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The effects of ionized gas exposure on the toughness and fatigue properties of aluminum alloys and composites

Zaat, Stephen Vincent, Ph.D.

Case Western Reserve University, 1992

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THE EFFECTS OF IONIZED GAS EXPOSURE ON
THE TOUGHNESS
AND
FATIGUE PROPERTIES OF ALUMINUM ALLOYS AND COMPOSITES

by

MAJOR STEPHEN VINCENT ZAAT

Submitted in partial fulfillment of the requirements
for the Degree of Doctor of Philosophy

Thesis Advisor: Professor John F. Wallace

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January 1992
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GRADUATE STUDIES

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THE EFFECTS OF IONIZED GAS EXPOSURE ON THE TOUGHNESS AND FATIGUE PROPERTIES OF ALUMINUM ALLOYS AND COMPOSITES

Abstract

by

MAJOR STEPHEN VINCENT ZAAT

The effects of an oxygen ionized gas from simulated space exposure on the toughness and fatigue properties of several aerospace aluminum alloys and aluminum composites have been analyzed. The test matrix consisted of four aluminum systems: the 6000 and 1100 series and two 8090 aluminum-lithium alloys. The test specimens were prepared as Charpy V-notched impact and disk-shaped compact fracture toughness specimens. A small specimen size is used for the compact tension specimens to facilitate exposure in a Radio Frequency (RF) Plasma Prep II unit. Radio frequency plasma, sometimes referred to as the “electrodeless plasma”, is used in disassociative ionization of molecular oxygen to simulate high fluence, relatively low energy, low earth orbit (LEO) space atomic oxygen. The plasma reacts with the aluminum alloy systems and forms a thin scale of alumina a few microns thick. Monolayer sensitive Variable Angle Spectroscopic Ellipsometry (VASE) is used to determine the growth characteristics and effect on optical constants, the reflective index, n and absorption coefficient, k.
Atomic Force Microscopy (AFM) was also employed to determine the effect of short exposure to the oxygen plasma environment. AFM indicates that sharp spikes of oxygen rich material are produced above the aluminum specimen surface resulting in stress concentrations with gradual roughing of the surface. After 10 years of simulated exposure, the increased roughness results in a 34% increase in the fatigue crack growth, \( \frac{da}{dN} \), for the 6000 series; The crack growth rate of the aluminum-lithium material was increased about 28%. The results of instrumented Charpy V-notched impact specimens indicated that exposure reduced the energy for fracture initiation by 29%. Plane strain fracture toughness effects could not be established because the influence of oxygen was concentrated at the surface of the specimens.
DEDICATIONS

To Cindy, my wife, and my sons Zachary, and Andrew, with gratitude for their support and championship.
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CHAPTER 1
INTRODUCTION

Commercialization of Space

In 1987, the National Aeronautics and Space Administration (NASA) completed the creation of several centers for the commercialization of space. These Centers were developed to draw industry and academia towards a joint role with NASA in capitalizing on the potential of space and the existing space transportation system. Current and previous administrations have invested in a belief that the United States of America can benefit economically through the development of the technological base necessary to support the exploitation of unique space opportunities. This increased involvement in space places a new and unique demand on materials for space structures.

Space explorers and entrepreneurs are currently facing multiple material science challenges, many of which exist because of the brutal environment of space and the high cost related to the energy required to transport our culture’s objects into space. Space investigators continue to demand higher levels of performance from the systems which sustain their presence in space. Logically, the light weight structural materials transported into space in the future must have a known ability to withstand the environment of space for the planned service life.

Many successful spacecraft have been launched, and several are
currently in service with relatively short service periods planned [1]. However, the long-duration performance of a spacecraft can be linked to the resistance of materials to degradation from extended exposure. The technical literature presents little data on the combined influence of the several environmental factors of space, and is not specific on the individual effect of many of these factors. Information on the joint influence of the various features of the space environment and mechanisms by which individual factors degrade materials must be established [2].

Materials to withstand continued space exposure for several decades must be certified as stable or suitable in the space environment. Environmental factors of major concern are solar radiation degradation caused by ultraviolet (UV) and thermal cycling [3], atomic oxygen [4], micrometeoroids and space debris [5], space vacuum [6], contamination [7], and particulate radiation [8].

The primary objective of the present work is to establish the susceptibility of an evolving aluminum alloy system containing lithium to atomic oxygen attack and characterize the effect of atomic oxygen exposure on the material's engineering properties.

**Atomic Oxygen Environment of Space**

A serious problem for longevity of spacecraft in low earth orbit (LEO) has been identified as recession of organic materials resulting in mass loss and degradation of sensitive, thin-film optical coatings, organic
matrix composites and oxidation of some metals, notably silver and osmium, by atomic oxygen (AO) [9]. Even considering the fact that atomic oxygen effects on materials has been the topic of much research in recent years, very little information is available on the long term exposure of aluminum alloys. This shortcoming in the literature can be attributed to the manner in which the finding of experiments are being reported and the presence of deferring mechanisms. Materials are considered susceptible if there is a physically measurable mass loss. Materials which gain some mass, as aluminum alloys, or have no mass change are assumed to have limited or no susceptibility [10].

Given a nominal level of solar activity, atomic oxygen (AO) is the primary element in the atmosphere at an altitude of 180 km (97 nautical miles or nmi) to 640 km (345 nmi). Figure 1 illustrates the nominal low earth orbital atmosphere [11]. Formed by the dissociation of molecular oxygen by ultraviolet (100 to 200 nm) radiation, the atoms are a major repository of solar energy in the region of the atmosphere throughout which low earth orbit satellites operate and space vehicles pass. The oxygen atoms that result from the dissociation of oxygen molecules by solar UV (energies of greater than 5.115 eV), stay disassociated or collide with other atoms to reform molecular oxygen, ozone or other gas molecular species. As the altitude above the earth’s surface increases, gas density is lower than very near the earth and the disassociated molecular oxygen will tend to remain in a neutral AO atom state. The number density of AO per unit volume is therefore affected by altitude and level of solar activity. A typical density of atomic oxygen at 300 km altitude, 30
degree latitude and with mean annual and mean solar-cycle conditions is $N = 10^9$ atoms per cm$^3$ [12]. The flux of AO presented to an exposed surface is equal to $j = N \times v$, where $v = 8\times10^6$ km per sec., (ie. the velocity typically required to maintain orbit at 300 km), yielding an approximate flux of $8\times10^{14}$ atoms per cm$^2$ per sec. This atom flux over an annual period of space service produces a typical fluence in the order of $2.5\times10^{22}$ atoms per cm$^2$(10), ultimately yielding a total fluence of AO encountered over a 30-year life-cycle of approximately $7.6\times10^{23}$ atoms per cm$^2$ [9]. More detailed explanation of AO fluence calculations and models based on previous direct measurements are available [13-21] and were reported previously [9].

Recent Reported Effects of AO on Aluminum

A recent study by Variable Angle Spectroscopic Ellipsometry (VASE) on space exposed and unexposed (shielded) aluminum reports the presence of an $\text{Al}_2\text{O}_3$ film much thicker than would be expected for the natural oxide film. The high purity aluminum was mounted with a negative angle of incidence to the velocity vector on NASA's Long Duration Exposure Facility (LDEF) and exposed to a leeward stream of atomic oxygen plasma for five years. The measured thickness reported for the exposed and shielded samples was 400 nm and 60 nm, respectively [22]. Anodizing of aluminum in an oxygen plasma for the purpose of growing an aluminum oxide gate insulator has been practiced by the electronic material industry for some time [23]. The basic principles of
plasma anodizing were also applied and verified using ellipsometry, in work conducted to determine oxide thickness as a function of plasma ashing [24]. Figure 2 shows $\text{Al}_2\text{O}_3$ oxide thickness vs ashing time. Plasma anodizing mechanisms constitute a considerable portion of this dissertation and will be elaborated on in more detail in Chapter 3, “Experimental Program.”

**Lithium Containing Aluminum Alloy Aerospace Structures**

Low density lithium containing aluminum alloys are currently of considerable interest to the aerospace industry owing to cost benefits from potential 12% weight savings [25-29]. Despite early limitations of poor ductility and toughness properties, recent developments in alloying and processing of such alloys show strength-toughness combinations comparable, and in many instances superior, to traditional high strength Al-Cu and Al-Zn-Mg alloys [30].

Commercial Al-Li alloys are presently available in sheets, plates, extrusions, and forgings, and have been designed specifically for five primary categories: high strength; damage tolerant; stress corrosion cracking resistance; minimum density; and super plasticity. Prominent among these alloys are the IM (ingot metallurgy) Al-Li-Cu-Zr and Al-Li-Cu-Mg-Zr systems commercially registered as 2090, 8090, 8091 and 2091; alloys 2090 and 8091 are planned as high strength replacements for series 7000 alloys, whereas 8090 and 2091 are planned for medium strength and damage tolerant applications replacing series 2000 alloys [31].
Although the role of microstructure in influencing the strength, toughness, and fatigue crack growth properties of experimental Al-Li alloys is widely reported [25-32], insufficient data exists on the performance of these alloys in actual or simulated space service. Moreover, no data has been presented on atomic oxygen susceptibility and effects of space exposure. Since the commercial alloys show highly elongated and primarily unrecrystallized grain structure, due to zirconium additions, gamma prime $\text{Al}_3\text{Li}$ strengthening precipitates and extensive thermomechanical processing [32], the mechanical properties of such materials are strongly anisotropic and can be expected to be affected differently. Of these materials, the 8090 series is evaluated in this work because of high lithium weight percent, and proportionally lower copper and magnesium weight percent.

A plate of 8090-T8771 was obtained from Alcan and has been fabricated for use as the primary support structure of LDCE-1, the Case Western Reserve University space shuttle / space materials experiment. This work completes the qualification process for the original experiment and establishes the criteria for continued use of the the support disk after exposure to atomic oxygen of space. Engineering fracture mechanics principles shall be employed to quantify the effect of exposure.
CHAPTER 2
LITERATURE REVIEW

Aluminum

Aluminum is a strongly electronegative metal that exhibits a strong affinity for oxygen; this is apparent from the high heat of formation of its oxide. Although it is among the six most widely distributed metals, it was not isolated until well into the nineteenth century. The three main properties on which the application of aluminum is based are low density, high mechanical strength achieved by suitable alloying and heat treatment, and relatively high corrosion resistance. Other valuable properties include high thermal and electrical conductance, reflectivity, high ductility and resultant low working cost, magnetic neutrality, high scrap value, and the non-poisonous and colorless nature of its corrosion products.

The metal in its pure state has a relatively high corrosion resistance and needs less protection than most metals. On the other hand, the commercial metal and its alloys, though resistant, are distinctly more sensitive to corrosion, and the development of high-strength light alloys, containing quantities of copper, zinc or nickel, has heightened the need for protective surface treatments. The heavy metal additions influence the alloy’s susceptibility to corrosion, and combinations of high
mechanical strength and good corrosion resistance have so far proved largely incompatible. The development of satisfactory protective finishes for the metals has been, therefore, of very great importance.

Brief consideration is now given to the properties of the various aluminum alloys which are available in relation both to their corrosion resistance and applications.

**Alloy Development**

The chief alloying constituents added to aluminum are copper, magnesium, silicon, manganese, nickel and zinc. All of these are used to increase the strength of pure aluminum.

Two classes of alloys may be considered. The first are the ‘cast alloys’ which are cast directly into their desired forms by one of three methods (i.e., sandcasting, gravity die casting or pressure die casting), The second class, the ‘wrought alloys’, are cast in ingots or billets and hot and cold worked mechanically into extrusions, forgings, sheet, foil, tube and wire. The main classes of alloys are: the 2000 series (Al-Cu alloys), high-strength materials used mainly in the aircraft industry; the 3000 series (Al-Mn alloys), used mainly in the canning industry; the 5000 series (Al-Mg alloys), used unprotected for structural and architectural applications; the 6000 series (Al-Mg-Si alloys), the most common extrusion alloys, used particularly in the building industry; and 7000 series (Al-Zn-Mg alloys), high-strength alloys for aircraft and military vehicle applications.
Aluminum Oxide Film

When a freshly formed aluminum metal surface is exposed to the atmosphere, it is immediately covered with a thin film of oxide, and this oxide film quickly re-forms when damaged. An important and beneficial feature of this oxide film is that its molecular volume is stoichiometrically 1.5 times that of the metal used in oxidation. In this case, the oxide film is under compressive stress, and will not only cover the metal continuously, but can cope with a certain amount of substrate deformation without rupturing.

Reports of the structure of this low temperature, air-formed film vary widely although, in general, it is assumed to be amorphous, with the outer surface being a hydrated aluminum oxide. At higher temperatures (above 450°C), crystalline gamma-Al$_2$O$_3$ is formed, and then, in the molten state, alpha-Al$_2$O$_3$ can occur. However, in space the oxidation of aluminum and its alloys can be considered a low-temperature phenomena.

The kinetics of oxide growth on pure aluminum are complex. The currently accepted mechanism were developed by Wefers [33]. At ambient temperatures a limiting oxide film of 2 to 3 nm will be produced within one day. Thermal oxidation is controlled by diffusion of aluminum and oxygen ions at temperatures up to ~ 400°C. In this temperature range, asymptotically decaying rate laws are observed. As the temperature is
raised above 450°C, the exponential oxidation rate changes to a linear relationship between weight gain and time. This change in mechanism represents crystallization to gamma-Al$_2$O$_3$, which will disrupt the continuity of the film. At temperatures above 500°C, it has been reported [34] that the preparation of the sheet, i.e. both metallurgical and surface roughness features, can alter the oxidation kinetics.

The major alloying element incorporated within the oxide film for aluminum alloys is magnesium. At temperatures above 340°C [35], magnesium diffuses from the bulk of the alloy and, even at levels less than 50 ppm magnesium, a competitive oxidation reaction occurs between magnesium diffusion outwards and oxygen diffusion inwards. At higher levels of bulk magnesium (up to 4%), a duplex film of aluminum oxide close to the metal, and an outer layer of MgO, results. These oxide films will thicken until the bulk magnesium level is depleted. Additional information on the oxidation of aluminum-magnesium alloys can be found in the literature [33,36]. Table I indicates the thickness of typical natural oxide coating in comparison with chemical and anodic oxidation coatings.

Breakdown of the oxide film can result from mechanical rupture, or from chemical attack by anions. In most cases repair is instantaneous and depending on the moisture content of the environment, can either be accompanied by the oxygen reduction or the hydrogen evolution reaction shown below:

$$4\text{Al} - 12\text{e}^- \rightarrow 4\text{Al}^{3+}$$  anodic
\[ 3O_2 + 12e^- \rightarrow 6O^2^- \quad \text{cathodic} \]
\[ 2H_2O + 2e^- \rightarrow 2OH^- + H_2 \]

In the first cathodic reaction aluminum oxide results, whereas in the second, hydroxide is formed [38].

Oxide formation rate is known to be dependent on a number of factors such as temperature, time, surface finish of metal substrate, continuous or discontinuous nature of oxide, and internal stress in the oxide. In general, once a continuous oxide layer has become thick enough one of a number of mathematical relationships between oxide thickness and time may be obeyed at a particular temperature [39]. Much work has been reported on the rate and mechanisms of oxide growth as a result of anodizing.

**Anodizing of Aluminum - Conventional Process**

Anodizing of aluminum is well characterized and has been understood for several decades. The first investigations on anodizing were in the 1920's as a method of protection for Duralumin seaplanes. Recent work concentrates on morphological investigations of previous work with contemporary analytical tools.

When a current is passed through an electrolyte in which an aluminum anode is employed, the negatively charged anion migrates to
the anode where it is discharged with a loss of one or more electrons. In an aqueous solution an anion consists in part of oxygen, which unites chemically with aluminum, the result of the reaction depending on the nature of the electrolyte, the constituent reaction products formed, and the operating condition. The result of all oxidation reactions reported, however is the production of a porous film. No attempt shall be made to cite all references on commercial anodized film growth mechanisms. An adequate listing is available [37].

In simple terms, the following oxidation reactions at the anode can occur as a result of the different types of anodizing processes [37]:

1. The reaction products may be almost insoluble in the electrolyte, and form a strongly adherent and practically non-conducting film on the anode. This process constitutes the growth of a barrier film. In this case the film growth continues until the resistance of the film prevents the current from reaching the anode.

2. The reaction products may be sparingly soluble in the electrolyte and form a strongly adherent film, which is non-conducting when dry, over the anode. In this case film growth takes place as in 1, but is accompanied by localized dissolution of the film. Pores are thus formed in the coating which are wide enough to allow continuous access of the current to the metal. Film growth continues, though it is gradually retarded as the film grows thicker and the electrical resistance increases. When the rate of film growth has decreased until it is equal to the rate of dissolution of the film in the electrolyte, the film thickness remains constant. It is also important to realize that the continuing anodizing
reaction is taking place at the Aluminum/Aluminum oxide interface, and therefore that the film is effectively growing from within, rather than the film building up on the outer surface as it does with other surface preparation process.

3. The reaction products may be moderately soluble. Under these conditions, electropolishing may be possible if a suitable electrolyte is used.

4. The anode reaction products may be soluble in the electrolyte. In this case the metal is dissolved until the solution is saturated. This reaction takes place in some strong inorganic acids and bases.

Apart from the reactions considered, there are a variety of less important possibilities, where the reaction products may form loosely adherent, spongy or powdery deposits, as when anodizing solutions become contaminated or when anodizing under special operating conditions. A continuous adherent insoluble film, a few molecules thick, may render the metal passive [9,40]. This appears not to be the case however with pure aluminum in the anodizing plasma environment of low earth orbit space [22] where a nominal concentration of atomic oxygen exists [41].

