 C2
 X, X1, X2, X3, X4 - polynomial least square parameters
 Y, Y1, Y2, Y3
 B1, B2, B3
 D() - derivative of polynomial (da/dt)
 Y5, Y6
 S1, S2, S3 - parameters for M.C.C.
 D, N1 - steps to get stress intensity
 K1() - stress intensity at each data point

Figure A8. List of principal variables for program "SCCDAT".

```

SCCDAT 29-APR-81  MTS BASIC V01B-02C

60 DIM T(100),A8(100),A9(100),D(100),T1(100),A1(100),A2(8),T2(8),A(100)
61 DIM K1(100)
65 PRINT "INPUT SPECIMAN TO BE ANALYSED (##)"
70 INPUT Q$
71 PRINT "DO YOU WANT HARD COPIES OF RESULTS (YES)?"
72 INPUT C1$
73 Q1$="DX1:RD"&Q$&".DAT"
80 OPEN Q1$ FOR INPUT AS FILE #2
90 INPUT #2:N,B,H:INPUT #2:S$
100 FOR I=1 TO N
110 INPUT #2:T(I),A8(I),A9(I)
120 A(I)=(A8(I)+A9(I))/2
121 NEXT I
123 A1(I)=A(I)*T1(I)=T(I)
125 I1=1
127 I=A(I)+.3
129 FOR P=2 TO N
131 A3=0:T3=0
133 FOR J=1 TO N
135 IF A(J)>I GO TO 137
136 I2=J
137 NEXT J
138 IF I2<I1-1 GO TO 142
139 A1(P)=A(I1)*T1(P)=T(I1)
140 I1=I1+1
141 GO TO 151
142 I3=I2-I1+1
143 FOR K=11 TO 12
144 A3=A3+A(K)*T3=T3+T(K)
145 NEXT K
147 A1(P)=A3/I3:T1(P)=T3/I3

```

Figure A8. Listing of program 'SCCDAT'.

```

149 I1=I2+1
151 I=AK(I1)+3
154 IF I2=N GO TO 160
155 NEXT P
160 L=P
163 FOR J=1 TO L
166 PRINT T1(J),A1(J)
167 NEXT J
180 CNTR(5)=CNTR(0)+CNTR(3)
190 PHYL(100,800,55,750)
195 A2=A1(L)+5
200 SCAL(0,0,T1(L),0,A2)
210 AXES(0,0)=AXES(T1(L),A2)
220 LABL("TIME (HOURS)","CRACK LENGTH MM",400,10,1)
230 FOR I=1 TO L
240 MARK("X",T1(I),A1(I))
250 NEXT I
260 S0$="SCDCB - "&S0$ " &Q$
270 COMK S0$,10,3)
280 IF C1$="YES" THEN CNTR(4)
281 CNTR(3)
320 FOR I=1 TO L
325 M1=A1(I)/1000
326 H1=H/1000
330 D=4*((M1+.6*XH1))^3+H1^2*M1)
340 N1=SQRT(3*XH1*(M1+.6*XH1))^2+H1^3)
341 B1=B/1000
350 K1(I)=B1*71.7*10^9*XH1*N1/(D*10^6)
360 NEXT I
380 PRINT \PRINT \PRINT S0$
381 PRINT TAB(2),"T,HR$","TAB(17)","A,MM","TAB(29)","K",
382 PRINT TAB(46),"DA/DT","TAB(60)","FIT (MCC)" \PRINT
410 FOR I=1 TO 3
420 PRINT T(I),A1(I),K1(I)
430 NEXT I

