Nondestructive evaluation methods for determination of thermal history and mechanical properties of aluminum-lithium alloys

David J. Bracci
Iowa State University

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Nondestructive evaluation methods for
determination of thermal history
and mechanical properties of
aluminum-lithium alloys

by

David J. Bracci

A Thesis Submitted to the
Graduate Faculty in Partial Fulfillment of the
Requirements for the Degree of
MASTER OF SCIENCE

Department: Materials Science and Engineering
Major: Metallurgy

Approved:

In Charge of Major Work

For the Major Department

For the Graduate College

Iowa State University
Ames, Iowa
1987
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1. INTRODUCTION

The nondestructive determination of material properties has taken on added importance in recent years. For example, the evaluation of material properties can produce significant economic advantages through removal of defective material from the production line before value-added costs are incurred. The measurement of properties to control manufacturing processes is another case in point. For these and other reasons, it is desirable that nondestructive methods be developed for the measurement of various properties of materials of technological importance.

One specification commonly encountered pertains to the evaluation of material microstructure which is determined by particular heat treatments. For a specific microstructure, associations can then be made with the mechanical properties of the material. The scope of this investigation includes the development of nondestructive ways to characterize and sort aluminum-lithium alloys of different microstructures in ways that are compatible with a production environment. Furthermore, to explore ways that will lead to direct, nondestructive determinations of the mechanical properties of interest such as yield stress and fracture toughness.

1.1 Background

Al-Li alloys have been focused on by the aerospace industry in recent years to help reduce transportation costs by decreasing fuel consumption. Al-Li alloys are of significant commercial interest because they combine low density with high stiffness (1). An Al alloy with
additions such as copper, magnesium, and zirconium, along with a few weight percent Li, could replace presently used high strength Al alloys with reduced density of up to 15% (2) without great loss in mechanical properties. In production, the change-over from presently used alloys to Al-Li alloys could be made immediately. No new fabrication techniques and no design changes on aircraft would be necessary. Since these alloys are precipitation hardened, a precise heat treating schedule is certainly necessary to produce optimum properties. To achieve these, there are at least three steps to precipitation harden an Al alloy: solution heat treating, quenching, and aging.

During solution heat treatment, an alloy is heated from a multi-phase region to a single phase region. In order for an alloy system to be capable of precipitation hardening it is necessary that the solid solubility limit decreases with decreasing temperature (3). The temperature at which the alloy is to be solution heat treated is dependent on the alloy composition and can be critical to the outcome of the material's properties. If the alloy is heated at too low a temperature, or for an inadequate amount of time, complete dissolution of the second phase will not take place. It is necessary for the second phase to be dissolved in solution to produce the strengthening precipitates that form during aging. If solution heat treating is done at too high a temperature, eutectic melting may result (4) or dispersoid precipitates may lose their ability to pin grain boundary motion and recrystallization and growth may occur (5).

During quenching, or rapid cooling, the dissolved elements from the second phase stay in solid solution uniformly throughout the matrix.
This is a metastable state. It is necessary for the material to be cooled quickly enough to avoid precipitation of the second phase or premature precipitation of the strengthening precipitates. This could produce loss of strength and/or corrosion resistance in the finished material (4).

In the ideal aging process, small, coherent metastable strengthening precipitates form homogeneously and uniformly throughout the material. There are two terms for aging, natural aging and artificial aging. Natural aging occurs at room temperature but is very slow and does not produce the strength necessary for a high strength aluminum alloy. Artificial aging is conducted at elevated temperatures. The formation of the stable and metastable precipitates is a diffusional nucleation and growth process and thus is dependent on time and temperature (3); the higher the temperature, the greater the diffusion rate. If the aging temperature is too low or the aging time is not long enough, the alloying elements will remain "frozen" in solution and proper precipitation will not occur. At too high of an aging temperature or too long an aging time, overaging will develop. Overaging occurs when the metastable phase overcoarsens, or loses its coherency with the matrix.

Thus it can be seen that there are many variables associated with producing a precipitation hardened aluminum alloy with optimum mechanical properties. In order to assure consistent mechanical properties these processing variables must remain constant. This is sometimes difficult on a large production scale. Thus, nondestructive methods are needed to determine the condition of the microstructure immediately after production to assure that the mechanical properties meet the
requirements. In order for a nondestructive method to be relevant it must also be applicable to large scale industrial use.