**Anodizing of Aluminum - Plasma Process**

Aluminum oxide films are reported to have been successfully grown in dry oxygen plasmas since 1963 [42]. The rate of growth (800 to 1300 nm/min) and oxide characteristics have also been investigated [43]. Much
of the recent work in the literature use RF discharges in order to cover an electrode with an electrically insulating material [44].

The $\text{Al}_2\text{O}_3$ films used in most electronics industry investigations today are formed by first depositing aluminum on freshly cleaned p-type silicon wafers [23]. After deposition, the wafer is placed in a contact jig in a vacuum system. Dry oxygen is then admitted to the system and a plasma is ignited between an anode and cathode. With the sample biased positively with respect to the plasma the aluminum film is anodized to $\text{Al}_2\text{O}_3$. Ellipsometry measurements indicate that $\text{SiO}_2$ films formed during plasma anodization are less than 500 nm [23].

Plasma ashers, SPI Plasma Prep II, are currently used extensively to evaluate the atomic oxygen durability of materials. The device uses a typical 13.56 MHz RF discharge current to create a gas plasma of oxygen and nitrogen ions and atoms in various energy states in a glass sample chamber kept at 80-100 mTorr. Figure 3 is a typical Rf discharge plasma ashers schematic used for the purpose of plasma anodizing aluminum.

The intent of the present work is not to establish the suitability of plasma ashers to simulate the space environment. The device is considered generally suitable for plasma anodizing of aluminum [13] and has been successfully used previously [23,24,42-48]. The ideal laboratory test and simulation system would provide a pure, well collimated beam of neutral oxygen atoms with a kinetic energy of 5 eV and an atom flux greater than $10^{14}$ atoms/cm$^2$/sec. No such system exists at this time [45] and detailed mechanisms of plasma anodizing of aluminum have not been reported.
Effect of Anodizing on Aluminum Mechanical Properties

Edge cracks are effective stress raisers and reduce tensile strength and elongation. Therefore, any surface condition or coating that produces edge cracks will influence stress-rupture and fatigue properties of materials. It is reported that anodic coatings have no appreciable effect on tensile properties, but may reduce the fatigue strength as much as 30 percent, depending on factors such as thickness and sealing of coating [49]. Additionally, recent work concentrating on the technological importance of aluminum oxide as a high-temperature protective scale, has produced extensive studies of growth behavior and morphological effects on mechanical properties [50]. Thermal barrier Al₂O₃ coatings were reported to have lower cycles to failure when high compressive stress were produced [51]. The suggested failure mode was that of spalling of the oxide scale.

The effects of anodic treatment upon the tensile properties of the base material were believed to be so small as to be well within the range of variation in testing. In structural parts subjected to cyclic loads, however, the effects on fatigue resistance may sometimes be significant [52]. The tensile properties can be affected if the test specimen is very thin; then the brittleness of the coating may cause some decrease in the tensile strength. On the other hand, contradictory results have been obtained in investigation on the fatigue properties of anodized aluminum alloys [37]. Much of the contradictory results can be attributed to the
ability to measure the presence of the oxide effectively. A very thin oxide is expected to improve fatigue properties [52] whereas a thick anodized coating can cause a considerable reduction in fatigue properties [53]. The reported reduction in fatigue properties has been contradicted recently in a report that indicated an improvement [54]. No literature is available that characterizes the effects on the fatigue properties of plasma-anodized aluminum alloys, or lithium-containing alloys. Even though the general effects on fatigue properties of plasma anodized aluminum alloys is expected to be identical to that of conventionally anodized aluminum alloys, much consideration will be given in this present work to the quantitative measurement of this effect.

Fracture Mechanics and Safe-Life Analysis

Fracture mechanics is the mathematical analysis of the mechanical processes that lead to failure by fracture. It includes the process of crack growth by such mechanisms as fatigue and stress corrosion as well as the final fracture event by cleavage or dimple rupture. The analysis is based on established procedures used generally in solid mechanics [55]. Numerous books have been written on fracture mechanisms and much information is available on the fracture of lithium-containing aluminum alloys [56-70]. Because of the role an anodized coating has on fatigue life and safe-life predictions, sub-critical fracture mechanics (fatigue) analysis is considered for the present work.
The growth of the crack that eventually leads to fracture occurs by mechanisms entirely different from the fracture itself. During most of the cracking process, the crack is much smaller than that which would cause fracture at the prevailing stress. Therefore, the crack-tip stress field is less severe, and the size of the plastic zone smaller than at the time of fracture. Due to this small plastic zone, sub-critical fracture mechanics can often be approached using elastic concepts. If the crack size is close to critical (fracture), this may no longer be true. Since as much as 90 percent of fatigue life is spent in the growth of much smaller noncritical cracks, it is justifiable to use linear elastic fracture mechanics to obtain the time for crack growth with good accuracy. A notable exception is creep crack growth.

In the case of cyclic loading of space service materials, crack growth occurs by fatigue [6,71-76]. The crack growth per loading cycle depends on the crack tip stress field, $K$. During a load cycle, $K$ varies from $K_{\text{min}} = \sigma_{\text{min}} \beta / \pi a$ to $K_{\text{max}} = \sigma_{\text{max}} \beta / \pi a$, over a range of $\Delta K = \Delta \sigma \beta / \pi a$, where $\sigma_{\text{min}}$ is the minimum stress in the cycle, $\sigma_{\text{max}}$ is the maximum stress in the cycle, and $\Delta \sigma$ is the stress range $\Delta \sigma = \sigma_{\text{max}} - \sigma_{\text{min}}$. The amount of growth per cycle depends on $\Delta K$ and $K_{\text{max}}$. Stress ratio $R$ is defined as $R = K_{\text{min}} / K_{\text{max}}$. The amount of growth per cycle of crack growth rate, $\text{da}/\text{dN}$, also depends on $\Delta K$ and $R$. Use of $\Delta K$ and $R$ is often more convenient because $R = K_{\text{min}} / K_{\text{max}} = \sigma_{\text{min}} / \sigma_{\text{max}}$. Hence, in the case of constant-amplitude loading where $\sigma_{\text{min}}$ and $\sigma_{\text{max}}$ do not change from cycle to cycle, $R$ remains constant, while $K_{\text{max}}$ would depend on crack size. Measuring crack growth rate as a function of $\Delta K$ (calculated from $\Delta K = \beta \Delta \sigma / \pi a$)
determines the dependence of crack growth on $\Delta K$, and in a similar fashion, tests at different $R$ ratios provide the dependence on $R$.

Figure 4 shows typical fatigue-crack growth rate test results. Every time the same values of $\Delta K$ and $R$ occur, the material will respond with the same growth rate because the crack-tip stress conditions are the same. Data such as that shown in Figure 4 can be used to predict crack growth in a structure. Cyclic loading of the structure provides $\Delta K = \beta \Delta \sigma / \pi a$ and $R$ for the structure. If the geometric factor $\beta$ is known for the structure and the stress is known, the crack growth rate can be predicted. Crack size as a function of time (or number of cycles) is obtained by integration:

$$N = \int_{a_1}^{a_2} \frac{da}{(da/dn)}$$  \hspace{1cm} (1)

Figure 4 shows that the data for a fixed $R$ value falls on a straight line on a log-log scale. In this case, the rate data can be represented by the Paris equation [76]:

$$\frac{da}{dN} = C \Delta K^m$$  \hspace{1cm} (2)

where $C$ and $m$ are empirical constants.

Unfortunately, the Paris equation covers only one $R$ value. If the line for different $R$ values are parallel, one can use the modified equation:

$$\frac{da}{dN} = (C/(1-R)^n)\Delta K^m$$  \hspace{1cm} (3)
where \( n \) and \( C_0 \) are also empirical constants. By noting that \( R = \frac{K_{\text{min}}}{K_{\text{max}}} \), 
\[ K_{\text{max}} = \frac{(K_{\text{max}} - \Delta K)}{K_{\text{max}}} \text{ so that } K_{\text{max}} = \Delta K/(1-R), \]
equation 3 becomes:

\[
\frac{da}{dN} = C_0 \left( \Delta K^m/(1-R)^n \right) \Delta K^{m-n} \tag{4}
\]

or letting \( p = m - n \)

\[
\frac{da}{dN} = C_0 K_{\text{max}}^n \Delta K^p \tag{5}
\]

Equation 5 [77] is known as the Walker equation. The advantage over the Paris equation is that it covers all values of \( R \).

Many equations are used to model crack growth rates. It should be pointed out, however, that none of them have any physical meaning: all are curve-fitting equations. There is no objection to their use if there is a good fit of the data. However, if the numerical integration must be done by a computer, use of the original data in tabular form is as convenient as use of a sometimes poor fitting equation. NASA currently uses a form of these equations to calculate safe-life for shuttle experiments [78].

The advantages of crack growth analysis are:

1. Can be used to calculate remaining life if cracks of a known or assumed size are present;

2. In many situations, nucleation and steady-state crack growth consumes the majority of the fatigue life.

Limitations are:

1. Stress intensity factors for typical component geometries
are sometimes difficult to obtain;

(2) Notch root plasticity effects are difficult to incorporate into the analysis.

Other methods of fatigue life estimation

The fracture mechanics method of life estimation is only one way of determining theoretical life remaining in a space structure. Other variable amplitude fatigue life estimation models will be reviewed next. Fatigue life estimation techniques were developed through the efforts of many researchers and practicing engineers. No attempt is made to cite individual contributions [79-92].

Three additional approaches to component fatigue life estimations are:

1. Load-Life
2. Stress-Life
3. Strain-Life

Load-Life

The load-life method requires that the constant amplitude fatigue properties of the component and nominal loading history are known. Specifically, a load life curve obtained from component testing and a rainflow counted histogram of the applied loads must be available. Reliable life estimates cannot be achieved from level crossing and other
types of histograms.

Empirical/load data must be converted into appropriate scale
factors. The scale factor incorporates actual load range, maximum load
from history (designed), and range from histogram information. Constant
amplitude load life data for the component may be fit to a simple power
function.

$$\Delta P = P' (2N_r)^m$$  \hspace{1cm} (6)

where $\Delta P =$ load range, $P'$ = intercept at 1 reversal, $m =$ slope and $2N_r =$
reversals to failure.

Fatigue damage for this method of variable amplitude loading
history is calculated from Miner's linear damage rule [55].

$$D_i = 2N/2N_r$$  \hspace{1cm} (7)

where, $D_i =$ fatigue damage for each load, $2N =$ number of reversals at
each load range and $2N_r =$ reversals to failure for each load range.

The total damage for the history is then obtained by summing the
damage for each range in the history. At failure this should equal 1.

The fatigue life calculated by this method represents the number of
times the loads in the histogram can be applied to the component before
failure. Failure is defined to be the same in both variable amplitude and
constant amplitude loading and is arbitrarily chosen. For example,
failure may be defined as the first detectable crack or complete collapse of
the structure. The definition of failure must be consistent in both types of
Advantages of this method are:

1) It is an actual test of the component or structure of interest;

2) Manufacturing effects and local stress concentration effects are automatically included;

3) Stress analysis is not required.

Limitations are:

1) The method cannot be used in the early stages of design before the first prototype is built;

2) A new set of tests must be conducted whenever material or geometry changes are made;

3) Mean stress effects cannot be included.

**Stress-Life**

Stress life analysis requires a stress life curve for the material, stress concentration factor for the most highly stressed region and a rainflow counted histogram of the nominal stress ranges.

Stress life data can be related to the fatigue life projection by the following relationship:

\[ \Delta \sigma/2 = \sigma' (2N)^b \]  \hspace{1cm} (8)

where \( \Delta \sigma/2 \) = stress amplitude, \( \sigma' \) = Intercept at 1 reversal (if the stress life data was obtained from a strain controlled test, this would be the
fatigue strength coefficient), \(2N_r = \) Reversals to Failure and \(b = \) Slope of \(\sigma-2N_r\) curve (if the stress life data was obtained from a strain controlled test, this would be the fatigue strength exponent). Fatigue life for the component is calculated from a notched stress life curve. These curves may be estimated from unnotched smooth specimen data and stress concentration factor using one of two methods.

For both methods, the fatigue strength at \(10^8\) reversals is divided by the stress concentration factor. The notched stress-life curve may then be approximated by a line drawn between this point and either the unnotched fatigue strength at 1 reversal, or the unnotched fatigue strength at \(10^3\) reversals. In both methods, the stress concentration factor \((K_r)\) can be replaced by the fatigue notch factor \((K_f)\), with stress life curves for notched components estimated from the following relationships:

Method I: \[2N_r = (\Delta\sigma/2S_f)^{1/q}\]  \( \text{(9)} \)

where \(q = b - (\log_{10} K_r)/6.0\)

Method II: \[2N_r = (\Delta\sigma/2K_f S_f)^{1/P}\]  \( \text{(10)} \)

where \(P = b - (\log_{10} K_f)/3.0\).

To conduct a stress-life analysis, load ranges in the histogram must be converted into nominal stress ranges. The nominal mean stress correction can only be used when the notch stresses remain elastic, which
is usually not the case in service.

Advantages of this approach are:

1) It can be used for initial design.
2) Changes in material and geometry can be evaluated.

Limitations are:

1) It does not account for notch root plasticity, which is the cause of fatigue.
2) Mean stress effects could be incorrect.
3) It requires empirical $K_I$ factors for good results.

**Strain-Life**

Strain-life analysis accounts for notch root plasticity and requires strain life material properties, nominal loading history and the stress concentration factor of the component of interest. In addition, it requires an analysis relating nominal stresses and strains to the critical stresses and strains at the notch root. If the loading data is given as a rainflow-counted histogram of nominal stresses, a simple analysis similar to load-life can be carried out. Neuber's rule may be employed to estimate notch stresses and strains from nominal stresses and strains [56]:

$$K_I^2 \Delta S/2 \Delta E/2 = \Delta \sigma /2 \Delta \epsilon /2 \quad (11)$$

where, $K_I = $ stress concentration factor, $\Delta S = $ nominal stress range $\Delta \epsilon = $ nominal strain range, $\Delta \sigma = $ notch stress range, and $\Delta \epsilon = $ notch strain range.
For strain-life fatigue analysis, the stress concentration factor, $K_t$, is replaced by the fatigue notch factor, $K_r$. When the elastic nominal strain is converted to stress, Neuber's rule may be written in the following form:

$$(K_r^2\pi\Delta S)^{\beta}/4E = \Delta \sigma/2 \Delta \varepsilon/2$$ \quad (12)$$

The left-hand side of this equation is a constant for most nominal fatigue geometries. After the notch strain amplitudes have been determined for the given fatigue test geometry, the fatigue life may be calculated from a strain life equation:

$$\Delta \varepsilon/2 = (\sigma'/E)(2N_r)^b + (\sigma''(2N_r)^c$$ \quad (13)$$

Iterative techniques must be employed to solve the equations. Fatigue damage is calculated for each cycle in the loading history and summed.

Advantages of strain life analysis:

1. Accounts for notch root plasticity,
2. Correctly accesses mean stress effects if a sequential analysis is performed,
3. Results in more accurate life estimates for ductile metals.

Limitations are:

1. Requires empirical $K_t$ factors for best results,
2. Not applicable to long life situations where surface finish and other processing variables have a large effect.
The choice of a particular fatigue crack growth prediction model will depend on the information available to the researcher. The strain based approach provides the best estimates for crack initiation whenever notch root plasticity is present. At long lives where plastic strain effects are small, both stress and strain approaches will give satisfactory fatigue life estimates.

In many components, the majority of the useful life is consumed in forming a crack 1 to 2 mm long. Components with a mild stress concentration ($K_t < 4$), made from ductile, materials and with loading histories that have a zero or compressive mean stress are in this category [93]. Crack growth is considered typically for those components that have more severe stress concentrations and/or are loaded with a tensile mean stress.

Most of the life of a space structure will be spent in the nucleation and propagation of a crack. Space structures after long exposure also have a high concentration of micro-meteoroid impact sites which increase roughness and damage of anodized surfaces. This secondary effect produces a population of severe stress concentrations. The section which follows will outline measurements of a material’s ability to resist fracture as under these conditions.

**Toughness**

Toughness describes the ability of a material to deform plastically and to absorb energy before and during rupture. Brittle failures are
characteristic of materials with low-toughness, where ductile failures are characteristic of high-toughness materials. Most low-toughness brittle failures result in cleavage fracture, which is the breaking of bonds along crystallographic planes. Normally face-centered-cubic (fcc) materials, like aluminum, show extensive plastic deformation before fracture stress is reached [56].

The stress distributions at a crack tip is explicitly referred to as the stress intensity factor $K$ and is related to a measure of toughness that takes the general form:

$$K_I = \text{geometric factor} (\beta) \times \frac{\sigma}{\pi a} \quad (14)$$

Any crack in any body of a material will cause fracture at the same critical value of the stress intensity. Fracture stress can be calculated because fracture will occur when stress intensity, $K$, is equal to the toughness:

$$\beta \frac{\sigma}{\pi a} = \text{toughness} \quad (15)$$

for a given $a$ and $\beta$, the fracture stress is predictable. The first fracture mechanics test method used in the United States to calculate fracture toughness was ASTM E399 [94-96]. The use of this specification enables the researcher to establish the critical opening mode stress intensity ($K_{oc}$) for a material of a given geometry. Once this value is determined, the design engineer can determine service load levels through the
relationship:

\[ S = \frac{K_{ic}}{\pi a} \]  \hspace{1cm} (16)

where, \( S \) is the design stress, \( K_{ic} \) is the selected material property and \( a \) is the allowable flaw size or NDT flaw detection.