```

Figure A8. (continued).

```

520 N1=L-6
530 Q=0
535 FOR I=1 TO N1
540 M=0
550 Q=Q+1
560 Q1=Q+6
570 FOR J=Q TO Q1
580 M=M+1
590 A2(M)=A1(J)*T2(M)=T1(J)
600 NEXT J
610 C1=.5*(T2(1)+T2(7))/C2=.5*(T2(7)-T2(1))
620 X1=0/X2=0/X3=0/X4=0/Y1=0/Y2=0/Y3=0
630 FOR J=1 TO 7
640 X=(T2(J)-C1)/C2
650 Y=A2(J)
660 X1=X1+X/X2=X2+X^2/X3=X3+X^3/X4=X4+X^4
670 Y1=Y1+Y/Y2=Y2+Y*Y/Y3=Y3+Y*Y^2
680 NEXT J
690 D=7*(X2*X4-X3^2)-X1*(X1*X4-X2*X3)+X2*(X1*X3-X2^2)
700 B1=(Y1*(X2*X4-X3^2)-Y2*(X1*X4-X2*X3)+Y3*(X1*X3-X2^2))/D
710 B2=(7*(Y2*X4-Y3*X3)-X1*(Y1*X4-Y3*X2)+X2*(Y1*X3-Y2*X2))/D
720 B3=(7*(X2*Y3-X3*Y2)-X1*(X1*Y3-X3*Y1)+X2*(X1*Y2-X2*Y1))/D
730 D(1)=B2/C2+2*B3*(T2(4)-C1)/C2^2
740 Y5=Y1/7/S1=0/S2=0
750 FOR J=1 TO 7
760 X=(T2(J)-C1)/C2
770 Y6=B1+B2*X+B3*X^2
780 S1=S1+(A2(J)-Y6)^2/S2=S2+(A2(J)-Y5)^2
790 NEXT J
800 S3=1-S1/S2
810 Q9=I+3
820 IF I=23 THEN GOSUB 3000
850 PRINT T1(Q9),A1(Q9),K1(Q9),D(1),S3
860 NEXT I
870 IF I=20 THEN GOSUB 3000

```

Figure A8. (continued).

```

900 FOR I=Q9+1 TO Q9+3
910 PRINT T1(I),A1(I),K1(I)
920 NEXT I
999 IF C1$="YES" THEN CNTR(4)\GOSUB 3030
1000 CNTR(3)
1010 PHYL(100,700,55,750)
1020 SCAL(1,0,40,1,00000E-04,1)
1030 AXES(0,1,00000E-04)\AXES(40,1)
1040 LABEL("K,MPA.SQRT(M)","DA/DT,MM/HR",5,1,1)
1050 FOR I=1 TO N1
1060 MARK("+",K1(I+3),DX(I))
1070 PLOT(K1(I+3),DX(I))
1080 NEXT I
1085 H$=STR$(H)
1090 S1$="HALF HEIGHT= "&H$&"MM"
1100 PHYL(710,950,55,750)
1110 SCAL(0,40,45,1,100)
1120 COMM(S0$,40,50)\COMM(S1$,40,70)
1125 IF C1$="YES" THEN CNTR(4)
1129 Q1$="DX1:AD"&Q$&" DAT"
1130 OPEN Q1$ FOR OUTPUT AS FILE #5
1140 PRINT #5:N1
1150 FOR I=1 TO N1
1160 PRINT #5:DX(I),"K1(I+3)
1170 NEXT I
1180 CLOSE #5
1190 STOP
3000 IF C1$="YES" THEN CNTR(4)
3001 CNTR(5)\CNTR(3)
3010 PRINT \PRINT \PRINT 80$
3020 PRINT TAB(2),"T,HR$;TAB(17),"A,MM";TAB(29),"R";
3021 PRINT TAB(46),"DA/DT";TAB(60),"FIT"\PRINT
3030 FOR Z=1 TO 800\Z9=SINK Z)\NEXT Z
3040 RETURN
3050 END

```

Figure A8. (continued).

Figure A9. Flowchart for computer program "CAD" (Compare Analyzed Data).


```

CAD. 22-MAY-81 MTS BASIC U01B-02C

10 DIM G(200),K1(200)
20 PRINT "NUMBER OF SETS TO BE COMBINED"\INPUT N
30 PRINT "ENTER SPECIMAN NUMBERS, (AD#), ONE PER LINE"
40 FOR I=1 TO N
50 INPUT A$(I)\NEXT I
60 B$(1)="+"\B$(2)="*\B$(3)="X"\B$(4)=" "\B$(5)="<"\B$(6)=">"
70 CNTR(3)
80 PHYL(100,700,55,750)
90 SCAL(1,0,40,1,00000E-04,1)
100 AXES(0,1,00000E-04)\AXES(40,1)
110 LABEL("K,MPA.SQRT(M)","DA/DT,MM/HR",5,1,1)
120 FOR I=1 TO N
130 C$="DX1:"&A$(I)&".DAT"
140 OPEN C$ FOR INPUT AS FILE #1
150 INPUT #1:M
160 FOR J=1 TO M
170 INPUT #1:G(J),K1(J)
180 MARK(B$(I),K1(J),G(J))
190 NEXT J
200 CLOSE #1
210 NEXT I
220 PHYL(710,950,55,750)
230 SCAL(0,40,45,1,100)
250 FOR I=1 TO N
260 L=I*10
270 A$(I)=A$(I)&" "&B$(I)
280 CONTK A$(I),40,L)
290 NEXT I
300 CNTR(4)
310 STOP

```

Figure A10. Listing of program 'CAD'.