1.2 Al-Li Binary Alloy

The aluminum-lithium alloy of principal interest contains three known phases which are \( \alpha \) (Al-Li solid solution), \( \delta' \) (Al\(_3\)Li coherent, metastable, ordered phase), and \( \delta \) (AlLi incoherent, equilibrium intermetallic). The phase of particular interest is the \( \delta' \) precipitate for through its presence the alloy gains its strength.

The concentration of lithium can not exceed its solid solubility limit in Al which is about 14at\% at 600°C (see Fig. 1.1). The alloy must be heated into the single phase \( \alpha \) region (solution heat treating), held, and then rapidly cooled (quenched) fast enough to avoid premature precipitation of the \( \delta' \) phase or precipitation of the \( \delta \) phase. In contrast to the \( \delta' \) phase, this \( \delta \) phase is detrimental to the mechanical properties. The formation of the \( \delta \) phase, which is heterogeneous and usually located at the grain boundaries, depletes the surrounding matrix of its Li content. Therefore, the \( \delta' \) precipitates dissolve or are unable to form, leaving a precipitate-free-zone. This zone is lacking in strengthening precipitates and thus is weakened, leaving regions of low strength throughout the material, namely at the grain boundaries. After the quench, the alloy then must be reheated to an intermediate temperature below the \( \delta' \) solvus line and held to produce the \( \delta' \) precipitate. Overall, the precipitate sequence for Al-Li binary alloys, as shown by Silcock (6), is

\[
\alpha_{SS} \rightarrow \delta'(Al_3Li) \rightarrow \delta(AlLi).
\]
Fig. 1.1 Aluminum-lithium phase diagram (7)
The addition of Li to Al, followed by an appropriate heat treatment scheme, does increase the strength but not significantly enough to satisfy the needs of a load bearing material. There must be additions of other alloying elements as well, as discussed in the following.

1.3 Al-Li-X Alloy

The exploration into an acceptable ternary Al-Li-X system is not new. Much work was done in the late '50s and early '60s. In fact, Alcoa had moderate success with its Al 2020 alloy with a nominal composition of Al-4.5%Cu-1.1%Li-0.5%Mn-0.2%Cd (weight percent). The Al 2020 alloy was used for skins on the wings and horizontal stabilizers of certain aircraft giving them a 6% weight savings (8). No corrosion and no fatigue cracking has been reported in the more than 20 years of service life of these aircraft. A disadvantage of this alloy is its low fracture toughness which eliminated its further use on other aircraft (8).

Lately however, after many years of little activity, interest in the Al-Li-X system has been rekindled. One of the alloys under development by Alcoa is the Al 2090 alloy, nominal composition given as Al-2.7%Cu-2.2%Li-0.12%Zr-0.08%Si-0.12%FeMax (9). Hopes are that this material will eventually replace Al 7075-T6X as a high strength structural aluminum alloy (10).

The precipitation scheme for the Al 2090 alloy is of course much more complex than the binary Al-Li system. A ternary composition of Al-2.7%Cu-2.2%Li at room temperature would position the alloy in a three phase region of \( \alpha, T_1, \) and \( T_2 \) (9). The precipitation sequence is thought to be

\[
\alpha_{ss} \rightarrow \delta' + T_1' + T_2' \rightarrow T_1 + T_2 (4).
\]
T₁ (Al₂CuLi) is an equilibrium phase with a hexagonal crystal structure with a=4.97Å and c=9.34Å (11). T₁' is a precursor to the T₁ phase (9). The exact stochiometry of T₁' is not known but it has possibly a composition of Al₂(Cu,Li). Similarly, T₂' is a precursor to T₂ (9). T₂' is a metastable phase that is thought to be similar to the Θ' phase (Al₂Cu), present in the Al-Cu binary system, with Li as a substitutional atom. T₂ (Al₅Li₃Cu) has a body-centered cubic structure with a lattice parameter of 13.914Å (9).