Measurements of toughness are quite common. Plane-strain fracture toughness, \( K_{ic} \), is particularly pertinent in materials selection because, unlike other measures of toughness, it is independent of specimen configuration. For comparison, the notch toughness of a material, most commonly measured by Charpy testing [97], depends on the configuration on the specimen. Changes in the size of the specimen or in the root radius of the notch will affect the amount of energy absorbed in a Charpy test. The Charpy test measures the total energy required for initiation of the crack from the notch, for propagation of the crack across the specimen, and for complete fracture of the specimen [98]. A \( K_{ic} \) test measures only the critical load required for a small extension of a pre-existing crack [99]. The Charpy test is often used as a screening test for the more difficult plane-strain fracture toughness testing.

The effect of a plasma-anodized coating on the ability of 8090 Al-Li structures to withstand accidental high loads in service has not been determined. In order to ensure the safe life of a material, or to extend the service life of a material, the in-service damage must be analyzed. Specifically for a space structure made of aluminum, it has to be predicted how fast cracks will grow and how fast the remaining strength will
decrease as a result of atomic oxygen attack. Utilizing only the basics of 
fracture mechanics, this work will discuss the re-flight and safe life of 
aluminum structures. The prediction model will also be checked 
experimentally. The results of this work will be used to establish 
procedures to predict safe life of aluminum aerospace structures which 
have been exposed to a simulated space atomic oxygen plasma.
CHAPTER 3
EXPERIMENTAL PROGRAM

Research Objective

The primary objective of this research is to determine the effect of a plasma-anodized film on the engineering properties of aluminum. Physical considerations include those elements of the experimental program which relate to the physical presence of the plasma-anodized film. Mechanical considerations include the mechanical testing used to determine the effects of the measured physical oxides.

Physical Considerations

Five sets of experiments were conducted to evaluate the oxide formed as a result of plasma anodizing. The first test determined the bulk temperature of an aluminum alloy placed in the air plasma. An ESCA survey was subsequently conducted to determine the surface composition of the test specimens. A scanning electron microscope equipped with an energy dispersive X-ray spectrometer was also used to evaluate oxide presence. An experiment designed to determine the existence of a surface compressive stress as a result of plasma exposure followed the SEM/EDS
analysis. Ellipsometry was also used to record the characteristic growth of the surface oxide with increasing exposure time. These five experiment procedures will be detailed in the following sections.

**Reaction Temperature**

The plasma asher, SPI Plasma Prep II, used extensively to generate atomic oxygen durability data of materials, is not equipped with any temperature recording equipment or sensors. The device uses a typical 13.56 MHz RF discharge current to create a gas plasma of oxygen and nitrogen ions and atoms in various energy states without any knowledge of the plasma temperature. However, for this heat treatable alloy work the bulk temperature of the the specimen was considered most critical for the toughness properties. The reaction temperature in the center of the specimen was approximated.

Aluminum which is plasma anodizing in a glass sample chamber of an asher that is kept at 80-100 mTorr forms Al$_2$O$_3$ [24,100]. Al$_2$O$_3$ is extremely stable, with a heat of formation of $\Delta H$[Al$_2$O$_3$,c,298] = -400.5±0.3 kcal/mole [101]. At 298 K the reaction

$$2 \text{Al}(c) + 3 \text{O}(g) \rightarrow \text{Al}_2\text{O}_3(c)$$

(17)

is exothermic by 578 kcal/mole. At orbital energies the reaction is more exothermic; therefore, a measure of the bulk temperature is an adequate temperature approximation for this work. Aluminum exposed to the low-
earth-orbit environment does not lose mass, which suggests that

\[ \text{Al(c)} + \text{O(g)} \rightarrow \text{AlO(g)} \]  

(18)

does not occur, even though it is exothermic in the low earth environment
by 43 kcal/mole at 298 K \([\Delta H_{\text{AlO,g,298}}] = 16 \pm 2 \text{ kcal/mole}\) [102]. This
observation illustrates the difficulty of relating the high impact velocity of
O to a thermodynamic temperature, since the vaporization of \(\text{Al}_2\text{O}_3(c)\) is
known to yield other gaseous oxides [103].

The bulk temperature of a specimen of conventional 6061-T651
aluminum was estimated with a model 755 Fluoroptic temperature probe. The
temperature probe was inserted into the center of the plasma
chamber by removing the glass end plate and replacing it with a sheet of
acrylic plastic. After the fiber optic had passed through the substituted
end plate, a 0.75-in diameter test specimen was core-drilled and
suspended on the end of the sensing tip. Figure 5 is a cross-section of the
standard sensor tip of a Fluoroptic temperature probe.

The sensor consists of a small amount of a temperature sensitive
phosphor mounted at the tip of the fiber-optic probe. The phosphor sensor
material is manganese-activated magnesium fluoropermanate. When
excited with blue-violet light, the phosphor exhibits a deep red
fluorescence. After excitation the optical signals from the probe tip are
filtered and processed by optical, analog, and digital electronics. A
filtered xenon flash-lamp is used as the blue-violet light source for the
probe tip. When the filtered light source is flashed, the phosphor material
is excited. After the excitation pulse is over, the intensity of fluorescent radiation decays. The fluorescent decay time is measured and then correlated with the phosphor temperature by comparing the measured decay time with a digital table for the material. The table produces a RS-232-C data port output signal of the prob tip temperature. Figure 6 is a plot of the specimen heat-up and cool-down profile of temperature in °C vs time in seconds. The temperature of the center of the specimen stabilized to 46.2 °C after 15 minutes.

**ESCA Analysis**

X-ray photoelectron spectroscopy (XPS) or electron spectroscopy for chemical analysis (ESCA), is a surface sensitive spectroscopic tool that provides information about the composition and structure of the outermost surface layers of a solid. The presence of surface modifications as a result of plasma exposure can be documented by determining the surface chemistry with ESCA after exposing the aluminum specimen to the plasma environment [113-115].

The analysis conducted recorded the presence of an oxygen-rich region of the surface of an aluminum specimen after a 40-hour exposure. Figure 7 is a low-resolution, 0 to 1000 eV, scan of a 1 micron finished surface specimen of 6061-T651. The scan shows that the surface chemistry is primarily aluminum, carbon and oxygen. A 40 hour plasma exposure of an identical specimen produces a subtle shift in the oxygen peaks, as can be seen in Figure 8. Silicon is also present in Figure 8 and
is believed to be a silicon-rich region of the specimen under analysis or reaction chamber deposition. Only the oxygen peaks were evaluated because oxygen is the species under consideration. Figures 9 and 10 show a comparison of control vs 40 hours of plasma anodization, respectively. Examination of these two figures shows a characteristic shift and form change of the oxygen peak of the aluminum specimen as a result of plasma anodizing. As would be expected, an oxygen-rich surface resulted after plasma anodizing.

**Energy-Dispersive Spectrum Analysis**

The energy-dispersive x-ray analyzer, in conjunction with the scanning electron microscope was used extensively in this work. These techniques resolve the distinct fractographic information necessary to determine the presence of an oxygen-rich region on the specimen used in the test program. By exposing specimens to the plasma environment and growing a thin film of Al₂O₃ on the samples, the surface modification could be seen. Preliminary work was conducted with the aid of high-power optical microscopy, but the presence of the optically transparent oxide could not be documented [104].

As in the ESCA analysis, two specimens were evaluated. One specimen was unexposed and overloaded to fracture, and a second specimen was exposed to the plasma environment and then fractured in the same manner as the unexposed specimen. As would be expected, the EDS light element analysis raveled that the fracture initiation site of the
ashed sample was an oxygen rich area of the surface. The plasma-exposed specimen fracture initiation site is shown in Figure 11. It shows characteristic $\text{Al}_2\text{O}_3$ film growth, evidenced by the smooth metal/oxide interface layer under the rougher outer oxide layer. Figure 12 and 13 can be used to compare the results of the EDS analysis.

**Reaction Stress**

Reaction stress for this work is defined as that stress that is a result of the plasma transformation of Al to $\text{Al}_2\text{O}_3$. A simple cantilever beam experiment was designed to determine the post reaction stress condition. Internal stresses have been measured by anodizing one side only of strips of aluminum of suitable gauge and measuring the deflection of the free end while the other is rigidly clamped [105]. The stress can be calculated from:

$$S = E \left[ \frac{(t^2 \cdot x)}{3 \cdot l^2 \cdot d} \right]$$  \hspace{1cm} (19)

where $E$ is Young's modulus of the aluminum, $l$, $t$ and $d$ are the dimensions of the strip and $x$ is the displacement of the free end. Since the stress in the oxide film depends on the point of growth (i.e. at the surface, at the metal/oxide interface, etc.), a compressive stress is to be expected in the oxide film formed by the plasma. The oxide will grow at
the metal/oxide interface with a larger lattice spacing than that of the underlying aluminum. Previous researchers have reported that stresses in the film are initially compressive, but become tensile when the limiting coating thickness 700 to 1400 nm is reached [106].

Figure 14 shows the residual stress experiment conducted for this work. Six thin 3003 aluminum foil strips where barrier-coated on all sides but one, with approximately 1000 nm of gold. The coated strips were fastened on one end between two round glass rods with cotton string. Three of the small beams were shorter and placed forward as vertical controls, while the coated side of the center two beams were reversed to act as horizontal controls. The miniature beam experiment was placed in the plasma chamber in front of a lined grid supported by two microscope slides. Figure 15 is a photograph of the experiment installed in the Plasma Prep II device.

The beam experiment was conducted using two different attachment geometries and was repeated twelve times. Each of the plasma exposure durations was 24 hours and produced results that agree with the literature. A thin beam of aluminum 2 inches long, 0.01 inches thick, and 0.1 inches wide is displaced 0.02 inches as a result of a slight compressive stress equal to approximately 180 lbs/in², as calculated by equation (19). The simple beam experiment at 12 hours of operation is shown in Figure 16. Figure 16 also shows the plasma field of the asher.

**Ellipsometric Analysis**

When aluminum is exposed to singlet oxygen atoms, Al₂O₃ is the
energetically preferred reaction product. If reaction conditions are idea for Al₂O₃ crystal growth and the seed material is prior aluminum, then the reaction product would be a single crystal material (sapphire), which is transparent. Less ideal conditions would produce a polycrystalline and fully dense (nonporous) material, which is translucent. Even less ideal growth conditions could produce a polycrystalline material that contains at least 5% porosity and is opaque [107-110]. Less than ideal conditions for this work is assumed because of the presence of the alloying elements in the structural materials under investigation. A mass spectrometer was not used to determine reaction species, which creates a challenging situation in determining a relationship between plasma exposure and fatigue crack growth. Ellipsometry was chosen as a convenient and accurate non-contact technique for the measurement of thicknesses and refractive indexes of the plasma anodized surface. Once a relationship between plasma exposure and fatigue crack growth can be determined, then ellipsometry techniques could be used to monitor quantitatively the effects of plasma exposure.

The lower limit of film thicknesses that can be studied by ellipsometry is at least an order of magnitude smaller than can be studied by other means such as interferometry. Surface artifacts, such as those caused by vacuum in the case of electron microscopy, are not encountered. Neither interferometry nor electron microscopy are as adaptable as ellipsometry to the study of films under plasma interactions. Only ellipsometry will provide the index of refraction of films of unknown thickness [111]. The technique measures changes in the state of
polarization of light upon reflection from a surface. For a clean reflecting surface, the optical constants of the surface and the reflection coefficients of the system may be calculated from these changes. This technique is known to be affected considerably by surface roughness [112].

The Variable Angle Spectroscopic Ellipsometry (VASE) equipment and procedures will be explained Chapter 5. Given the roughness limitation of the equipment preliminary investigations were conducted concerning surface modifications of plasma anodizing [24]. Atomic Force Microscopy (AFM) was used to aid the VASE study of the surface roughness and oxidation of Al as a function of substrate deposition temperature and oxygen plasma exposure. VASE analysis requiring effective media analysis modeling can be significantly hindered by spike-like changes in surface roughness. Figure 17 and 18 show the early and well-developed stages of spike growth, respectively. Each fracture toughness fatigue crack growth specimen was analyzed using ellipsometry before mechanical testing.

**Mechanical Considerations**

Mechanical considerations were defined elsewhere as those experimental procedures which were required to determine the affect plasma anodizing has on the engineering properties of the aluminum systems. Hardness, impact toughness, fracture toughness and fatigue crack propagation were the engineering properties under consideration. Tensile properties were not considered here, because the anodization
reaction is a surface phenomenon. The tensile properties would have been considered if the oxide thickness to specimen thickness ratio had been closer to one. Given that the ratio was expected to be several orders of magnitude lower, and that the chemical reaction under consideration was not expected to modify the bulk properties, only the engineering properties which are affected by surface modifications were considered.

**Hardness**

Hardness testing is one of the most versatile tools available to a materials engineer. Among its many applications, hardness testing can be used to evaluate heat treatment, to approximate the tensile strength of steels, and to detect work hardening or to detect softening or hardening caused by overheating, decarburization, or carbon or nitrogen pickup. Hardness can be generally defined as a materials resistance to permanent indentation. It was used in this investigation to check that the reaction temperature or species did not modify the bulk strengthening characteristics of the test specimens. All of the aluminum systems evaluated later are precipitation strengthening systems and had an unknown susceptibility to the temperature or undesirable species of the plasma chamber.

Preliminary testing was conducted to all test matrix materials using the Rockwell E test [116]. Several measurements of the test matrix material showed, no measurable difference in treated surface hardness vs untreated surface hardness. The exothermic reaction necessary to grow the oxide film in the plasma does not modify the hardness properties of
the materials under consideration.

**Impact Toughness**

Several of the test matrix materials were evaluated by conducting the Charpy impact test. The preliminary tests determined the sensitivity of the instrument. Tests were also conducted to gain insight into the damage mechanisms involved. Plasma-anodized and control specimens of the same orientation were used. Specimen preparation, test equipment and procedures will be discussed in Chapters Four and Five. Toughness and crack-growth behavior depend strongly on a variety of metallurgical, environmental, and mechanical parameters. The effect of exposure on the test materials' impact toughness was evaluated.

The scarcity of toughness data for common alloys raise the question of whether impact properties, such as Charpy-V values, can be used to estimate toughness. The Charpy value is one measure of the fracture energy, and $K_c$ is another measure of the fracture energy. There are differences between a Charpy specimen test and a toughness test.

The Charpy specimen contains a notch, while a toughness specimen contains a crack, which leads to a different state of stress in the two specimens. The state of stress has a considerable influence on the fracture condition. All tests conducted for this work were notched tests in compliance with ASTM E23-88.

A Charpy test measures the total energy required to fracture a specimen. The measured energy includes the plastic energy to “fold” the
specimen. At the upper shelf, where fracture often does not occur, this folding energy is the only component measured. Instrumentation allows the evaluation of a particular point on a specimen by generation of a load vs time plot.

The Charpy test provides the energy to fail the entire uncracked ligament, whereas the fracture toughness test provides the fracture energy $dW/da$, that is, the energy for a small crack extension $da$. If $W$ were independent of $a$, then $dW/da$ could still be obtained from the Charpy result, but in general, $dW/da$ depends strongly on $a$. Finally, because the Charpy is an impact test, strain rates are much higher than those commonly used for toughness tests. ASTM Committee E24 is attempting to establish a recommended test procedure for dynamic notch toughness determination [116].

No attempt is made in this work to establish a relationship between impact properties and toughness. However, one may search for an empirical relationship by plotting Charpy data versus available toughness data for the same materials and by curve fitting the plot if it shows any trends. On the basis of such efforts, certain empirical relationships have been established [117-123].

**Fracture Toughness**

ASTM E-399 is the accepted method for determining $K_{IC}$, the critical value of stress-intensity factor, $K_i$. The measurement corresponds to at most a 2% extension of a pre-existing fatigue crack. The specimen must
be large enough that plane-strain conditions predominate around the crack. Three types of test specimens can be used with the method: the bend specimen, the compact specimen, and the arc specimen. The specimens may be taken from plate and other products forms in any of six orientations for crack growth directions, as shown in Figure 20. The first letter designates the direction perpendicular to the crack plane; second letter designates the direction parallel to the direction of crack growth. For cylindrical products, a similar procedure is used, except that the circumferential and radial (C and R) directions are indicated instead of long transverse and short transverse (T and S) directions.

The critical dimensions for each of the test specimens are thickness B, width W, and overall crack length a, which includes the machined starter notch and the fatigue pre-crack. One of the important requirements of this test method is that the specimen thickness B and the crack length a must be at least equal to the quantity $2.5(K_{IC}/\sigma_{YS})^2$, where $\sigma_{YS}$ is the yield strength of the material. Thus, it is desirable to determine the yield strength and the $K_{IC}$ value for test materials before the specimens are prepared. For some materials, often those of lower strength, the dimensions required by the relation $2.5(K_{IC}/\sigma_{YS})^2$ are frequently greater than can be obtained from the available section sizes of material. For such material and section size combinations, measurement of $K_{IC}$ is not possible. In these cases alternative methods of fracture toughness measurement are necessary. When measurement of $K_{IC}$ is possible, the $K_{IC}$ specimen dimensions should be somewhat greater than the minimum requirements estimated from $2.5(K_{IC}/\sigma_{YS})^2$. For most
aluminum alloys, dimensions about twice those normally required give more consistent $K_{ic}$ results.

Another important set of requirements for a valid $K_{ic}$ test involves the fatigue pre-cracking process. The test results will be valid only if the length of the fatigue crack and the straightness and flatness of the crack are within the prescribed limits. Furthermore, the maximum cyclic load used in producing the fatigue crack must be held below a designated limit to restrict the size of the plastic zone ahead of the fatigue crack.

Once the test specimen has been pre-cracked, the procedure of the $K_{ic}$ test itself is quite similar to the load-versus-displacement procedure of a standard tension test. The displacement used in the $K_{ic}$ test is the crack-mouth-opening displacement of the notch surfaces at the notch mouth in the direction perpendicular to the plane of the notch and crack. A calibrated displacement gage and recording equipment are to measure and record the test data. Special fixtures with loading pins free to rotate during the test are required. This insures that the same free-rotation loading condition used for the stress and $K$ analysis of the specimens is also present during the test. A load-versus-displacement plot for the tests were not available, because the specimens were too small to mount the clip gauges practically. Load-versus-displacement of the crossheads of the testing fixture was available.