APPENDIX B

Procedure to Photoetch Grids Onto Double Cantilever Beam

Specimens

This appendix outlines the procedure to photoetch the cracking surfaces of double cantilever specimens with the 1 mm spaced grid lines. The procedure had two distinct sections. Firstly, making a negative of the grid pattern desired and secondly application of the grid pattern onto the double cantilever beam specimen.

To obtain a negative of the grid pattern, the pattern was drawn accurately enlarged six times. In this case, then, the 1 mm grid lines could be drawn 6 mm apart. A photograph of this grid was taken such that on the negative, the grid line spacing was 1 mm as shown in figure B1.

To transfer the grid pattern from the negative to the specimen surface the following procedure was developed.

- 1) The surfaces of the double cantilever beam specimens to receive the grid pattern were vapour blasted by the Manufacturing Technology Research Branch of the Division of Mechanical Engineering, National Research Council. Vapour blasting is essentially a mild sand blast, with the particles of sand suspended in a high pressure stream of water. The

vapour blasted surfaces must not be touched as this will leave fingerprints.

- 2) To the cleaned and well dried vapour blasted surfaces a photosensitive coating is applied. The photosensitive coating is a mixture of "Eezy-Kote negative coating solution" and "Eezy-Kote Diazo Powder". Once mixed the solution should be refrigerated for best results. A thin layer of the photosensitive solution can be applied with a small paint brush or a cotton swab. It should be allowed to dry at least 5 minutes.
- 3) The negative of the grid pattern is aligned on the specimen surface. To keep the negative firmly against the specimen a piece of clean glass can be placed on top of the negative.
- 4) The negative is then exposed to a bright flood-lamp placed about 12" from the surface, for about 7 minutes.
- 5) Apply "Eezy-Kote Jet Black Developer" liberally with a paint brush and brush it around until photosensitive layer is removed and grid pattern becomes clearer. The "Jet Black Developer" is then removed by rinsing with water.
- 6) Apply "Eezy-Kote Gum Sealer" with a sponge, allow it to dry and rinse with water. This last step assures that the grid pattern will not fade and also prevents it from marking easily.

Figure B1. Negative used for applying grid pattern to double cantilever beam specimens.

POOR COPY
COPIE DE QUALITEE INFERIEURE

3 3
OF / DE

APPENDIX C

Thin-Foil Preparation

This appendix contains a brief outline of the method used to prepare thin-foils for examination in the transmission electron microscope. In order to fit into the microscope specimen holder the foils should be 3 mm in diameter and 0.18 to 0.25 mm thick, with the center of the foil thinned down so as to be transparent to the electron beam (approximately 1100 Å thick).

The thin-foils for this work were machined from the threaded section of extruded bar tensile samples. The first machining step was to turn the broken tensile specimen threaded end down to a 3 mm diameter. Then using a diamond cut-off wheel disks were cut approximately 0.75 mm thick. Both sides of the disk were then ground using a 3µm diamond particle size until the disk was 0.18 to 0.25 mm thick.

A Struers "Tenupol" electrolytic jet polisher was used to thin down the disk centers. In this device the disk specimen is made the anode in a suitable electrolyte, with jets of electrolyte impinging on the center of both sides of the disk specimen. In order to obtain a good foil consistently, the thinning conditions must be set as follows:

Electrolyte: approximately 1 liter of $\frac{1}{3}$ HNO_3 plus $\frac{2}{3}$ methanol.

Temperature: electrolyte should be -30°C to -15°C ,
(cool using liquid N_2)

Cell Voltage: 6 V D.C.

Cell Current: 70 to 100 mA.

Flow Rate: 5.5 - 7.0 on "Tenupol" scale.

Photocell: light source set to maximum intensity,
and photocell sensitivity knob set to
about 10 o'clock position.

Time: depending on foil thickness; nominally
5 min.