Zirconium is added to the alloy to produce dispersoids (5). The solubility of Zr in Al is very low and with the addition of a very low weight percentage of Zr, as in the Al 2090 alloy, fine homogeneous precipitates of Al₃Zr will form. These precipitates are insoluble at solution heat treatment temperatures and restrict recrystallization by retarding subgrain boundary migration and coalescence (5). Zr does not interact with Li when alloyed, therefore it does not alter the precipitation sequence. However, it has been found that alloys which contain Zr tend to age faster than those that do not (12).

1.4 Previous NDE Findings on Al Alloys

Nondestructive evaluation studies have been done on precipitation hardened Al alloys other than the Al-Li system. Primarily, those investigated were through the use of changes in ultrasonic velocity (3,13,14), ultrasonic attenuation (3,13-15), electrical conductivity (4,16-20), and hardness (3,13,14,15-19).

The Al 2014, Al 2024, and Al 2219 aluminum alloys have been investigated for changes in conductivity by Rummel (4), M. Rosen et al.
(20), and Chihoski (16-18), respectively. The alloys differ in composition but all have copper as their major strengthening element (Al 2024 also has some Mg which forms strengthening precipitates). The precipitation sequence is basically the following
\[ \alpha \rightarrow \text{Guinier-Preston zones} \rightarrow \Theta' \rightarrow \Theta' \rightarrow \Theta(\text{CuAl}_2) \] (21).

Rummel found that eddy current conductivity varied with changes in microstructure (4) for the Al 2014 alloy. Conductivity measurements were taken after quenching to determine if a proper quench was achieved. Also, it was found that the conductivity of an overaged Al 2014 specimen was directly correlated to the material's ultimate strength.

In the investigation of Al 2024, Rosen et al. found that there was an initial drop in conductivity from the as-quenched state that varied with aging temperature, see Fig. 1.2. This conductivity drop was due to formation of precipitates small enough to scatter the conduction electrons. This initial drop was followed by a period of little change in conductivity but a significant increase in hardness, as seen in Fig. 1.3. During this period, \( \Theta' \) was precipitating out. The conductivity did not show much of a change until the formation of \( \Theta' \) at which time the conductivity increased due to increasing precipitate size and less electron scatterers in the matrix. It was reported that the hardness peak, formed at the onset of overaging, did not correspond to a drop in conductivity.

Chihoski observed a similar phenomenon on the Al 2219 alloy and added an additional variable into his investigation. Along with varying the aging time and temperature he varied the quench rate from solution heat treatment. This produced specimens that before aging varied in
Fig. 1.2  Electrical conductivity versus aging time for Al 2024 (20)
Fig. 1.3  Hardness versus aging time for Al 2024 (20)
amounts of \( \Theta' \), \( \Theta' \), and \( \Theta \) precipitates. A hardness vs. conductivity plot for these specimens varying in aging time and quench rate produces an envelope of points. With one aging temperature and a variety of quench rates and aging times, an envelope of points was produced as seen in Fig. 1.4. Therefore, with such a plot, an unknown quench rate and aging time should be revealed from hardness and conductivity measurements.

Rosen et al. also investigated the Al 2219 for changes in ultrasonic velocity, ultrasonic attenuation, and hardness with various aging times and temperatures (3). They observed a maximum change of 0.6% in ultrasonic velocity with aging temperatures of 220°C and 175°C and 0.3% for 150°C. The higher aging temperatures produced a greater increase in velocity over time. The increase in velocity was due to an increased volume fraction of precipitates with a higher elastic modulus since ultrasonic velocity is related to the elastic moduli. Transducers with frequencies ranges from 6-10MHz were used.

Rosen et al. also found that hardness increases more rapidly with higher aging temperatures and reached peak hardness sooner. Loss in hardness after peaking was caused by the loss in coherency between the \( \Theta' \) precipitate and the matrix. In addition, the peak increased as the aging temperature decreased for temperatures of 150°C, 175°C, and 200°C.