Interpretation of the load-versus-displacement plot and calculation of $K_{ic}$ is described in detail in ASTM E-399. The procedures are described briefly here for the compact disk specimen. The provisional value of the load at which onset of crack propagation occurs, $P_{c}$, is obtained from the
test record. The $P_q$ value is either the maximum load during the test or the load corresponding to 2% crack growth as determined by the intersection of a secant line and the plot. $K_q$ is determined using equations such as the following for the compact disk specimen:

$$K_q = \left(\frac{P_q}{B \cdot W^2}\right) \cdot f(a/w) \quad (20)$$

where $K_q$ is in units of MPa* m$^{1/2}$ (psi*in.$^{1/2}$)

$$f(a/w) = [2+(a/w)][0.76+4.8(a/w)-11.58(a/w)^2+11.43(a/w)^3-4.08(a/w)^4]/[1-(a/w)^3]^2$$

and where $P_q$ is load, in MN (lbf); $B$ is specimen thickness, in m (in); $W$ is specimen width, in m (in); and $a$ is crack length, in m (in). For the compact specimen, the crack length is measured from the center line of the loading-pin holes. Equation 20 is considered to be accurate within 0.3% over a range of crack lengths, for $0.2 < a/W < 1$, so it can be used for a variety of fracture mechanics tests and analyses, as well as in the $K_{ic}$ range, $0.45 < a/W < 0.55$.

A $K_q$ value calculated from Eq 20, must meet several criteria in order to be a valid measurement of $K_{ic}$. First, the specimen thickness and overall crack length must be equal to or greater than $2.5(K_{ic}/\sigma_{yB})^2$. The maximum load supported by the specimen prior to complete fracture must not be more than 10% greater than $P_q$, to ensure that $P_q$ corresponds to
the load at which crack extension takes place rather that the load at which excessive plastic deformation occurs around the crack. The fatigue crack portion of the fracture surface is examined and measured as designated in the test method, in regard to straightness of the crack front and other criteria.

Because of the complexity of the calculations and the requirements for validity, a computer program was written to compute $K_q$ values and indicate validity based on appropriate input data. The compact disk specimen is a new $K_{ic}$ test geometry and was selected for this work because of the ease of fabrication, and a maximum number of specimens could be obtained from the minimum stock material. Fabrication is less expensive since turning operations are often faster than milling. The disk geometry mimics the geometry of the truss construction rod, the construction of choice for many existing and future space structures.

**Fatigue Crack Propagation**

ASTM Method E-647 describes the use of center-cracked and compact specimens, but does not supply procedures for fatigue crack growth rate test methods for the compact disk geometry. Because of this lack of detail on fatigue crack propagation for compact disk specimens and the small size of the specimens used, instrumented fatigue pre-cracking was conducted. The specimen thickness-to-width ratio, $B/W$, is smaller than the 0.5 value for $K_{ic}$ tests; the maximum $B/W$ values for center-cracked and compact specimens are 0.125 and 0.25, respectively. With
the thinner specimens, crack lengths measured on the sides of the specimens can be used to represent through-thickness crack growth behavior. The $K_{ic}$ loading fixtures were used for tension-tension fatigue loading, and the maximum and minimum loads were tensile. The load ratio $R = P_{\text{min}}/P_{\text{max}} = 0.1$ was used for all testing. Testing was performed in laboratory air at room temperature. For constant amplitude loading, a set of crack-length versus elapsed-cycle data ($a$ versus $N$) is collected, with the $R$ ratio held constant. The minimum crack length increment, $\Delta a$, between data points is required by ASTM Method E-647 to be larger than 0.10 mm (0.004 inches). The growth rates may be calculated by either of two methods. The secant method is simply the slope of the straight line connecting two adjacent data points. This method results in more scatter in measured crack growth rate. The polynomial method fits a second order polynomial expression to seven adjacent points, and the slope of this expression is the growth rate. The polynomial method eliminates some of the scatter. The measured values of growth rate typically are plotted where $\Delta K$ is calculated from $\Delta P$ using the $K$ expression of equation 20.

A reasonable determination of the presence of structural degradation as a result of plasma anodizing can be made during pre-cracking of the $K_{ic}$ specimens. Also, it is feasible that after long periods of space exposure a section of damaged rod type structure could be removed from service and analyzed, in a like manner to this work, for space radiation effects. This work does not include solar radiation effects but was structured with future use of aluminum lithium extrusions in mind.
CHAPTER 4
SPECIMEN PREPARATION

Material Systems

Several material systems were investigated, and are given in Table II. Of primary interest were the lithium-containing aluminum alloys, because of their applicability to space structure use, and the deficiency of applications data. The other materials were also investigated because of the relative absence of data and the need to consider numerous mechanisms. Most of the work was conducted on the 8090-T8771 plate material and the 6061-T651 bar.

The first section of Table II lists mechanical properties for the monolithic alloys. The 6061 Al-Mg-Si alloy was used to compare the performance of the 8090 Al-Li alloy after exposure to the anodizing plasma. The second section of Table II contains the lists of supporting materials. The 8090-MMC and 7075-MMC materials supporting materials were selected because of their high strength and low ductility, whereas the 1100-MMC was selected because of its low strength and high ductility. The final section of Table II lists toughness values for all of the aluminum alloy systems. Most of the toughness values are from the literature where the values marked with an "*" were produced as a result of this work. Table III also presents the composition of the alloy systems used [124].

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Design

Three specimen designs were used, one for impact and two for fracture toughness testing. Hardness measurements were taken from fractured and sectioned impact toughness samples. The materials for this work were selected to cover a wide range of toughness characteristics. Each sample geometry evaluated low, moderate and high toughness aluminum alloy systems for the effects of plasma exposure.

ASTM E-23-88 standard type A, Charpy (simple-Beam) impact test specimens were used. Figure 21 shows the general specimen design for all of the impact tests conducted. Also, the dashed lines of Figure 21 show possible grain orientations.

The three point bend $K_{ic}$ test geometry for the monolithic Al-Li 8090 plain-strain fracture toughness test have the same general specimen geometry with dimensions as specified in ASTM E-399-83: 85mm (length) by 12.5 mm (thickness) by 19 mm (width). Figure 22 shows the disk-shaped compact $K_{ic}$ test specimen geometry used for fracture mechanics testing. Both $K_{ic}$ and da/dN tests were conducted with this geometry.

Circular bars simulated a “truss” type aerospace structure, common to space applications. Test specimens can be obtained from a limited source of materials which are representative of in-service load paths. The load considered most significant is a bending load, since the application of such a load, caused by service activities as “docking” or “orbit adjustments”, will place the material in tension. This material may be located around or near the ridge of a small crater present after the impact of a micrometeoroid. Micrometeoroid impact is believed to be the only
mechanism by which a fracture would cause plasma anodization of new surfaces.

Machining

Figure 23 shows the sample location for all test specimens machined from the 1/2" x 20" x 26" plate of 8090-T8771 Al-Li. All rough cuts were made with a low-speed steel blade band saw. Area "A" was used for the materials degradation space flight experiment conducted by the Center of the Commercial Development of Space Located at Case Western Reserve University. The space flight experiment is conducted under a joint agreement with NASA and is not part of the present investigation. However, this work is expected to aid in the understanding of some of the post-space flight exposure phenomena. The areas marked "P" are reserved for post-flight analysis. The slanted lines on the bottom of the figure indicate the discarded area very near the stretcher grips. Areas marked "B", were used for the three-point bend $K_{IC}$ test specimens, and areas indicated by the "D" were reserved for post-flight button-head tension test specimens. The other sections, "C" and "E" were used for the Charpy V-notched specimens.

Figure 24 shows the sample layout for the 30mm x 60mm x 90mm bar of 7075 + SiC MMC used strictly for Charpy V-notched impact toughness test samples. The two views show a) test sample location, and b) T-S notch orientation. The 7075 specimens where also rough-machined into sections for milling with a low-speed steel band saw.
After milling to the tolerances indicated the applicable ASTM specifications, the necessary notches where cut using a SiC grinding wheel with an oil-water solution to control cutting temperatures. Figure 25 shows is the notch geometry used for this work.

The remainder of the test material was electo-discharge machined (EDM) from circular bars. The conventional 6061 monolithic aluminum was cut from a bar 6.3 inches long and 0.85 inches in diameter. The non-conventional 8090 monolithic aluminum was from a bar 5.0 inches long and 1.00 inch in diameter. The 8090 aluminum-lithium matrix SiC particulate composite samples were fabricated from a bar 3.6 inches long and 0.9 inches in diameter. The conventional 1100 aluminum matrix Al₂O₃ particulate composite specimens were cut from a small section of bar 1.6 inches long and 0.74 inches wide. Electro-discharge machining (EDM) was used to produce the intricate details needed for the fracture toughness test samples. EDM effects mechanical properties and microstructure near the cut region on the work piece [125]. This effect was eliminated because all EDM specimens were pre-cracked outside the EDM-affected region. The Al-Li with SiC particulate reinforced composites showed signs of oxidation because of the higher energy required in EDM. Figure 26 shows a rough-finished fracture mechanics sample.

No effort was made to stress relieve any of the samples after machining. The as-machined specimen was designed to replicate a fabricated space structure that had been in service for some time.
Cleaning and Polishing

After the samples were fabricated, they were cleaned with methanol, rinsed with tap water and air dried. This first cleaning removed any surface residue from machining and minimized contamination. All compact fracture toughness and the 8090 plate impact test specimens were polished on one side. The samples were first lightly ground using 600 grit paper to adjust dimensional tolerances where necessary with a SiC impregnated grinding disk. Additional polishing was performed using 800, 1200, and 1800 grit alumina. Final polishing was completed using 2400 and 3-micron polishing disks. The final 3-micron finish improved the ellipsometric signal for measuring the changes in optical properties as a result of exposure to the anodizing plasma.

After polishing, all samples were ultrasonically cleaned. Cleaning was repeated after exposure to the plasma environment. The ultrasonic cleaning removed any low-adhesion contamination products occurring as a result of plasma exposure. Optical property experiments conducted with ellipsometric equipment are known to be susceptible to surface roughness and contaminants.

Specimen Dimensions

Table IV-A gives sample dimensions in inches and shows dimensions of all Charpy impact test specimens. The specimens which are marked with an “*” have before and after commercial anodizing
dimensions tabulated. Several of the “E” series 8090 specimens were intentionally left oversized after milling. The oversized samples facilitated evaluation of the microstructural sensitivity of the 8090 plate material.

Table IV-B, shows dimensions of all fracture mechanics specimens. Even though toughness was theoretically different for most of the materials, the same general dimensions were used so that fatigue pre-crack data could generate empirical relationships between da/dN and plasma exposure time. The dimension of the three-point bend $K_{ic}$ specimens are not included on Table IV-B because samples B1 through B5 were the same and the computational data has been reported previously [126]. These control $K_{ic}$ samples were necessary to qualify the disk made from sample area “A” for flight on the NASA space shuttle.

**Plasma Anodizing**

Normally the first stage in the oxidation of a perfectly clean metal surface is chemisorption of oxygen, in which oxygen molecules absorbed at the metal surface dissociate to form atoms, which then share electrons with adjacent metal atoms. This step is greatly enhanced in plasma oxidation/anodizing because of the radio frequency dissociation of the oxygen molecules and conservation of energy. As this process occurs, some metal atoms move into the plane of the absorbed atoms. The result is an extremely stable, adherent, continuous monolayer of alumina.

Further film growth proceeds by diffusion of electrons and metal
and oxygen ions. At this stage, growth is confined to energetically favorable nucleation sites where local epitaxial accumulations of oxide form. The nuclei then grow laterally by surface diffusion to produce, ideally, a continuous oxide layer of uniform thickness. For low-temperature formation and thin films (less than 200 nm), the growth relationship is known to be logarithmic and has been measured. Figure 2 shows the form of the growth, but suggests that the films are less than ideal.

The test specimens were exposed to the oxygen plasma chamber and anodized on all surfaces. Specimens were placed in the chamber so that the maximum surface area was exposed to the plasma. Figure 26 shows chamber placement for the compact toughness specimens. The Charpy test samples were exposed so that the notch received the highest exposure to the oxygen plasma. Test specimens were exposed to the plasma for varying lengths of time. No attempt was made to control or vary the atomic oxygen plasma environment because the oxidation rate is not under investigation.

Six of the Charpy V-notched test specimens were subjected to a conventional commercial anodizing treatment. The specimens are indicated one Table V by an “*”。The commercial anodizing treatment is a 12-15% solution of H$_2$SO$_4$ at 46°F. The anodizing voltage was kept at 30 to 36 V and the specimens were removed at different times. The varied times produced several mils variance in oxide thickness. This chemical treatment aided in the evaluation of the extremes of anodization. Alumina as thick as 4.9 mils was measured via a conductance method on
one test specimen.

Table V records the exposure times. The uneven plasma environment of the chamber, as seen in Figure 16, is compensated for by specimen's chamber location. Plasma anodizing produced a light-gray iridescent film which, with heavier treatment, turned dark gray. Figures 27 through 35 show some of the interesting macroscopic characteristics of oxygen plasma exposure.

A plasma-anodized sample of the 6061 series material after 512 hours of exposure is shown in Figure 27. The sample appears to be more spectrally diffuse than the sample seen in Figure 26. Comparison of Figures 28 and 29 indicates that there is a connection between iridescent rings and exposure time. This result is believed to be due to the increased number of chamber opening cycles seen by the -11 samples, since samples were removed from the chamber one at a time after the desired exposure time was obtained. Figures 28 and 29 show that the Al-Li monolithic samples appear to become more reflective with increased exposure time. Comparison of Figures 30 through 33 also show a general increase in the optical reflectance, exaggerated grain structure and iridescent film growth on the Al-Li + SiC samples. A comparison of Figures 34 and 35 clearly shows the size of the EDM-affected region, increase in surface roughness, and increase in reflectance.

The dark gray regions around the EDM sections of sample 1100-03 were examined using an optical microscope. Figures 36 and 37 are dark field photomicrographs of the area and showing the presence of a thin oxide film after 512 hours of plasma exposure. Although these effects are
entirely qualitative some surface chemistry changes are present as a result of oxygen plasma exposure.

**Instrumentation**

The instrumentation attached to the test specimens is limited because ellipsometry is strictly non-contact. Only the circular compact fracture toughness specimens were directly instrumented. The instrumentation used was a crack propagation gage bonded to the unpolished plasma anodized side of the test specimens. Data recording equipment will be detailed in Chapter 5.

A successful test requires precise gage installation. The specimen surface preparation, cleaning, installation, bonding, curing and wiring techniques for the installation of a KRAK-GAGE are identical to the well-known procedures for foil-type strain-gages. Standard commercially available two-component strain-gage type adhesives were used in two batch applications of the gages. Batch installation was used to minimize data scatter caused by variance in installation procedures. The relatively large bonding area of a KRAK-GAGE demanded required a high-quality bond. A very thin, void-free and brittle glue line is the ideal interface. Bonding jigs achieved required clamping pressure of 30 psi during the bonding/curing cycle. The adhesive was cured under pressure for 40 minuets at a temperature of 100°C.

Surface preparation steps for gage installation included: solvent degreasing; surface preparation (abrading); layout of alignment marks;
surface conditioning and neutralizing; and gage lay-out alignment mark. Proper alignment of the KRAK-GAGE on the specimen was important in order to etch a line across the root of the starter notch. Each KRAK-GAGE has suitable triangular alignment marks for precise mounting. Figure 38 shows the 5-mm KG-A050CE KRAK-GAGE mounted on three disk shaped compact-tension specimens. The instrument interface with the FRACTOMATIC recording equipment will be discussed in Chapter 5.
CHAPTER 5
TESTING MACHINES AND PROCEDURES

Variable Angle Spectroscopic Ellipsometry

Ellipsometry was introduced in the 1890's, but until computers became available, it was slow to perform (127). Advances in computer technology have made ellipsometric data acquisition and analysis rapid and accurate.

Early ellipsometry work focussed on improving the technique, whereas attention now emphasizes application to materials analysis. Ellipsometry is used to determine changes in optical constants as a result of plasma anodization. Common examples of other uses of ellipsometry are measurements of oxide and nitride films on silicon wafers, and dielectric films deposited on optical surfaces (127-133).

A collimated linearly polarized light beam was directed at the test specimen, and the polarization state of the reflected light was determined. To maximize sensitivity, the angle of incidence to the sample normal was varied between 69, 71 and 73 degrees. The wavelength of the incident light was also controlled between 350 and 800 nm (127-132). The general form of a working ellipsometric model is shown in Figure 39.

The measured quantities are expressed in terms of a complex number having magnitude \( \tan \psi \) and phase \( \Delta \):

\[
P = \tan \psi \exp j \Delta
\]

(21)

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The instrument used for these experiments measures both a $\psi_i^m$ and a $\Delta_i^m$ for the $i$th angle of incidence and wavelength combination. An analytical relationship enabling transformation of the measured values into the desired optical constants, film thicknesses, or constituent materials fraction does not exit (133). The software supporting the experiment performed a regression analysis in which $\psi_i^c$ and $\Delta_i^c$ are calculated from an assumed model for the structure using the Fresnel equations for reflection of polarized light from stratified materials (127).