APPENDIX D

Tensile Test Results

i) Tracker Wing-Lock Rittings

Material Condition	0.2% Yield Stress (MPa)	Ultimate Stress (MPa)	Electrical Conductivity (% IACS)
T6	511	585	33.0
15 m/180°C + RA	509	573	35.3
1 m/220°C + RA	495	565	34.6
T73	426	501	40.6

ii) 7075-T6 Extruded Bar Retrogressed at 160°C.

Retrogression time	Retrogressed Only			Retrogressed and Reaged		
	0.2% Yield Stress (MPa)	Ultimate Stress (MPa)	% Elongated	0.2% Yield Stress (MPa)	Ultimate Stress (MPa)	% Elongated
20 sec	509	592	8.8			
60	488	577	9.6	504	575	9.3
90	487	573	9.1	498	571	9.4
2 min	492	577	10.1	495	574	9.1
4	491	580	9.8	515	587	8.9
6	473	562	9.2	502	576	9.0
10	455	546	9.9	509	582	9.5
20	458	550	9.3	508	580	9.7
30	469	540	9.7	511	581	9.3
1 hr	472	553	9.1	508	571	10.2
8	477	551	10.1	501	568	9.6

iii) 7075-T6 Extruded Bar Retrogressed at 180°C.

Retrogression time	Retrogressed Only			Retrogressed and Reaged.		
	0.2 % Yield Stress (MPa)	Ultimate Stress (MPa)	% Elongated	0.2% Yield Stress (MPa)	Ultimate Stress (MPa)	% Elongated
30 sec	489	571	9.2	510	579	9.5
60	478	568	9.6	494	570	9.1
90	468	558	9.1	507	581	9.3
2 min	468	557	10.0	507	574	9.3
4	458	553	9.4	501	571	9.7
6	461	557	9.8	512	580	9.2
10	456	547	9.3	523	584	8.9
20	459	549	9.5			
30				522	578	9.7
50	480	549	9.2			
1 hr	482	553	9.1	521	575	9.9
8	462	536	9.7	470	538	9.9

iv) 7075 Extruded Bar Retrogressed at 200°C.

30 sec				519	588	9.1
60				504	573	9.4
90				519	583	9.8
2 min	494	575	8.7	508	578	9.7
3	479	567	9.3	511	580	9.8
4	466	555	9.2	518	585	10.0
5	427	529	9.6	509	575	9.1
10	456	541	9.5	523	580	9.3
20	479	548	9.1	519	573	9.6
30	484	546	9.2	520	572	9.6
40	482	545	9.7	514	568	9.4
50	471	537	10.2	506	561	9.3
1 hr	468	537	9.0	498	554	9.8
2	479	543	9.8	511	565	9.6
3	439	521	9.4	465	537	9.7
4	437	459	9.7	460	532	9.9
5	421	515	9.6	430	521	10.0

v) 7075 Extruded Bar Retrogressed at 220°C.

10 sec	446	556	9.0	505	579	8.6
15	452	549	9.7	515	584	9.4
20				501	572	9.2
30	439	540	9.6	514	578	9.7
40	430	534	9.2			
1 min	423	528	9.9	498	570	9.0
5	440	525	9.5	513	571	9.3
30	388	491	10.0	419	510	9.8
1 hr	360	464	9.2	384	482	9.6

APPENDIX E

C-Ring Stress Corrosion Testing and Results

Introduction

This appendix describes C-ring stress corrosion tests that have been performed as a supplement to the work outlined in chapters 5 through 8. The purpose of the C-ring tests was to establish whether during RRA treatments, the stress corrosion resistance of the extruded bar material increases, as was demonstrated for the plate material by the double cantilever beam tests.

C-Ring Test Procedure

C-ring specimens were used for stress corrosion testing of the 1.9 cm (0.75") diameter extruded bar. Using C-ring samples the extruded bar can be loaded in the transverse direction, see figure E1, which is the most stress corrosion susceptible.

C-ring specimens were machined from the extruded bar stock, as shown in figure E1, allowing the specimens to be loaded in the transverse direction. The C-ring dimensions, shown in figure E1, are in accordance with ASTM standard

G38-73¹. Heat treatments were applied in a silicone oil bath before the loading bolt and 60° slot (shown in figure E1) were machined. The loading bolt was tightened so that the stress in the C-ring was 75% of the yield strength. The change in the C-ring outside diameter, induced by the loading bolt, to give the required stress was calculated from the following equation¹:

$$\Delta = \frac{f D^2}{4 E t Z} \quad \dots (E1)$$

where: Δ - change in outside diameter,
 f - desired stress,
 t - C-ring wall thickness,
 D - mean diameter (outside diameter - t),
 E - elastic modulus,
 Z - correction factor for curved beam.
 (given in ref. 1)

The C-ring samples were alternately immersed in a standard seawater solution², as outlined in ASTM standard G44-75³. The alternate immersion cycle consisted of the normal 10 minute immersion in seawater, followed by 50 minutes in laboratory air. This alternate immersion cycle was continued 24 hours a day, by means of a platform, with the specimens attached, being raised and lowered on a guide by a small electric motor. The seawater solution was at the

bottom of the guide in a tray, in which the platform was submerged for 10 minutes of each cycle.