The amount of energy a precipitate absorbs from an ultrasonic wave is dependent on the precipitate coherency in the matrix (22). Rosen et al. found that the formation of coherent or semicoherent precipitates and the loss of coherency of these precipitates produced plateaus and peaks in attenuation values. The peak was produced from the loss of coherency of the \( \Theta' \) precipitate. They found this attenuation peak to be associated with peak hardness.
Fig. 1.4 Hardness versus conductivity for Al 2019 as a function of aging time and temperature (18)
1.5 Objectives of This Investigation

The purpose of this study is to further investigate nondestructive methods useful to determine thermal history of Al-Li and to correlate nondestructive properties with mechanical properties. Two alloys were studied. One was a binary alloy of Al-8wt% Li and the other was Alcoa's Al 2090 alloy. The alloys were subjected to two quench rates (water quenched and air cooled), five aging temperatures (150°C, 164°C, 175°C, 200°C, and occasionally 275°C), and six aging times (0, 3, 6, 12, 24, and 33 hrs). The nondestructive measurements included eddy current impedance change, d.c. conductivity, microhardness, ultrasonic velocity, and ultrasonic attenuation. These responses were compared to the mechanical properties of yield stress and an estimate of the fracture toughness. The fracture toughness estimate was determined using a relation discussed by Dieter (23) who suggested

\[ K_{IC} = \left( \frac{2}{3E} \varepsilon_f \sigma_0 n^2 \right)^{1/2} \]  

where \( K_{IC} \equiv \text{estimated fracture toughness} \), \( E \equiv \text{the Young's modulus} \), \( \varepsilon_f \equiv \text{strain to failure} \), \( \sigma_0 \equiv \text{yield stress} \), and \( n \equiv \text{strain hardening exponent} \).
2. EXPERIMENTAL PROCEDURE AND MATERIALS

Two alloys were investigated, a binary Al-8at%Li and a commercial Al 2090. In the binary alloy, changes in the NDE and mechanical properties were due only to the contribution of Li precipitates. The changes associated with additional alloying elements, Cu and Zr, were studied on the Al 2090 alloy.

The binary alloy was produced by the Materials Preparation Center at Ames Laboratory. The Al and Li used were 99.9% pure. A methanol-dry ice solution was used to clean the Li and a sodium-hydroxide solution was used to clean the Al. Both elements were placed in a graphite crucible and quickly put under vacuum in order to minimize oxidation. Induction heating was used to melt the alloy with the graphite crucible being the susceptor. After heating to 800°C, a plug on the bottom of the graphite crucible was pulled, pouring the molten alloy into a two inch diameter, chilled copper mold.

The chemical homogeneity of the ingot obtained was excellent. Chemical analysis determined the top and bottom of the ingot to have Li concentrations of 8.04at% and 7.94at%, respectively. However, there were large differences in grain size from top to bottom due to faster cooling at the bottom and slower cooling at the top; Fig. 2.1 shows these differences. Since large grains cause a decrease in strength and ductility, and since the large variations in grain size would cause inconsistency in these properties as well, an attempt to refine the grain size was made by cold work and recrystallization. The binary ingot was homogenized at 450°C for 2.5 hours in an argon atmosphere with subsequent water quench before cold working to insure chemical homogeneity and to
Fig. 2.1a As-cast microstructure of the top of the Al-8at%Li ingot

Fig. 2.1b As-cast microstructure of the bottom of the Al-8At%Li ingot
dissolve the $\delta'$ and $\delta$ phase precipitates. The presence of such precipitates would have made cold working the ingot difficult due to increased strength and brittleness. The ingots were cold worked by rod rolling. The two inch diameter ingots were reduced to a square cross section about 1 x 1 inches to obtain 70% cold work. The samples were then held at 425°C for one hour to produce recrystallization. The recrystallized grains in the 70% cold worked sample were about 0.2mm in diameter and deemed acceptable, see Fig. 2.2.

The Al 2090 alloy was received from Alcoa in the form of a plate one half inch thick in the T8E41 condition. Chemical analysis indicated the following alloying additions: 2.2wt%Li, 2.7wt%Cu, 0.2wt%Zr, and 0.04wt%Fe.

2.1 Sample Preparations

A series of blocks were cut from the binary bar with dimensions of 1 x 1 x 0.25 inches, as well as from the 2090 plate with dimensions of 1 x 1 x 0.5 inches. These blocks were used to determine microhardness, d.c. conductivity, eddy current response, ultrasonic velocity, and attenuation. Tensile specimens were machined in the rolling direction with the dimensions as shown in Fig. 2.3 and used to determine mechanical properties such as yield stress, strain hardening exponent, and strain-to-failure.