The unknown parameters of the model, such as film thicknesses, optical constants, or constituent material fraction, are varied until a best fit between measured $\psi_i^m$ and $\Delta_i^m$ and calculated $\psi_i^c$ and $\Delta_i^c$ is found. The mean square error (MSE) is used as a measure of the fit:

\[
\text{MSE} = \frac{1}{N} \sum_{i=1}^{N} (\psi_i^c - \psi_i^m)^2 + (\Delta_i^c - \Delta_i^m)^2
\]  

(22)

where $N$ is the number of wavelength and angle of incidence combinations used.

A problem in applying ellipsometry is knowing when the parameters of the model are mathematically correlated; for example, a thicker film with a lower index of refraction might give the same MSE as some other combination of index and thickness. Several models may produce equivalent low MSEs. All available information about the sample should be evaluated to develop a physically realistic model. The simple model used to support this work, shown in Figure 40, is a two-layer structure with a varying amount of void fraction in the top layer.
Access to the correlation matrix generated during the regression analysis is available, if necessary, for more specific applications (127,133). The solution to the correlation problem is to make measurements at optimum wavelength and angle combinations, and to keep the assumed model simple yet realistic. Even then, it is sometimes not possible to avoid correlation, yet it is important to know the degree of correlation when it exists. Predictive modeling can be performed prior to making any measurements to determine the optimum wavelength and angle combinations, and when there are likely to be correlated variables (130,133).

The incident light beam was in the visible range of wavelength ranging from 350 nm (blue) to 750 nm (red) for ease of equipment operation (133). These waves have associated electric (E), and magnetic (H) components. These are related mathematically to each other, the (E) component can be treated alone. Figure 39 shows the locus of projection of electric vector of light wave on a screen perpendicular to the propagation direction for elliptical polarized light. As polarized light propagates in space and time, separated into its x and y vector components, screen projection are straight lines. The elliptical shape of the x and y components occurs as a result of an out-of-phase condition caused by surface modification.

A complete description of the polarization state includes: the azimuthal angle relative to a plane of reference; the ellipticity (e = b/a); the handedness: right-handed rotation describes clockwise rotation when looking into the beam; the amplitude \( A = (a^2+b^2)^{\frac{1}{2}} \); and the absolute
phase. In ellipsometry only the azimuthal angle and the ellipticity are determined. The absolute intensity or phase of the light doesn’t need to be measured, which simplifies the instrumentation. A built-in filter was used to eliminate the need of knowing the handedness.

All electromagnetic phenomena are governed by Maxwell’s equations, and one of the consequences is that certain mathematical relationships can be determined when light encounters boundaries between media (127,133). Three important conclusions result for ellipsometry. First the angle of incidence equals the angle of reflectance, as shown in Figure 39. Second, Snell’s law applies \( n_i \sin \theta_i = n_o \sin \theta_o \) where \( n_i \) and \( n_o \) are the indexes of refraction in media 1, and media 0, and the angles \( \theta_i \) and \( \theta_o \) are the angle of incidence and reflection). Third, Fresnel reflection coefficients are:

\[
    r_s = \frac{E^r_s}{E^i_s} = n_o \cos \theta_o - n_i \cos \theta_i / n_o \cos \theta_i + n_i \cos \theta_o
\]

(23)

and

\[
    r_p = \frac{E^r_p}{E^i_p} = n_i \cos \theta_i - n_o \cos \theta_i / n_i \cos \theta_i + n_o \cos \theta_i
\]

(24)

where \( s \) refers to the light vector component perpendicular to the plane of incidence, \( p \) is the component parallel to the plane of incidence, and \( r \) and \( i \) are reflected and incoming light. The plane of incidence is defined by the incoming and outgoing beams and the normal to the sample. The complex indices of refraction for media 1 and 0 are given by \( n_i \) and \( n_o \). The relations \( r_s \) and \( r_p \) are the complex Fresnel reflection coefficients.
Their ratio is what is measured in ellipsometry:

\[ p = \frac{r'}{r_s} = \tan \psi \exp j \Delta \]  
(25)

Thus measurements of \( \psi \) and \( \Delta \) are related to the properties of matter via Fresnel coefficients derived from the boundary conditions of electromagnetic theory (127,133).

Equations 23 and 24 describe a simple system like an air-to-bulk interface. The materials under investigation have natural oxides and surface roughness. Each layer and interface shown in Figure 40 was represented by a 2x2 matrix as a function of instrument software, and the overall reflection properties were calculated by matrix multiplication (127). The software performs regression analysis to determine the system physical parameters (127,128,132).

Automated ellipsometers are currently available in three general types: polarization modulation, rotating analyzer, and rotating polarizer (127-129). Figure 41 is the schematic of the rotating analyzer system used in this investigation (133), illustrating the light source, monochromator, collimating optics, polarizer, sample mount, rotating polarizer (called the analyzer), and a detector. The intensity of the light measured at the detector oscillates sinusoidally according to:

\[ I = 1 + \alpha \cos 2A + \beta \sin 2A \]  
(26)

where \( \alpha \) and \( \beta \) are the Fourier coefficients, and \( A \) is the azimuthal angle
between the polarizer “fast axis” and the incident plane. By recording the intensity vs. A in a computer, the α and β, and thus ψ and Δ, can be determined. By changing the incident angle and wavelength, it is possible to determine sets of ψᵢ and Δᵢ values for the regression analysis in order to derive the changes in optical characteristics of the test specimens after plasma exposure.

The polarizer and analyzer azimuthal angles relative to the incident plane were calibrated before each experiment. Calibration is based on the minimum signal observed when the fast axes of two polarizers are perpendicular to each other, and was provided by the instrument supplier. The VASE instrument was set up in a class 10 clean room, which minimized airborne optical impurities and maintained calibration values because of controlled temperature and stable relative humidities. Experimental data plots are contained in Appendix A and shall be discussed in the next chapter.

**Dynatup 830 Impact Tester**

Instrumented impact testing can be performed with a drop weight impact test machine or with a pendulum impact test machine. A Wiedemann-Baldwin impact machine (Model S1-1D) was used to break the test specimen in the 60 ft-lb range. A model 830-I data acquisition system acquired, analyzed, plotted and stored a complete record of each impact test. The equipment assisted in the determining whether impact characteristics of the materials tested were affected primarily in the
propagation or initiation stage of fracture. The major components of the
tester and data acquisition system are shown in Figure 42.

The data acquisition board was mounted in a computer and
perform all the signal conditioning and data acquisition functions
necessary to acquire load and time information during the impact event.
The tester used a strain-gage load cell transducer automatically
calibrated by the data acquisition board prior to every test. Velocity
detection was done using the beam of an optical switch that was
interrupted by a metal double-pronged flag. Time was measured from the
moment the leading edge of the first prong interrupted the beam to the
moment the leading edge of the second prong interrupted the beam.

Data computation was completed by the software using accepted
mathematical formulas. To calculate specimen deflection, specimen
velocity and energy absorbed by the specimen, the instrument software
uses measured load-time data and the instantaneous velocity at the
moment of impact. The test procedures were in accordance with ASTM E-
23-88 [97]. Figure 43 shows the test support equipment and the upper
portion of the tester.

**KRAK-GAGE Fatigue Crack Growth Tester**

The KRAK-GAGE is essentially a thin-film bonded electrical
transducer which provides an infinite resolution DC voltage output
proportional to a crack length. As the disk-shaped compact fracture
toughness test specimens are fatigue pre-cracked, the crack length
recorded will extend and the resistance of the bonded KRAK-GAGE will change.

More specifically the nominal resistance of the “uncracked” constantan KRAK-GAGE used was less than one ohm across the measuring section. The resistance increases proportional to crack length or a nominal 1 ohm increase for the 5-mm crack gage length. By applying a constant current of approximately 100 mA, gage output voltage for a crack 0 to a full 5-mm long is 100 mV [134]. During the fatigue pre-crack-growth and fracture tests, the KRAK-GAGE measuring section cracked coincident with the crack in the actual test specimen. Therefore, the change in gage resistance is linear and directly proportional to crack length in the specimen.

A constant current ran through the outside pair of leads on the KRAK-GAGE as shown in Figure 44. The voltage change between points was monitored via the inside pair of leads, resulting in output vs. crack length. By applying the proper level of excitation current I, the change in output voltage is 100 mV, for 100% of the 5-mm gage length. Therefore, the initial output voltage is 88.8 mV for an uncracked gage according to the resistance ratio [134]. By using a razor blade to pre-cut the gage and balancing the gage output (zero calibration) against an internal reference voltage, this initial output voltage is offset, and the change in voltage is automatically reported. The FRACTOMAT is the controller interface for the gages and contains the adjustable constant precision current sources, the required internal reference voltage, zero calibration circuits and amplifiers that raise the signal to a full 0 to 10 V DC [134].
Zero shifts, which would result in an “apparent crack-length” due to temperature changes are less than 0.0025%/°F of the 5-mm crack length [134]. The test temperature and relative humidity has been recorded and found to be no greater than 72°F ± 2 and 45°F ± 5 respectfully. All voltages were converted to digital signals to control the tension-tension pre-crack fatigue load application on the compact fracture toughness test specimen. Appendix B contains the fracture mechanics crack growth da/dN plots and supporting data obtained by FRACTOMAT signal conditioning. Figure 45 shows the instrumented fatigue pre-crack test in operation on the MTS. Test procedures were in accordance with ASTM E-399-83 [94].

**MTS Mechanical Testing Machine**

An MTS testing machine with a 500 lb. load cell, mechanical grips, load plane alignment clevis, and a Hewlett Packard data acquisition system, was used for the fracture mechanics testing. Load application control was digital and a standard load vs crosshead displacement record kept. The raw data to support the fatigue-crack growth analysis and plane strain fracture toughness testing is plotted in Appendix B.

The plane-strain fracture toughness test was use for qualitative toughness evaluation because of its widespread use and acceptance. Both the three-point-bend beam and the disk-shaped compact specimens were tested at room temperature. Temperature recording equipment and the test stand setup are shown in Figure 45. Test were run in accordance with ASTM E-399-83 [94].
Digital input variables were maintained on specimen log sheets in order to calculate $K_q$ in accordance with ASTM E-399-83. The fatigue cracking was done at a $R$ ratio of 0.1 and load was shed to ensure that the maximum $k$ during fatigue was well below the suggested limit of 60% $K_{IC}$. Once the fatigue cracking was complete, the specimens were tested under displacement control as per standard, and plots of load vs displacement curves were made for calculation of $K_{IC}$. Load vs displacement curves are found in Appendix B.

The data file format used the formulas in the ASTM specification E-399-83 for the test geometry. The data files contain lists of $\Delta K$, $da/dN$, $a$, cycles and plastic zone size. The starting point for the fatigue pre-crack tests was determined by data input from the FRACTOMAT reading, and the specimen dimensions table. Starting point input variables were desired $\Delta K$, $W$, $B$, $a$, and pre-cut reading. Material yield strength was also input for the calculation of plastic zone.

After the MTS digital control program was started, the $R=0.1$ (tension-tension) load was applied to obtain the desired $\Delta K$ value until the KRAK-GAGE indicated that the fatigue pre-crack had propagated 0.1 mm. Then a digital signal was sent to the HP controller for the MTS to command a 90% shed of the applied load for $\Delta K$s greater than 10 MPa/m. For $\Delta K$s less than 10 MPa/m the load shed was at 95% increments of applied load. The results of these mechanical tests and the other tests conducted to support this work are discussed in the results section.
CHAPTER 6
RESULTS AND DISCUSSION

Plasma Anodizing Study

The results of the ellipsometer experiments contained in Appendix A will be discussed as four separate sets of data. The data for the 1100 series fracture toughness samples are presented first and the 6061 series of samples are discussed second. The data reviewed covers the two 8090 sequences of samples. The 1100 series materials are discussed first because the analysis of these samples is ideal, since the specimens were commercially pure aluminum with aluminum oxide as a particulate reinforcement.

As was expected, Figures 34 and 35 clearly show some of the effect of exposure to the anodizing atomic oxygen plasma. The area around the load pin hole and the starter crack of Figure 35 shows that a thick film has formed as a result of natural oxide film thickening because of EDM preparation. This observation indicates that this 1100-03 sample sets the limits of oxide thickness obtainable from a 512 hour exposure to be less than about 3300 nm or 3.3 micron. This thickness is only approximate and is derived by comparison with the 7000 series samples that were commercially anodized to form alumina coating .459 to .579 mil thick measured by conductance. Thickness of the oxide was also estimated by measuring the oxide on a SEM photomicrograph. The 512 hours exposure
to the plasma asher is equal to approximately 10 years of space exposure since the flux of oxygen atoms in the asher was equal to 4.0 to 6.0 E+15 atom/cm²*sec [16,22,24,48,100].

If the film is conservatively stated to be one half of the value approximated, the complex film structure suggests that an ellipsometric analysis of the oxide thickness is not practical. Figure 46 is a plot of the experimental and generated data based on the model in Figure 40. These findings are in agreement with the literature, since the 160 nm oxide scale measured using the model is known to be porous and rough [24,46,110-112]. The change in optical properties of the exposed surface forms the focus of the ellipsometric analysis reported in this work. Table VI contains a summary of the optical constants determined.

Two 1100 series specimens were prepared for this work and exposed to the anodizing plasma gas. These specimens were subsequently evaluated optically with the VASE. The results of the ellipsometric experiments are shown in Figures A-29 and A-30. The specimens, as listed in Table V, were exposed to the ionized gas for 32 and 512 hours respectively. Comparison of Figures A-29 and A-30 presents evidence that fairly large shifts in the ellipsometric waveform data can be expected from plasma exposure. Large shifts are noted in both phase and intensity data as a result of increased exposure time. This is because of the increased surface roughness and surface chemistry modification indicated by equation (17).

The experimental values were obtained by using the equations (21-26) of Chapter 5 with the aid of the software provided by the instrument
manufacturer. The calibration sequence was verified several times with the manufacturer. After storing the experimental data points, the data were used for extracting the optical constants of the exposed specimen. Figure 47, a) and b) are the optical constants of the refractive index, \( n \) and absorption coefficient, \( k \) respectively determined with the ellipsometer. Examination of Figure 47 indicates that a gradual increase in the refractive index and a decrease in absorption coefficient has occurred. It is also noted that the modifications to the waveform across the experimental data spectrum became less linear as the exposure time for refractive indexes values increased. This is not the case with the absorption coefficient measurements. This indicates that a mechanism is active which affects the optical constants differently. This mechanism is believed to be a structural change from the primary dielectric to an increased amount of ceramic material at the surface. This results in the collimation of the previously diffuse signal. This mechanism is supported by the fact that after ashing the signals in to the instrument analyzer became stronger. This is to say that the 1100 and 8090 MMC specimens were much easier to obtain an accurate signal for analyzing after exposure to the ionizing gas.

The optical constants for the mercury green line near wavelength 550 nm obtained from the ellipsometric experiments are recorded for the 1100 series samples and other specimens, in Table VI. Table VI consists of two sections: a) is a record of refractive index, and b) is a record of absorption coefficient, \( k \). The results presented in Table VI are in general agreement with previous actual space exposure from similar
materials [3,13,22].

Ten 6061-T651 series specimens were prepared, exposed to the anodizing plasma gas and evaluated optically with the VASE, in the same manner as the previous samples. The results of the ellipsometric experiments are shown in Figures A-1 through A-10. The specimens, as listed in Table V, were exposed to the ionized gas for 1, 2, 4, 8, 16, 32, 64, 128, 256 and 512 hours respectively. Comparison of Figures A-1 through A-10 presents evidence that the same large shifts in the ellipsometric waveform data from the 1100 samples occur from plasma exposure of the 6000 series samples. The large shifts in both phase and intensity data with increased surface roughness and surface chemistry modification become apparent after 64 hours but before 128 hours of exposure.

Figure 48, a) and b) are the optical constants for the 6000 series specimens that were determined by ellipsometry. Examination of Figure 48, indicates that the same gradual decrease in the refractive index for the pure aluminum MMC occurs for the aluminum alloy. This alloy has very near the same “n” as published for pure metallic aluminum. The decrease in absorption coefficient is not apparent, because this system generates more positive values of “k” as the exposure time is increased. The modifications to the waveform across the experimental data spectrum are linear as a function of exposure time vs the characteristic constant. This did not occur with the optical constants determined experimentally for the pure aluminum. The mechanism which produced the collimated signal, is not controlling the surface structure of this alloy. The texture is the controlling mechanism over 64 hours of exposure. The sharp decrease
in both "n" and "k" values for the 128 hours exposure time shown in Figure A-8 is attributed to the presence of a deposition product or a surface inclusion that is optically "dead" from wavelength 350 to 550 nm at an angle of incident of 69 degrees.

The optical constants for ellipsometric experiments are recorded for the 6000 series samples Table VI. The refractive index and absorption coefficient values in Table VI were insufficient to establish a comparison for the 1100 series samples. Some indications are evident for the 8090 Al-Li series specimens of the VASE experiments. These will be discussed in the subsequent paragraphs of this section on VASE experiments.

Eighteen 8090 aluminum-lithium specimens were prepared and exposed to the anodizing plasma gas. All samples were subsequently evaluated optically with the VASE. Ten of the eighteen specimens were artificially aged. These included the monolithic 8090 Al-Li systems and the remaining eight 8090 specimens of metal matrix composite systems containing SiC as particulate reinforcements. The results of the ellipsometric experiments are shown in Figures A-11 through A-20 for the monolithic system and Figures A-21 through A-27 for the MMC samples.

Comparative analysis of Figures A-11 through A-20 presents evidence that the same large shifts in the ellipsometric waveform data from the 1100 and the 6000 samples can be expected from plasma exposure of the 8090 series samples. The large shifts in the phase and intensity data are believed to be a result of increased surface roughness and surface chemistry. The modification of the surface is apparent in the readings between 128 and 256 hours of exposure. The same waveform
shifts after 64 hours of exposure occur in the 8090 alloy as observed for the 6000 series samples.