For aluminum alloy 7075 C-ring tests are often run for 30 days, at which time the specimens "pass or fail" the test, based on whether a stress corrosion crack is visible. Alternately, the specimens can be visually examined periodically and the time required for a stress corrosion crack to form recorded.

Six heat treatment conditions were C-ring stress corrosion tested. For each of the six treatments, six samples were tested, giving a total of 36 samples. The six heat treatments tested were the T6 and T73 conditions and the four retrogressed and reaged conditions given in Table E1. The RRA treatments were selected so that a wide range of electrical conductivities would be represented.

C-Ring Results

The results of the C-ring stress corrosion tests are summarized in Table E1. The results are presented as the number of samples of each heat treated condition with stress corrosion cracks after 7, 10, 14 and 30 days testing. The appearance of four of the C-ring samples after 24 days of testing is shown in figure E2.

The C-ring stress corrosion results exhibit similar trends as the double cantilever beam tests of the plate material. "Short time" retrogression and reaging treatments resulted in only marginally better stress corrosion resistance compared to the T6 temper. However, "intermediate and long time" RRA treatments have stress corrosion resistance that compares favourably with the T73 condition. The C-ring stress corrosion tests verify the trend of increasing electrical conductivity with stress corrosion resistance.

The C-ring results of the "intermediate time" treatment of 5 minutes at 220°C plus reaging is of particular interest. It should be recalled that the tensile strength of extruded bar in this condition was in excess of the "as received" T6 strength. From Table E1 it is evident that the stress corrosion resistance of this RRA condition is only slightly less than that of the T73 condition.

The C-ring tests indicate that RRA treatments can improve stress corrosion resistance of 7075 extruded bar. As well, stress corrosion resistance of 7075 is proportional to electrical conductivity. Similarly to the plate material, The RRA treatment of 5 minutes at 220°C plus reaging results in stress corrosion resistance similar to that of the T73 temper, without sacrificing T6 tensile strength.

References

1. ASTM G38-73; "Standard Recommended Practice for Making and Using C-Ring Stress Corrosion Test Specimens",/ ASTM Book of Standards 1980, Part 10. p. 913.
2. ASTM D1141-75; "Standard Specification for Substitute Ocean Water", ASTM Book of Standards, 1980, Part 10. p. 1044.
3. ASTM G44-75; "Standard Recommended Practice for Alternate Immersion Stress Corrosion Testing in 3.5% Sodium Chloride Solution", ASTM Book of Standards, 1980, Part 10, p. 951.
4. United States Military Specification; "Heat Treatment of Aluminum Alloys", MIL-H-6088E, Feb. 5, 1971.

TABLE E1

C-Ring Alternate Immersion Stress
Corrosion Test Results

Material Condition	Electrical Conductivity % IACS	Number of Samples Cracked (maximum 6) after:			
		7 days	10 days	14 days	30 days
T6	35.5	5	6	6	6
T73	42.4	0	0	0	0
"short time" 15/180°C + RA	36.9	6	6	6	6
1m/220°C + RA	37.6	5	5	6	6
"intermediate time" 5m/220°C +RA	40.1	1	1	1	1
"long time" 8h/180°C + RA	44.6	0	0	1	1

Figure E1. a) Orientation of C-ring samples with respect to extruded bar stock.
 b) Geometry of C-ring samples used O.D. (outside diameter) = 0.75".

Figure E2. Appearance of C-ring stress corrosion samples after 24 days of testing.

- a) T6 condition, arrows indicate crack,
- b) T73 condition,
- c) 1 minute at 220°C + reage, arrow indicates crack which is partially obscured by corrosion products,
- d) 5 minutes at 220°C + reage.

Samples shown slightly enlarged.

END

3 0 H 0 8 H 8 2

FIN