2.2 Heat Treatment

While in the cold worked state the binary alloy blocks were solution heat treated at 430°C for 1 hour. Two cooling rates were used, a water
Fig. 2.2a Microstructure of the top of the Al-8at%Li ingot after cold work and recrystallization

Fig. 2.2b Microstructure of the bottom of the Al-8at%Li ingot after cold work and recrystallization
Fig. 2.3 Dimensions of tensile specimens used in this study.
quench and an air cool to room temperature. The water quenched specimens were cooled at about 130°C/sec and the air cooled specimens were cooled about 100°C/min as determined by thermocouples mounted to the blocks.

The specimens were aged at five temperatures (150°C, 164°C, 175°C, 200°C, and occasionally 275°C) and six aging times (0hr, 3hr, 6hr, 12hr, 24hr, and 33hr at each temperature). This selection of aging temperature and time followed previous work on binary Al-Li (24) according to which the peak hardness for a binary Al-2.8wt%Li alloy occurred after aging between 8-12 hours at 175°C. Also, an alloy of composition Al-2.3wt%Li-2.8wt%Cu-0.12wt%Zr that had been stretched about 1% before aging (25) developed peak hardness at 160°C temperature after about 15 hours.

It was expected that the selection of aging temperatures and times used in the present work included a broad enough range to cover the optimum aging conditions variations expected for commercial material. The 2090 alloy was heat treated at 540°C for 0.5 hour but otherwise in a manner similar to the binary.

Unfortunately, the furnace used in this study did not have the desired stability to maintain a constant temperature. At times temperature variations of ±5°C were noticed.

2.3 Measurements

The overall plan was to evaluate the sensitivity of several measurement techniques to changes in microstructure produced by the variety of heat treatments as given above. The NDE techniques of interest were microhardness, eddy current response, d.c. conductivity, ultrasonic velocity, and ultrasonic attenuation. The mechanical measurements were yield stress, and an estimate for the fracture
toughness given by the yield stress, work hardening exponent, and strain-to-failure (25).

2.3.1 Microhardness

The microhardness measurements were performed on a Tukon hardness machine using a diamond indenter of square pyramidal shape with an opening angle of 136° and a 3000g load. The sampling area in microhardness testing is much less than in conventional hardness testing (for example Rockwell hardness tests). Therefore, the error is slightly greater. However, the indentation left is much smaller and thus more acceptable as an NDE measurement. To insure proper correlation between hardness and the other properties, hardness measurements were taken on both the blocks and tensile specimens.

2.3.2 Eddy current

A Nortec NDT 15 eddyoscope with a 5kHz probe was used for the measurements. Such a probe has a sampling depth of about 0.5cm in Al, calculated from the skin depth formula (26)

$$\delta = \frac{1}{\sqrt{\sigma \mu f}}$$

where $\delta$ = skin depth, $\sigma$ = the material's conductivity, $\mu$ = the material's permeability, and $f$ = the probe frequency. Both the resistive ($\Delta R$) and the reactive ($\Delta X$) parts of the complex impedance were determined using digital voltmeters. The total change in impedance, $\Delta Z$, was calculated by using (27)

$$\Delta Z^2 = (\Delta R^2 + \Delta X^2)$$
2.3.3 D.C. conductivity

D.C. conductivity was determined using the Van Der Pauw method (28). This method is a four point method which gives results that are independent of the location and nature of the electrical contact. In the first step, current was passed through two adjacent contacts, A and B, and the voltage was measured across the other two, C and D. The resistance $R(AB,CD)$ was then calculated using Ohm’s law. Thereafter, current was passed through A and C, also adjacent, and the voltage measured across B and D to find resistance $R(AC,BD)$. The resistivity, $\rho$, is then given by (28)

$$
\rho = \frac{md}{\ln 2} \frac{R_{AB,CD} + R_{AC,BD}}{2} f
$$

where $d$ is the specimen thickness and $f$ is determined by

$$
cosh \left[ \frac{(R_{AB,CD}/R_{AC,BD}) - 1}{(R_{AB,CD}/R_{AC,BD}) + 1} \frac{\ln 2}{f} \right] = \frac{1}{2} \exp \frac{\ln 2}{f}
$$

2.3.4 Ultrasonic velocity measurements

For these measurements an apparatus was designed which held a half inch 10MHz immersion transducer, a column of water as couplant, and the specimen to be measured. This apparatus is shown in Fig 2.4. The immersion transducer was at the bottom of a water column an inch in diameter and directed up toward the specimen. The back surface of the specimen was not immersed in order to maximize the reflection from this surface.