Figure 49 & 50, a) and b) are the optical constants for the 8090 Al-Li series specimens and the 8090+SiC MMC specimens respectively. Examination of Figure 49 & 50, indicates that the same gradual decrease in the refractive index for the pure aluminum system occurs with the 8090 aluminum alloy and MMC. The absorption coefficient also increases. The modifications to the waveform across the experimental data spectrum are more linear as a function of exposure time for both characteristic constants “n”. This was not the case with optical constant “k”, however; the largest shift in the absorption coefficient occurs at 512 hours for the 8090+SiC MMC shown in Figure 50 b). The determined values for “k” recorded for the 8090 monolithic system are linear and similar to the pure aluminum values.

The uniform waveform shown on Figure A-19 a) is attributed to a intentional change in conditions in the reaction chamber. The A8090-10 specimen was placed in an ionized gas produced from dry bottled air, instead of ambient air. The purpose of this modification was to determine the effect of water vapor as oxidation occurs. Lithium hydride is expected to occur on the surface as a result of the induction of ambient air into the ionizing gas chamber [135]. The presence of the unstable lithium hydride is evidenced by the remnants of gas bubbles that can be seen on Figure 61. The characteristic positive phase change in VASE data occurs as a result of this exposure.

The optical constants reported for the ellipsometric experiments are
also recorded for the 8000 series samples in Table VI. In Table VI, the refractive index and absorption coefficient trends are sufficient to present some general observations for all series of the alloys tested. These trends occur as two distinct observations; one is related to refractive index and the other to the absorption coefficient. Across all categories of alloys tested, the refractive index “n” is inversely proportional to the exposure time and the absorption coefficient, “k” increases proportionally to exposure time.

The longest exposure time produces slight reversing effects. The gradual reversal of the reported effects that are shown on Table VI for 512 hours, is attributed to the surface of the alloy taking on more of the refractive properties of the alumina. Alumina has a “n” value of 1.67 for the reported wavelength and a “k” value of zero.

Fatigue Crack Growth Behavior

The data shown on the da/dN Figures contained in Appendix B will not be discussed individually. The data is provided, however, to aid the interested reader in extracting quantitative data from which to make service projections for the aluminum alloy systems examined. The FRACTOMAT and KRAK-GAGE data presented in Appendix B has been summarized on Table VII and is in reasonably good agreement for similar materials [47,52,53, 136]. Table VII consists of three separate presentations of recorded data during the mechanical testing portion of this work. Section a), is a summary of the data from the 6000 series
specimens and contains more detail. The expansion of detail for this aerospace alloy system was necessary to establish the baseline from which the less commonly used 8000 series alloys can be compared. Sections b) and c) are the sub-critical fatigue crack growth behavior of the 8090 and 8090+SiC series specimens. Changes in specimen thickness to oxide thickness ratio would have produced different results. The affect of the ionizing gas would have been much greater if the specimen thickness was considerably less. This also indicates that a very thick specimen should not have as much affect.

Review of Table VII values after 256 hours of exposure to the ionizing gas shows that 44 % ± 2 % fewer cycles are required to grow the same 1 mm length crack over a ΔK range of 10 to 7 MPam^{1/2}. The percentage reduction in cycles is lower at lower ΔK. The value decreases to 8 % ± .4 % for ΔK values of 5 to 7 MPam^{1/2} because the mechanism controlling the crack propagation is less surface and more bulk sensitive. As a result of plasma exposure, an average value of approximately 30 % ± 2 % fewer cycles are calculated across the upper and lower ranges tested. This reported average value is obtained by selecting the appropriate values for crack length and cycles for a given test from the experimental data. The experimental data was analyzed using formulas (1) through (5) and formula (20) for the specific specimen geometry tested. The approximate value stated above was estimated by adding the experimental data together for all ranges and dividing by the number of ranges reported. This general procedure was followed for all the materials tested. The cause of this reduction in cycles to grow the same
length crack is attributed to the increase in surface roughness and the brittle nature of the oxide which is formed as a result of the exposure. Figure 51 is a macrophotograph of two specimens. The photo shows that the propagating crack front moves faster \( \perp \) perpendicular to the polished surface after plasma exposure. The same macroscopic observation can be made after viewing the specimen fracture surfaces in Figure 52. The specimens in Figure 52 were exposed for less hours but still produced results characteristic of the lower \( \Delta K \) experiments.

The data produced in the fatigue experiments was also analyzed using statistical methods. The simple linear regression statistical model used produced the equation of a best fit line to the data. The first step in regression analysis involving two variables, \( \Delta K \) and \( da/dN \), was to construct a scatter plot of the observed data. In such a plot, each \((x,y)\) is represented as a point plotted on a two-dimensional coordinates system. Figure 4 contains a scatter plot of the data from specimens 6061-1 and 6061-9. No simple curve passes through all the points in the plot. However, a strong tendency exists for a high \( \Delta K \) to be associated with a high value of \( da/dN \). This suggests a relationship of these two values. For the model \( y = x^\alpha \Delta K^\beta \), the actual observed value of \( y \) is a power function of \( x \) over the propagation region tested. The appropriate generalization of this to a probabilistic model assumes that the expected value \( Y \) is a power function of \( x \), but that for fixed \( x \), the variable \( Y \) differs from its expected value by a random amount. The error term in the model was calculated using a computer and the true regression line of exposed and not exposed specimens is shown on Figure 4. This regression analysis determined the
relationship between the variables $\Delta K$ and $da/dN$. The standard error of
the $y$ values was computed using the square root of the sum of squares
(SSE) divided by degrees of freedom. Sum of squares is defined as
$A_1x_1 + \ldots + A_kx_k + C - y$, where the $y$-intercept ($C$) is computed rather than
forced to zero. Somewhat low values of $R^2$ squared were consistently
obtained for all data points analyzed. The $R^2$ squared values of 0.98 to 0.99
were a result of the known tendency of the fatigue crack data to be
represented by the Paris law presented in equation (2). The comparison
of the data shown in Table VII and the power regression model is the
basis for the $\pm$ error bands reported.

Two of the issues which should be considered are: 1) the effect of
KRAK-GAGE installation on retardation of crack growth; and 2) the
susceptibility this alloy to an increased rate of crack propagation as a
result of surface roughening [32-40]. Figure 53 is a specimen which
received a 15 psi higher cure pressure at the same temperature and
duration, but was left unpolished. The unpolished surface characteristics
in Figure 53 resulted in about the same percentage change (approximate
42% fewer cycles unpolished) for 512 hour plasma exposure. This
observation indicates that the roughness of the surface is the dominant
mechanism over KRAK-GAGE crack growth retardation.

Figure 53 through Figure 56 demonstrate the tendency for the 8090
monolithic materials to fracture along preferential grain directions [25-
31]. It is noted that the fatigue pre-crack region of the fracture surfaces
are also affected by the grain orientation of the specimen. This behavior
produced the unexpected fatigue crack growth measurement for the
experiments with the 8000 series alloy. The polished 8090+SiC materials after plasma exposure shown in Figures 57 and 58, shows clearly the segregation of the particulate reinforcements as grain. The results of specimen B8090-05 of Figure 57 were intended to be compared with specimen B8090-06; however, the KRAK-GAGE failed in the latter one. A conservative assessment of the fatigue live is taken from the along the grain orientation of the one hour cycle data and no report of the higher properties across the grain will be presented here for the data. This stated conservative measurement is taken because the local stresses are higher at the reinforcement than in the bulk of the specimen. As a result of plasma exposure, an average value of 26 % ± 1.5 % fewer cycles can be calculated for the 8090 alloy across all DK ranges tested.

The surface topography of the fracture surfaces will be discussed in additional detail in a subsequent section on the results of the plane strain fracture toughness test.

**Charpy Impact Tests**

The results of the Charpy V-notched impact test are tabulated in Table VIII. The data listed in Table VIII was obtained from the Dynatup 830 load records. Figure 59 shows a typical load vs time experimental record and for the instrumented Charpy impact test. The record in Figure 59 was made from a 8090 Al-Li specimen with a T-S orientation. The crack arrester nature of the material is demonstrated by the “load shelf” located just before the point that is labeled fracture load. Figure 60 shows
the characteristic plots of the materials used in this investigation and photographs of the typical fractured test bar. Figure 60 indicates that changes in the notch depth of the 8090 Al-Li T-S specimens can shift the "load shelf" to either side of the maximum load point. The accompanying photographs in Figures 60 a-d clearly show that increases in impact energy are consumed in the generation of fracture surface area.

Review of the load traces focused on the energy required to initiate a fracture because of the inconsistency of fracture path shown in Figure 60 and the scatter produced by the many possible fracture paths in these alloys. The column in Table VIII entitled "% change" is the percent reduction of energy absorbed by the specimen before onset of fracture. The negative values in the table indicate an increase in energy required to initiate the fracture. The presence of the negative value for 8090 aluminum alloy with the L-T orientation, is believed to be caused by the preferential decomposition of the strengthening phase Al₃Li during machining and the presence of lithium hydride at the notch root [135]. The presence of the unstable lithium hydride is evidenced by the remnants of gas bubbles that can be seen on Figure 61 b). The oxide and other unknown reaction products formed as a result of machining a specimen in a water-oil solution after testing are shown in Figure 61. The effects of a trapped micro-gas bubble which burst as a result of machining are shown in Figure 61 a). Figures 62 and 63 show the fracture surfaces of several broken specimens of the 7075 composite material. The effects of the layered structure and notch orientation of the 8090 specimens are shown in Figure 64 through Figure 66.
The reduction in energy required to initiate fracture is because of the coating of the notch with oxide. This coating produces a sharper radius v-notch as shown in Figure 67 [52]. In addition to the effect of this coating, the brittle oxide produces many crack nucleation sites in the specimens that have been commercially or plasma anodized. This effect is shown in Figure 68 for the commercially coated specimen and Figure 69 for the plasma anodized specimen. The data from Table VIII is plotted on Figure 70 for the 7075+SiC MMC and Figure 71 for the 8090 Al-Li material. Any significant effect on impact toughness from the surface coating has been hidden by the change in notch root radii and the generation of additional crack nucleation sites. The surface area generated away from the primary fracture surface is shown in Figure 72. The 8090 material has demonstrated its capability of absorbing and transferring impact energy. The spalling of the oxide shown in Figure 72 a), near the point of impact of the striker site shows the wave effect of the transfer of energy [137]. Some of the energy that is absorbed during the early stages of fracture is transformed into a sub-surface crack such as shown coming to the surface in Figure 72 b). Further investigation of this observation was conducted by painting two thin layers of a brittle polymer coatings on several of the Charpy V-notched impact specimens. The stress field around the notch could then be evaluated after the impact test was completed. Examination of the SEM photomicrographs in Figure 73 indicates that paths of high stress occur around the notch. This is caused by the brittle nature of the interface between the layered structure of the 8090 alloy. Fracture surfaces were located 20 mm away from the
striker impact site under the anvil of the tester. The formation of a perpendicular branch crack away from the notch in the Charpy V-notch bar is shown in Figure 74.

**Fracture Toughness Determination**

The plane strain fracture toughness tests results include the plots of the load, displacement, and crack length data plots are contained in Appendix B for the disk-shaped compact specimens DC(T). The test values and dimensions used to determine $K_q$ are contained in Table IX. The tests conducted that met the general requirements of ASTM 399-83, ASTM 645-84, and ASTM 646-87 arc recorded on Table II with a “*”. $K_{IC}$ values for the C-R orientations were not available in the literature. The values recorded in Table II were estimated by dropping the high and low values of $K_q$ and averaging the remaining $K_q$ values for each alloy tested.

The aim of this procedure was to determine the plane strain fracture toughness of the 8090 Al-Li alloy in general accordance with the standard procedure ASTM E-399-83. The effects of ionized gas exposure on the toughness were also measured. Recent data indicates that evidence of oxygen implantation into aluminum is scarce; microvoids or small bubbles have been observed with AO fluence of $10^{18}$ atoms/cm² [138-140].

When the fatigue cracking was complete, the specimens were tested under displacement control and the $K_{IC}$ values were determined as listed in Table IX. The tests failed to demonstrated that the ionized oxygen gas
had any effect. Any suggested effect in the $K_{IC}$ data is attributed to the
detrimental influence of EDM to the damaged layers consisting of a brittle
recast layer, a hardened layer and a softer over tempered region, as well
as tensile residual stresses [125].

The surface morphology of the EDM notch in 6061 aluminum alloy
is shown in Figure 75 a). Fatigue testing and pre-cracking insured that
all of the fracture toughness specimens were pre-cracked out of the EDM
affected region [125]. The plane stress condition of the 6061 specimens
produced considerable plastic deformation in Figure 75 b). Comparison of
Figure 76 a) and b) shows evidence that the oxide restricts plastic
deformation by blocking dislocation slip at the surface. This produces the
closer packed dislocations and microvoids shown in Figure 76 b).

The micrographs of Figure 77 show the fracture surface morphology
of through the thickness intergranular delamination of typical 8090
monolithic specimen shear mechanism (through the thickness splitting).
The interface between the KRAK-GAGE and the specimen is shown in
Figure 77. The presence of the gauge adhesive tends to seal the pores in
the oxide. A coherent interface bond appears to exist between the
specimen and the gage after completion of tests. The splitting that occurs
in fractures across the thickness results in a significant increase in
toughness and accounts for the wide range of values listed in Table IX for
the 8090 monolithic specimens. Figure 78 is a high magnification
micrograph of the fracture initiation site of the A8090-08 recorded as the
highest $K_Q$ value of Table IX. The ductile intergranular delamination
shown initiated at a weak grain boundary. The fine dimpled substructure
shown in part a) of Figure 78 is also typical of the aged alloy. Figure 79 demonstrates fracture surfaces with the typical microvoids coalescence of the 8090+SiC and 1100+Al₂O₃ specimens.

**Relative Behavior of Various Alloys**

This investigation studied the properties and behavior of several different alloys and composites of aluminum. The relative behavior of these different materials in space structures is compared in this section. The addition of lithium to aluminum increases the strength, the specific modulus by some 30%, reduces the density and decreases the influence of the simulated space environment. The increase in fatigue cracking rate, the decrease in toughness and the effect on reflectively and absorption from exposure was less for the lithium containing alloys than for the 6000 and 1100 series alloys. The lithium containing alloys exhibit higher strength, higher modules, less deleterious effects of the space environment and higher ductility than the 6000 series alloys. The 6000 series does have higher strength than the 1100 series alloy.

This investigation also measured the effect of a space environment on the properties and behavior of composites of SiC particulate in Al-Li, (8090), 7075 and 1100 alloys. Composites are known to exhibit higher modules of elasticity and usually increased strength. While the modules and strength were somewhat higher than the plain matrix alloys, the toughness and ductility of the composites was so low that adequate comparisons of the effects of the space environment could not be obtained.
It was noted that the Al-Li matrix composite had the highest strength, the 7075 matrix composite was intermediate strength and the 1100 matrix composite had the lowest strength.

This thesis also provided some useful information on the effect of processing and specimen orientation on the behavior of the aluminum alloys. The properties of these alloys in the presence of severe stress concentration are not affected by oxide coatings. When these alloys may have to withstand impacts the orientation of the aluminum structure should be such that the fracture direction is across the laminates.
CHAPTER 7
CONCLUSIONS

The results indicate that the surface properties of aluminum alloys are significantly affected by the atomic oxygen environment of simulated low earth orbit in several significant areas.

1.) An exposure of five hundred twelve hours to a simulated low earth orbital environment influences the surface of the aluminum alloy sufficiently to alter the fatigue crack-growth behavior; an exposure similar to ten years in space resulted in fatigue crack growth rates that increased by 30%, ± 2%. This was measured by averaging results for different values of ΔK and crack growth lengths and verified statistically with a simple regression analysis. The 8090 Al-Li series alloys were affected 4% less than the 6000 and 1100 series alloys. The results would have been higher if thinner specimens were used.

2.) Exposure for sixty four hours to atomic oxygen is sufficient to alter the energy required for fracture initiation in Charpy V-notched impact specimens. Fracture initiation energy is reduced by 28% ± .25% when tested with the notch in the arrested position. The alloy 8090-T8771 with a T-S notch orientation has superior impact properties to all other alloys tested. Propagation energy for impact specimens was also reduced,
but the numerous potential propagation paths and the generation of surface area away from the stress concentration renders the values unreliable. Termination of the v-notch in a ductile vs brittle area of the 8090-T8771 alloy produces improved test results.

3.) One hundred twenty eight hours exposure in the ionizing gas reduces the refractive index, “n” by as much as 50% and increases the absorption coefficients, “k” by as much as 25% These changes in “n” and “k” will affect the thermal gradients of structures and solar arrays.

The results of this thesis indicated that the fracture toughness ($K_{IC}$) of the aluminum alloys was not significantly affected by exposure to atomic oxygen as encountered in space. This is because the influence of atomic oxygen occurs only at the surface of the aluminum. The fracture toughness test employed already had a fine crack initiation location that was formed by EDM with its deleterious effect on the properties at the end of the crack.
REFERENCES


[66] Findley, W.N. and Sutherland, R.L., “Comparison of fatigue tests of two plastics, three composites and an aluminum alloyu obtained on


[130] P.G. Snyder, M.C. Rost, G.H. Bu-Abboud, J.A. Wollam and S.A.


surface reaction of aluminium-lithium alloy on the porosity of welded areas”, Materials Science and engineering, AI36, pg 131-139, 1991.


**Table I**

**Thickness of Oxide Coating on Aluminum [37]**

<table>
<thead>
<tr>
<th>Description</th>
<th>Thickness</th>
</tr>
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<tbody>
<tr>
<td>Natural oxide film Al or Al-Mg alloy (formed a below 300 °C)</td>
<td>1 to 3 nm</td>
</tr>
<tr>
<td>Space Plasma Anodized [22]</td>
<td>6 to 40 nm</td>
</tr>
<tr>
<td>Natural Oxide film on pure Al (formed above 300 °C)</td>
<td>up to 30 nm</td>
</tr>
<tr>
<td>Light Plasma Anodized [24]</td>
<td>2 to 700 nm</td>
</tr>
<tr>
<td>Natural Oxide film on Al-Mg (formed above 300 °C)</td>
<td>up to 3000 nm</td>
</tr>
<tr>
<td>Normal Chemical Oxide Coating</td>
<td>2500 to 5000 nm</td>
</tr>
<tr>
<td>Normal barrier layer anodizing</td>
<td>250 to 750 nm</td>
</tr>
<tr>
<td>Normal protective anodic coating</td>
<td>5000 to 30000 nm</td>
</tr>
<tr>
<td>Hard anodic coatings</td>
<td>25000 to 150000 nm</td>
</tr>
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</table>
### Table II
**Materials Test Matrix**

#### a) Monolithic Materials

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Form</th>
<th>Temper</th>
<th>Yield (ksi)</th>
<th>UTS (ksi)</th>
<th>E (10^6 psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8090</td>
<td>Plate</td>
<td>T8771</td>
<td>L 68.5</td>
<td>75.9</td>
<td>11.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>T 64.5</td>
<td>75.0</td>
<td>11.5</td>
</tr>
<tr>
<td>8090</td>
<td>Bar</td>
<td>Aged</td>
<td>L 74.3</td>
<td>79.8</td>
<td>11.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>T 56.6</td>
<td>70.6</td>
<td>11.5</td>
</tr>
<tr>
<td>6061</td>
<td>Bar</td>
<td>T651</td>
<td>L or T 42.0</td>
<td>47.0</td>
<td>10.6</td>
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</tbody>
</table>

#### b) Metal Matrix Composite Materials

<table>
<thead>
<tr>
<th>Alloy/Form/Temper</th>
<th>Reinforcement</th>
<th>Yield (ksi)</th>
<th>UTS (ksi)</th>
<th>E (10^6 psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8090 Bar/Aged</td>
<td>12 WT.% SiC</td>
<td>75.7</td>
<td>80.6</td>
<td>13.9</td>
</tr>
<tr>
<td>7075 Bar/AS-REC.</td>
<td>15 WT.% SiC</td>
<td>86.6</td>
<td>93.7</td>
<td>13.3</td>
</tr>
<tr>
<td>1100 Bar/AS-REC.</td>
<td>10 WT.% Al2O3</td>
<td>10.8</td>
<td>20.6</td>
<td>10</td>
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</tbody>
</table>

#### c) Toughness

<table>
<thead>
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<th>Alloy</th>
<th>Orientation</th>
<th>$K_{ic}$ (ksi/in)</th>
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</thead>
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<td>8090-Plate</td>
<td>T-L</td>
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</tr>
<tr>
<td>8090-Bar</td>
<td>L-T</td>
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</tr>
<tr>
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<td>T-L</td>
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</tr>
<tr>
<td></td>
<td>ST-L</td>
<td>15.2</td>
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<tr>
<td></td>
<td>C-R</td>
<td>14.4*</td>
</tr>
<tr>
<td>8090-MMC</td>
<td>C-R</td>
<td>8.2*</td>
</tr>
<tr>
<td>7075-MMC</td>
<td>L-T</td>
<td>~19</td>
</tr>
<tr>
<td>6061-Bar</td>
<td>L-T</td>
<td>27</td>
</tr>
<tr>
<td></td>
<td>T-L</td>
<td>27</td>
</tr>
<tr>
<td></td>
<td>C-R</td>
<td>20**</td>
</tr>
<tr>
<td>1100-MMC</td>
<td>C-R</td>
<td>6.1**</td>
</tr>
</tbody>
</table>

* determined by present work
** determined by present work does not meet ASTM E-399-83 requirements
### Table III
Test Matrix Materials Major Element Composition wt% [124]

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<tr>
<th>Alloy</th>
<th>Li</th>
<th>Si</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Cr</th>
<th>Ni</th>
<th>Zn</th>
<th>Zr</th>
<th>Al</th>
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<td>3003</td>
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</tr>
<tr>
<td>7075MMC**</td>
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<td>.6</td>
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<td>.13</td>
<td></td>
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<td></td>
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</tbody>
</table>

* 12 volume % Al₂O₃ Metal Matrix Composit
** 15 volume % SiC Metal Matrix Composit
*** 10 Volume % SiC Metal Matrix Composit
### Table IV-A

**Charpy Test Specimen Dimensions**

<table>
<thead>
<tr>
<th>Specimen#</th>
<th>W N</th>
<th>W</th>
<th>L</th>
<th>N</th>
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</thead>
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<td>7075-01</td>
<td>0.394</td>
<td>0.393</td>
<td>2.163</td>
<td>0.078</td>
</tr>
<tr>
<td>7075-02</td>
<td>0.395</td>
<td>0.394</td>
<td>2.163</td>
<td>0.078</td>
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<tr>
<td>7075-03</td>
<td>0.394</td>
<td>0.394</td>
<td>2.163</td>
<td>0.079</td>
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<td>7075-04</td>
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<td>0.393</td>
<td>2.165</td>
<td>0.079</td>
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<tr>
<td>7075-05</td>
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<td>0.393</td>
<td>2.165</td>
<td>0.079</td>
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<td>0.393</td>
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<tr>
<td>7075-07</td>
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<td>0.394</td>
<td>2.162</td>
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<tr>
<td>7075-08</td>
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<td>0.395</td>
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</tr>
<tr>
<td>7075-09</td>
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<td>0.394</td>
<td>2.165</td>
<td>0.078</td>
</tr>
<tr>
<td>7075-07*</td>
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<td>0.394</td>
<td>2.165</td>
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<td>7075-08*</td>
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<td>0.393</td>
<td>2.162</td>
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</table>

All measurements are in inches.

* Measurements after commercial anodization.
### Table IV-B

Disk-shaped Compact DCT(D) Test Specimen Dimensions

<table>
<thead>
<tr>
<th>Specimen#</th>
<th>D</th>
<th>W</th>
<th>B</th>
<th>c</th>
<th>a</th>
<th>N</th>
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<tbody>
<tr>
<td>6061-01</td>
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<td>0.559</td>
<td>0.275</td>
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<tr>
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<td>0.059</td>
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<td>0.215</td>
<td>0.059</td>
</tr>
<tr>
<td>6061-05</td>
<td>0.741</td>
<td>0.542</td>
<td>0.276</td>
<td>0.140</td>
<td>0.216</td>
<td>0.058</td>
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<td>0.741</td>
<td>0.542</td>
<td>0.276</td>
<td>0.138</td>
<td>0.216</td>
<td>0.059</td>
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<tr>
<td>6061-08</td>
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<td>0.276</td>
<td>0.139</td>
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<td>0.059</td>
</tr>
<tr>
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<td>0.541</td>
<td>0.276</td>
<td>0.138</td>
<td>0.216</td>
<td>0.059</td>
</tr>
<tr>
<td>6061-10</td>
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<td>0.215</td>
<td>0.059</td>
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<tr>
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<td>0.544</td>
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<td>0.139</td>
<td>0.217</td>
<td>0.058</td>
</tr>
<tr>
<td>6061-13</td>
<td>0.746</td>
<td>0.544</td>
<td>0.278</td>
<td>0.139</td>
<td>0.217</td>
<td>0.058</td>
</tr>
<tr>
<td>6061-14</td>
<td>0.740</td>
<td>0.544</td>
<td>0.279</td>
<td>0.138</td>
<td>0.217</td>
<td>0.059</td>
</tr>
<tr>
<td>A8090-01</td>
<td>0.740</td>
<td>0.542</td>
<td>0.268</td>
<td>0.140</td>
<td>0.214</td>
<td>0.059</td>
</tr>
<tr>
<td>A8090-02</td>
<td>0.741</td>
<td>0.540</td>
<td>0.274</td>
<td>0.140</td>
<td>0.212</td>
<td>0.059</td>
</tr>
<tr>
<td>A8090-03</td>
<td>0.740</td>
<td>0.547</td>
<td>0.274</td>
<td>0.140</td>
<td>0.215</td>
<td>0.059</td>
</tr>
<tr>
<td>A8090-08</td>
<td>0.741</td>
<td>0.545</td>
<td>0.273</td>
<td>0.140</td>
<td>0.214</td>
<td>0.060</td>
</tr>
<tr>
<td>A8090-09</td>
<td>0.741</td>
<td>0.543</td>
<td>0.275</td>
<td>0.141</td>
<td>0.215</td>
<td>0.060</td>
</tr>
<tr>
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<td>0.542</td>
<td>0.275</td>
<td>0.142</td>
<td>0.214</td>
<td>0.060</td>
</tr>
<tr>
<td>A8090-11</td>
<td>0.741</td>
<td>0.540</td>
<td>0.272</td>
<td>0.140</td>
<td>0.212</td>
<td>0.059</td>
</tr>
<tr>
<td>B8090-01</td>
<td>0.742</td>
<td>0.548</td>
<td>0.277</td>
<td>0.139</td>
<td>0.216</td>
<td>0.063</td>
</tr>
<tr>
<td>B8090-03</td>
<td>0.742</td>
<td>0.545</td>
<td>0.281</td>
<td>0.139</td>
<td>0.216</td>
<td>0.062</td>
</tr>
<tr>
<td>B8090-05</td>
<td>0.742</td>
<td>0.544</td>
<td>0.279</td>
<td>0.139</td>
<td>0.216</td>
<td>0.063</td>
</tr>
<tr>
<td>B8090-06</td>
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<td>0.544</td>
<td>0.280</td>
<td>0.138</td>
<td>0.216</td>
<td>0.063</td>
</tr>
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<td>B8090-07</td>
<td>0.742</td>
<td>0.548</td>
<td>0.278</td>
<td>0.138</td>
<td>0.216</td>
<td>0.063</td>
</tr>
<tr>
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<td>0.546</td>
<td>0.278</td>
<td>0.132</td>
<td>0.226</td>
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<tr>
<td>1100-02</td>
<td>0.729</td>
<td>0.529</td>
<td>0.277</td>
<td>0.138</td>
<td>0.217</td>
<td>0.061</td>
</tr>
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<td>0.528</td>
<td>0.276</td>
<td>0.144</td>
<td>0.214</td>
<td>0.063</td>
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All measurements in inches.
### Table V
Ionized Gas Exposure Time

<table>
<thead>
<tr>
<th>Specimen #</th>
<th>A) Charpy Specimens Hours Plasma Anodized</th>
<th>B) Fracture Mechanics Specimens Hours Plasma Anodized</th>
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<td>7075-01</td>
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</tr>
<tr>
<td>7075-02</td>
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<tr>
<td>7075-09</td>
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<td>7075-07*</td>
<td>0.469</td>
<td>6061-13</td>
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<td>7075-08*</td>
<td>0.497</td>
<td>6061-14</td>
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<td>7075-09*</td>
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<td>A8090-02</td>
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<td>A8090-09</td>
</tr>
<tr>
<td>C8090-08A</td>
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<td>B8090-05</td>
</tr>
<tr>
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<td>B8090-06</td>
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<td>0</td>
<td>B8090-07</td>
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<td>B8090-08</td>
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<tr>
<td>C8090-13A</td>
<td>37</td>
<td>1100-02</td>
</tr>
<tr>
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<td>1100-03</td>
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<tr>
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<tr>
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</tr>
<tr>
<td>C8090-09A*</td>
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<td></td>
</tr>
<tr>
<td>C8090-10A*</td>
<td>4.9</td>
<td></td>
</tr>
<tr>
<td>C8090-11A*</td>
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<td>65.5</td>
<td>E8090-09</td>
</tr>
<tr>
<td>E8090-03</td>
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<td>65.5</td>
<td>E8090-12</td>
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<tr>
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<td>E8090-13</td>
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<tr>
<td>E8090-07</td>
<td>48</td>
<td>E8090-14</td>
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<tr>
<td>E8090-08</td>
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<td>E8090-15</td>
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<tr>
<td>E8090-09</td>
<td>48</td>
<td>E8090-16</td>
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* Hard Coat Anodized, 12-15% H₂SO₄, 46° F
# Table VI-A
Optical Constant "n", Refractive Index

Aluminum Alloy Optical Constant "n"
Effect of Ionized Gas Exposure

<table>
<thead>
<tr>
<th>Material</th>
<th>Al (published)</th>
<th>1 hr</th>
<th>32 hrs</th>
<th>128 hrs</th>
<th>512 hrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>6061-T651</td>
<td>1.219</td>
<td>0.476</td>
<td>0.493</td>
<td>0.798</td>
<td></td>
</tr>
<tr>
<td>8090 Al-Li</td>
<td>1.121</td>
<td>0.542</td>
<td>0.653</td>
<td>0.461</td>
<td></td>
</tr>
<tr>
<td>8090 + Sic</td>
<td>1.328</td>
<td>0.717</td>
<td>0.467</td>
<td>0.328</td>
<td></td>
</tr>
<tr>
<td>1100 + AL2O3</td>
<td></td>
<td>0.552</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Metallic Aluminum</td>
<td>1.09</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* measurements at wavelength 550 nm

Hours Plasma Anodized
Table VI-B
Optical Constant "k", Absorption Coefficient

Aluminum Alloy Optical Constant "k"
Effect of Ionized Gas Exposure

<table>
<thead>
<tr>
<th>Alloy System</th>
<th>1 hr</th>
<th>32 hrs</th>
<th>128 hrs</th>
<th>512 hrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>6061-T651</td>
<td>-4.321</td>
<td>-3.807</td>
<td>-1.593</td>
<td>-2.947</td>
</tr>
<tr>
<td>8090 Al-Li</td>
<td>-3.939</td>
<td>-2.158</td>
<td>-4.503</td>
<td>-4.514</td>
</tr>
<tr>
<td>8090 + Sic</td>
<td>-2.953</td>
<td>-3.099</td>
<td>-2.378</td>
<td>-2.703</td>
</tr>
<tr>
<td>1100 + AL2O3</td>
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<td>-1.575</td>
<td></td>
<td>-2.498</td>
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<td>Metallic Aluminum</td>
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<td></td>
<td></td>
</tr>
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</table>

Absorption Coefficient, "k"

* measurements at wavelength 550 nm
<table>
<thead>
<tr>
<th>ΔK*</th>
<th>Crack**</th>
<th>Cycles for hour exposed</th>
<th>Over Cured</th>
<th>Cured</th>
</tr>
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<tbody>
<tr>
<td></td>
<td></td>
<td>1 hr</td>
<td>256 hrs</td>
<td>Polished</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Not Polished</td>
</tr>
<tr>
<td>10-7</td>
<td>5.7-6.7</td>
<td>25920</td>
<td>14612</td>
<td>32968</td>
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<tr>
<td>7-6</td>
<td>6.7-7.7</td>
<td>59028</td>
<td>45588</td>
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<tr>
<td>6-5</td>
<td>7.7-8.7</td>
<td>223848</td>
<td>153552</td>
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<tr>
<td>5-4</td>
<td>8.7-9.7</td>
<td>692452</td>
<td>637988</td>
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</tr>
<tr>
<td>4</td>
<td>9.7-10.1</td>
<td>769259</td>
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Table VII (b), Sub-Critical Fatigue Crack Growth Behavior
8090 Al-Li

<table>
<thead>
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<th>ΔK*</th>
<th>Crack**</th>
<th>Cycles for hour exposed</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<td></td>
<td></td>
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</tr>
<tr>
<td></td>
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<td>2 hrs</td>
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<tr>
<td></td>
<td></td>
<td>128 hrs</td>
</tr>
<tr>
<td></td>
<td></td>
<td>512 hrs</td>
</tr>
</tbody>
</table>

18-15 | 5.5-6.0 | 2000 | 2040 |
10-7  | 5.5-6.5 | 11112 | 28264 | 18860 |
7-6   | 6.5-7.0 | 8724  | 14380 | 10252 |

Table VII (c), Sub-Critical Fatigue Crack Growth Behavior
8090+SiC

<table>
<thead>
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<th>ΔK*</th>
<th>Crack**</th>
<th>Cycles for hour exposed</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1 hr</td>
</tr>
<tr>
<td></td>
<td></td>
<td>128 hrs</td>
</tr>
</tbody>
</table>

5    | 5.5-5.6 | 95148  | 188372 |
5-4  | 5.6-6.6 | 67972  | 83756  |
4    | 6.6-7.0 | 42884  | 53944  |

* ΔK MPa m^1/2
** Crack Length mm
### Table VIII

**Charpy Impact Test Energy**

<table>
<thead>
<tr>
<th>Specimen #</th>
<th>Total (joules)</th>
<th>Initiation</th>
<th>Propagation</th>
<th>% Change</th>
<th>Treatment (hrs/mils)</th>
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<td>7075-01, T-L</td>
<td>1</td>
<td>0.54</td>
<td>0.46</td>
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<td>0</td>
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<tr>
<td>7075-02, T-L</td>
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<td>0.49</td>
<td>1.05</td>
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<td>0</td>
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<tr>
<td>7075-03, T-S</td>
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<td>0.95</td>
<td>0.66</td>
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<td>0</td>
</tr>
<tr>
<td>7075-04, T-S</td>
<td>1.15</td>
<td>0.91</td>
<td>0.24</td>
<td>10.88%</td>
<td>64</td>
</tr>
<tr>
<td>7075-05, T-S</td>
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<td>0.72</td>
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<td>0.459</td>
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<td>0.497</td>
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<tr>
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<td>0.51</td>
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<td>50.08</td>
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Table IX

Plane-strain Fracture Toughness Values

Disk-shaped compact specimen DC(T)

\[ K_c = \left( \frac{P}{B(W^{0.5})} \right) f \]

where all measurements are in inches &

\( f = f(a/W) \)

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<td>0.275</td>
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Sample | \( f \) | \( P \) | \( K_c \) | \( ? < a \& B \)
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<td>10.86</td>
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FIGURES
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Al$_2$O$_3$ Thickness vs Ashing Time

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**6061-T651 Optical Constant "n"**

**Effect of Ionized Gas on Index**

![Graph](image)

- 1 hr
- 32 hrs
- 128 hrs
- 512 hrs
- Al (published data)

**Figure 48a** Rotating polarizer automated ellipsometer extracted optical constants for 6061-T651 aluminum after simulated space exposure. Ellipsometric determined refractive index, "n".