Ultrasonic velocities were measured using a pulse-echo-overlap method which gives better accuracy than a simple pulse echo method.
Fig. 2.4a Top view of the apparatus used to hold specimens for the ultrasonic measurements

Fig. 2.4b Side view of the apparatus used to hold specimens for the ultrasonic measurements
Here, a 10MHz broad band transducer produces an echo train consisting of several echoes from the back surface of the sample. This echo train was displayed on an oscilloscope that was being triggered by an external source of low repetition rate. Through variation of the repetition rate, successive pulses in the echo train can be made to appear to overlap. Adjustment of the repetition rate to obtain a perfect overlap of several of the pulses in the echo train allows determination of the time between pulses and thus of the ultrasonic velocity. A schematic of the pulse-echo-overlap equipment is shown in Fig. 2.5 and the method is explained in more detail in (29).

2.3.5 Ultrasonic attenuation measurements

The attenuation coefficients were measured in the same apparatus used for the velocity measurements. A spike pulse generator produced an echo train which was displayed on an oscilloscope. The amplitude of successive pulses were fitted to the attenuation equation

\[ A = RR' A_0 \exp(-\mu 2x) \]

(2.5)

to calculate the attenuation coefficient \( \mu \). In the above equation \( A_0 \) = height of the echo of greater amplitude, \( A \) = height of the echo of lower amplitude, \( x \) = thickness of the specimen, \( R \) = reflection coefficient between water and the Al-Li alloy, and \( R' \) = reflection coefficient between air and the Al-Li alloy. \( R' \) was taken to be 1 and \( R \) was calculated from the acoustic impedances

\[ R = \frac{Z_1 - Z_2}{Z_1 + Z_2} \]

(2.6)

where \( Z_1 \) = impedance of the Al-Li alloy (equal to the velocity of sound times material density) and \( Z_2 \) = impedance of water (equal to the
Fig. 2.5  Schematic diagram for the pulse-echo-overlap technique (29)
velocity of sound times water density). R was calculated to be 0.837 for both alloys.

2.3.6 Mechanical measurements

The tensile specimens were deformed using an Instron tester at a strain rate of about $1.6 \times 10^{-4} \text{ sec}^{-1}$. From the load-elongation curves, both the 0.2% off-set yield stress, $\sigma_0$, as well as the strain hardening exponent, $n$, given by (30) $n = \frac{d \log(\sigma)}{d \log(\varepsilon)}$, were determined. The strain-to-failure was estimated from the ratio of fracture surface area to original cross sectional area as

$$\varepsilon_f = \ln \frac{A_0}{A}. \quad 2.7$$

Unfortunately, not enough material was available to determine a very important mechanical property, the fracture toughness $K_{IC}$. However, using a theory by Dieter (23), the above quantities, $\sigma_0$, $n$, and $\varepsilon_f$, should provide an estimate of the fracture toughness, given by

$$K_{IC} = \left(\frac{2}{3}\varepsilon_f \varepsilon_0 n^2\right)^{1/2}. \quad 2.8$$

Certainly, it is impossible to obtain valid $K_{IC}$ values for the materials. However, the estimated $K_{IC}$ should provide order-of-magnitude, relative values of this property.