**6061-T651 Optical Constant "k"**

**Effect of Ionized Gas on Coefficient**

![Graph](image)

- 1 hr
- 32 hrs
- 128 hrs
- 512 hrs
- Al (published data)

**Figure 48b** Rotating polarizer automated ellipsometer extracted optical constants for 6061-T651 aluminum after simulated space exposure. Ellipsometric determined absorption coefficient, "k".
8090 Al-Li Optical Constant "n"
Effect of Ionized Gas on Index

Figure 49a Rotating polarizer automated ellipsometer extracted optical constants for 8090 aluminum-lithium after simulated space exposure. Ellipsometric determined refractive index, "n".

8090 Al-Li Optical Constant "k"
Effect of Ionized Gas on Coefficient

Figure 49b Rotating polarizer automated ellipsometer extracted optical constants for 8090 aluminum-lithium after simulated space exposure. Ellipsometric determined absorption coefficient, "k".
8090+SiC MMC Optical Constant "n"

Effect of Ionized Gas on Index

![Graph showing the effect of ionized gas on the index of refraction for 8090+SiC MMC after simulated space exposure.

Figure 50a Rotating polarizer automated ellipsometer extracted optical constants for 8090+SiC MMC after simulated space exposure. Ellipsometric determined refractive index, “n”.

8090+SiC MMC Optical Constant "k"

Effect of Ionized Gas on Coefficient

![Graph showing the effect of ionized gas on the absorption coefficient for 8090+SiC MMC after simulated space exposure.

Figure 50b Rotating polarizer automated ellipsometer extracted optical constants for 8090+SiC MMC after simulated space exposure. Ellipsometric determined absorption coefficient, “k”.

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Charpy Impact Test
8090 Al-Li & 7075+SiC MMC

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Figure 69b Dark field micrograph of plasma anodized notch on 8090 Al-Li Charpy impact test specimen. 1500x magnification shows crack in oxide coating.
Charpy V-Notched Impact Test  
7075+SiC Aluminum MMC

![Graph showing impact energy for different specimen orientations.]

**Figure 70** Charpy impact test results for 7075+SiC MMC. Two test orientations presented and maximum error estimated.

Charpy V-Notched Impact Test  
8090-T8771 Aluminum-Lithium

![Graph showing impact energy for different specimen orientations.]

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APPENDIX A

Variable Angle Spectroscopic Ellipsometer (VASE) experimental data plots
Figure A-1. Plot of specimen 6061-01 ellipsometer experiment results: (a) Phase ($\psi$) vs. Wavelength ($\lambda$) and (b) Intensity ($\Delta$) vs. Wavelength ($\lambda$).
Figure A-2. Plot of specimen 6061-02 ellipsometer experiment results: (a) Phase (psi, Ψ) vs. Wavelength (lambda, λ) and (b) Intensity (delta, Δ) vs. Wavelength (lambda, λ).
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Figure A-4. Plot of specimen 6061-04 ellipsometer experiment results: (a) Phase ($\Psi$) vs. Wavelength ($\lambda$) and (b) Intensity ($\Delta$) vs. Wavelength ($\lambda$).
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6061 T651 Plasma Anodized 512 hours
Ellipsometric Waveform Phase Data

(a)

6061 T651 Plasma Anodized 512 hours
Ellipsometric Waveform Intensity Data

(b)

Figure A-10. Plot of specimen 6061-10 ellipsometer experiment results: (a) Phase (psi, \(\Psi\)) vs. Wavelength (lambda, \(\lambda\)) and (b) Intensity (delta, \(\Delta\)) vs. Wavelength (lambda, \(\lambda\)).
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(a) Phase (psi, Ψ) vs. Wavelength (lambda, λ) and (b) Intensity (delta, Δ) vs. Wavelength (lambda, λ).
Figure A-12. Plot of specimen A8090-03 ellipsometer experiment results: (a) Phase (psi, $\Psi$) vs. Wavelength (lambda, $\lambda$) and (b) Intensity (delta, $\Delta$) vs. Wavelength (lambda, $\lambda$).
Figure A-13. Plot of specimen A8090-04 ellipsometer experiment results: (a) Phase (ψ) vs. Wavelength (λ) and (b) Intensity (Δ) vs. Wavelength (λ).
Figure A-14. Plot of specimen A8090-05 ellipsometer experiment results: (a) Phase (psi, Ψ) vs. Wavelength (lambda, λ) and (b) Intensity (delta, Δ) vs. Wavelength (lambda, λ).
Figure A-15.  Plot of specimen A8090-06 ellipsometer experiment results:  
(a) Phase (psi, \(\Psi\)) vs. Wavelength (lambda, \(\lambda\)) and (b) Intensity (delta, \(\Delta\)) vs. Wavelength (lambda, \(\lambda\)).
**Figure A-16.** Plot of specimen A8090-07 ellipsometer experiment results: (a) Phase (ψ, Ψ) vs. Wavelength (λ, λ) and (b) Intensity (δ, Δ) vs. Wavelength (λ, λ).
Figure A-17. Plot of specimen A8090-08 ellipsometer experiment results: (a) Phase (ψ, Ψ) vs. Wavelength (λ, λ) and (b) Intensity (Δ, Δ) vs. Wavelength (λ, λ).
8090 T8771 Plasma Anodized 128 hours
Ellipsometric Waveform Phase Data

(a)

8090 T8771 Plasma Anodized 128 hours
Ellipsometric Waveform Intensity Data

(b)

Figure A-18. Plot of specimen A8090-09 ellipsometer experiment results:
(a) Phase (psi, $\Psi$) vs. Wavelength (lambda, $\lambda$) and (b) Intensity (delta, $\Delta$) vs. Wavelength (lambda,$\lambda$).
Figure A-19. Plot of specimen A8090-10 ellipsometer experiment results:
(a) Phase ($\psi$, $\Psi$) vs. Wavelength ($\lambda$, $\lambda$) and (b) Intensity (delta, $\Delta$) vs. Wavelength ($\lambda$, $\lambda$).
Figure A-20. Plot of specimen A8090-11 ellipsometer experiment results: (a) Phase ($\psi$, $\Psi$) vs. Wavelength (lambda, $\lambda$) and (b) Intensity (delta, $\Delta$) vs. Wavelength (lambda, $\lambda$).
Figure A-21. Plot of specimen B8090-01 ellipsometer experiment results: (a) Phase (\(\psi\), \(\Psi\)) vs. Wavelength (\(\lambda\)) and (b) Intensity (\(\Delta\)) vs. Wavelength (\(\lambda\)).
Figure A-22. Plot of specimen B8090-08 ellipsometer experiment results: (a) Phase ($\psi$, $\Psi$) vs. Wavelength ($\lambda$, $\lambda$) and (b) Intensity ($\Delta$) vs. Wavelength ($\lambda$, $\lambda$).
Figure A-23. Plot of specimen B8090-02 ellipsometer experiment results: (a) Phase (\(\psi\)) vs. Wavelength (\(\lambda\)) and (b) Intensity (\(\Delta\)) vs. Wavelength (\(\lambda\)).
Figure A-24. Plot of specimen B8090-03 ellipsometer experiment results:
(a) Phase (\(\psi\), \(\Psi\)) vs. Wavelength (\(\lambda\)) and (b) Intensity (\(\Delta\)) vs. Wavelength (\(\lambda\)).
Figure A-25. Plot of specimen B8090-04 ellipsometer experiment results: 
(a) Phase (psi, $\Psi$) vs. Wavelength (lambda, $\lambda$) and (b) Intensity (delta, $\Delta$) vs. Wavelength (lambda,$\lambda$).
Figure A-26. Plot of specimen B8090-05 ellipsometer experiment results: (a) Phase ($\psi$) vs. Wavelength ($\lambda$) and (b) Intensity ($\Delta$) vs. Wavelength ($\lambda$).
Figure A-27. Plot of specimen B8090-06 ellipsometer experiment results:
(a) Phase (psi, Ψ) vs. Wavelength (lambda, λ) and (b) Intensity (delta, Δ) vs. Wavelength (lambda, λ).
Figure A-28. Plot of specimen B8090-07 ellipsometer experiment results: (a) Phase ($\psi$, $\Psi$) vs. Wavelength ($\lambda$, $\lambda$) and (b) Intensity ($\Delta$) vs. Wavelength ($\lambda$, $\lambda$).
Figure A-29. Plot of specimen 1100-02 ellipsometer experiment results: (a) Phase (psi, \( \Psi \)) vs. Wavelength (lambda, \( \lambda \)) and (b) Intensity (delta, \( \Delta \)) vs. Wavelength (lambda, \( \lambda \)).
Figure A-30. Plot of specimen 1100-03 ellipsometer experiment results:
(a) Phase (\(\psi, \Psi\)) vs. Wavelength (\(\lambda, \lambda\)) and (b) Intensity (\(\Delta\)) vs. Wavelength (\(\lambda, \lambda\)).
APPENDIX B

Fracture mechanics crack growth \( \frac{da}{dN} \) data plots, data obtained by FRACTOMAT data analysis from KRAK-GAGE instrumented fracture toughness test specimens
Figure B.1. Fracture Mechanics $da/dN$ Subcritical Crack Growth and $K_c$ Toughness Experiment Records for Sample 6061-01, a) Un-optimized Output Data, b) ASTM 647 Seven Point Incremental Polynomial Technique Reduced Data.
Figure B-1. Fracture Mechanics $\frac{da}{dN}$ Subcritical Crack Growth and $K_o$ Toughness Experiment Records for Sample 6061-01, c) Expanded for Clarification.
Figure B-2. Fracture Mechanics $\frac{da}{dN}$ Subcritical Crack Growth and $K_I$ Toughness Experiment Records for Sample 6061-02, a) Un-optimized Output Data, b) ASTM 647 Seven Point Incremental Polynomial Technique Reduced Data.
Figure B-2. Fracture Mechanics $da/dN$ Subcritical Crack Growth and $K_J$ Toughness Experiment Records for Sample 6061-02, c) Expanded for Clarification, d) Load vs Displacement Record for $K_J$ Test.
Figure B-2. Fracture Mechanics $da/dN$ Subcritical Crack Growth and $K_\alpha$ Toughness Experiment Records for Sample 6061-02, e) Crack Length Determined for $K_\alpha$ Calculation by KRAK-GAGE Output.
Figure B-3. Sub-critical Crack Growth After Plasma Anodizing, No Load Shedding a) 8 hours, 6061-04, b) 16 hours, 6061-05.
Figure B-3. Sub-critical Crack Growth After Plasma Anodizing, No Load Shedding c) 32 hours, 6061-06, d) ASTM 647 Fitted Data for 6061-04.
Figure B-3. ASTM 647 Fitted Data for e) 16 hour Plasma Anodized 6061-05 and f) 32 hour Plasma Anodized 6061-06.
Figure B-4. Fracture Mechanics Data for Sample 6061-08, Plasma Anodized 128 hours. a) ASTM 647 Fitted da/dN and b) First Load Applications for $K_Q$ Determination.
Figure B-4. Fracture Mechanics Data for Sample 6061-08, Plasma Anodized 128 hours. c) Second Load Application for $K_{\text{q}}$ Determination and d) Crack Length by KRAK-GAGE Output.
Figure B-5. Fracture Mechanics \( \frac{da}{dN} \) Subcritical Crack Growth and \( K_c \) Toughness Experiment Records for Sample 6061-09, Plasma Anodized 256 hours. a) Un-optimized Output Data, b) ASTM 647 Seven Point Incremental Polynomial Technique Reduced Data.
Figure B-5. Fracture Mechanics da/dN Subcritical Crack Growth and Kₐ Toughness Experiment Records for Sample 6061-09. c) Expanded Data for Clarification.
Figure B-6. Fracture Mechanics Data for Sample 6061-10, Plasma Anodized 512 hours. a) ASTM 647 Fitted da/dN and b) Load Application Record for $K_q$ Determination.
Figure B-6. Fracture Mechanics Data for Sample 6061-10, Plasma Anodized 512 hours. c) Crack Length Determined for $K_q$ Calculation By KRAK-GAGE Output.
Figure B-7. Fracture Mechanics Data for Sample 6061-12, Plasma Anodized 512 hours, not polished. a) ASTM 647 Fitted $da/dN$ and b) Load Application Record for $K_q$ Determination.
Max Load = 365
Corresponding Crack = 7.52103

Figure B-7. Fracture Mechanics Data for Sample 6061-12, Plasma Anodized 512 hours, not polished. c) Crack Length Determined for $K_q$ Calculation By KRAK-GAGE Output.
Figure B-8. Fracture Mechanics $da/dN$ Subcritical Crack Growth and $K_I$ Toughness Experiment Records for Sample 8090-01 Al-Li. a) ASTM 647 Fitted Data, Expanded for Clarification.
Figure B-9. Fracture Mechanics Data for Sample 8090-02 Al-Li, Plasma Anodized 1 hour. a) ASTM 647 Fitted da/dN and b) Load Application Record for $K_Q$ Determination.
Figure B-6. Fracture Mechanics Data for Sample 8090-02 Al-Li, Plasma Anodized 1 hour. c) Crack Length Determined for $K_{Q}$ Calculation By KRAK-GAGE Output.
Figure B-10. Fracture Mechanics Data for Sample 8090-03 Al-Li, Plasma Anodized 2 hours. a) ASTM 647 Fitted $da/dN$ and b) Load Application Record for $K_q$ Determination.
Figure B-6. Fracture Mechanics Data for Sample 8090-03, Plasma Anodized 2 hours. c) Crack Length Determined for $K_Q$ Calculation By KRAK-GAGE Output.
Figure B-11. Fracture Mechanics Data for Sample 8090-08, Plasma Anodized 64 hours. a) ASTM 647 Fitted da/dN and b) Load Application Record for $K_q$ Determination.
Figure B-11. Fracture Mechanics Data for Sample 8090-08, Plasma Anodized 64 hours c) Crack Length Determined for $K_q$ Calculation By KRAK-GAGE Output.
Figure B-12. Fracture Mechanics $\frac{da}{dN}$ Subcritical Crack Growth and $K$ Toughness Experiment Records for Sample 8090-09 Al-Li, plasma anodized 128 hours, a) ASTM 647 Fitted Data, Expanded for Clarification.
Figure B-13. Fracture Mechanics Data for Sample 8090-10 Al-Li, Plasma Anodized 256 hours. a) ASTM 647 Fitted da/dN and b) Load Application Record for $K_q$ Determination.
Figure B-13. Fracture Mechanics Data for Sample 8090-10 Al-Li, Plasma Anodized 256 hours c) Crack Length Determined for $K_q$ Calculation By KRAKGAGE Output.
Figure B-14. Fracture Mechanics Data for Sample 8090-11 Al-Li, Plasma Anodized 512 hours. a) ASTM 647 Fitted $\frac{da}{dN}$ and b) Load Application Record for $K_D$ Determination.
Figure B-14. Fracture Mechanics Data for Sample 9090-11.1. Plasma Gases Output.
Figure B-15. Fracture Mechanics Data for Sample 8090+SiC-01, Plasma Anodized 1 hour. a) ASTM 647 Fitted da/dN and b) Load Application Record for $K_Q$ Determination.
Figure B-15. Fracture Mechanics Data for Sample 8090+SiC-01, Plasma Anodized 1 hour c) Crack Length Determined for $K_q$ Calculation By KRAK-GAGE Output.
Figure B-18. Fracture Mechanics Data for Sample 8090+SiC-03, Plasma Anodized 16 hours. a) ASTM 647 Fitted da/dN and b) Load Application Record for $K_Q$ Determination.
**Figure B-16.** Fracture Mechanics Data for Sample 8090+SiC-03, Plasma Anodized 16 hours c) Crack Length Determined for $K_\alpha$ Calculation By KRAK-GAGE Output.
Figure B-17. Fracture Mechanics Data for Sample 8090+SiC-05, Plasma Anodized 128 hours. a) ASTM 647 Fitted da/dN and b) Load Application Record for $K_q$ Determination.
Figure B-17. Fracture Mechanics Data for Sample 8090+SiC-05, Plasma Anodized 128 hours. c) Crack Length Determined for $K_Q$ Calculation by KRAK-GAGE Output.
Figure B-18. Fracture Mechanics Data for Sample 8090+SiC-08, Plasma Anodized 2 hours. a) Load Application Record for $K_a$ Determination and b) Crack Length Determined for $K_a$ Calculation By KRAK-GAGE Output.
Figure B-19. Fracture Mechanics Data for Sample 1100+Al₂O₃-02, Plasma Anodized 32 hours. a) Load Application Record for $K_C$ Determination and b) Crack Length Determined for $K_Q$ Calculation by KRAK-GAGE Output.
Figure B-20. Fracture Mechanics Data for Sample 1100+Al₂O₃-03, Plasma Anodized 512 hours.  a) ASTM 647 Fitted da/dN and b) Load Application Record for $K_q$ Determination.
Figure B-20. Fracture Mechanics Data for Sample 1100+Al₂O₃-03, Plasma Anodized 512 hours c) Crack Length Determined for $K_q$ Calculation By KRAKGAGE Output.