2.3.7 Density measurements

It was not expected that there would be a significant difference in density between the different heat treated material. To satisfy this expectation, density measurements were performed. Materials of different heat treatments were weighed in air and then weighed suspended in deionized water to an accuracy of 0.0001gm. The weight of the material
in air minus its weight suspended in water is the weight of the water displaced. By knowing the density of water, the volume of the water displaced can be calculated. This volume is the same as that of the Al-Li sample and by knowing its weight the density, \( \rho \), can be calculated. The relation is

\[
\rho_{H_2O} + \text{wt.} H_2O \times \text{wt.} Al-Li = \rho_{Al-Li}.
\]

The largest change for the two alloys was 0.08% and satisfied the assumption that the density change was not significant. The density of the binary and Al 2090 alloys were measured to be 2.56gm/ml and 2.59gm/ml, respectively.
3. RESULTS

3.1 Hardness

3.1.1 Binary

Results of the hardness measurements as a function of aging and temperature are shown in Figs. 3.1-3.4. In the binary alloy the hardness increased with time due to the precipitation and coarsening of the $\delta'$ phase. The initial hardness increased most dramatically for the water quenched specimens aged at 200°C as seen in Fig. 3.1. However, it did not reach the high hardness achieved at the other aging temperatures, most likely due to the loss of coherency and low number density of the $\delta'$ precipitates. The binary alloys aged at 164°C and 175°C achieved the highest hardness values. The material aged at 275°C experienced virtually no hardness change. This indicates that there is either no second phase precipitation or a loss of coherency between the precipitates and the matrix. As mentioned above, when $\delta'$, the strengthening precipitate, over-coarsens and loses its coherency with the matrix the hardness will decrease.

Results obtained on the air cooled specimens were similar to those on water quenched specimens; however, the increase in hardness was found to be more erratic for the air cooled specimens. This is most likely due to some nucleation of precipitates during cooling. As shown in Fig. 3.2, the binary alloy aged at 164°C, 175°C, or 200°C experienced a loss of hardness due to overaging if aged for 33 hours.
Fig. 3.1 Hardness of the water quenched binary alloy as a function of aging time and temperature
Fig. 3.2 Hardness of the air cooled binary alloy as a function of aging time and temperature
Fig. 3.3 Hardness of the water quenched Al 2090 alloy as a function of aging time and temperature
Fig. 3.4 Hardness of the air cooled Al 2090 alloy as a function of aging time and temperature
3.1.2 Al 2090

The water quenched Al 2090, aged at 150°C, 164°C, and 175°C, behaved in a way similar to that of the water quenched binary during the early stands of aging, as shown in Fig. 3.3. The hardness increased for the first 6 hours of aging time reaching a first small maximum at that time. Maximum hardness was achieved after 24 hours of aging. We believe that the loss of hardness between 6 and 24 hours of aging is most likely due to the dissolution of one precipitate phase and the precipitation of another. The hardness of Al 2090 aged at 200°C behaved quite differently. There was a dramatic increase in hardness with the peak hardness after 12 hours aging, achieving values significantly above that found at the lower aging temperatures.

The air cooled Al 2090 material aged at 150°C, 164°C, and 175°C responded similarly to the water quenched material, with a few differences. The hardness increase between the air cooled material aged at these temperatures was similar to those that were water quenched. The loss of hardness between 6 and 24 hours of aging that was observed in the water quenched material was not evident. The hardness achieved at 200°C aging temperature was slightly higher than obtained at lower aging temperatures.

3.2 Eddy Current and D.C. Conductivity

Eddy current response and the d.c. conductivity were determined on the same set of alloys. Figs. 3.5 and 3.6 show ΔZ and d.c. conductivity respectively for the binary and Al 2090 water quenched alloys as a function of aging time and temperature. Eliminating aging time and
Fig. 3.5a Eddy current impedance change for the water quenched binary alloy as a function of aging time and temperature

Fig. 3.5b D.C. conductivity of the water quenched binary alloy as a function of aging time and temperature
Fig. 3.6a Eddy current impedance change for the water quenched Al 2090 alloy as a function of aging time and temperature

Fig. 3.6b D.C. conductivity of the water quenched Al 2090 alloy as a function of aging time and temperature
temperature, \( \Delta Z \) can be plotted directly against the d.c. conductivity. As shown in Figs. 3.7 and 3.8, the results of the two measurements are directly related to each other. Therefore, particular attention was paid to the eddy current measurements since they are more easily adapted for nondestructive evaluation.

3.2.1 Binary

The eddy current responses for the water quenched and air cooled binary alloy as a function of aging time and temperature are shown on Figs. 3.9 and 3.10. The magnitude of the responses increased significantly during the first 12 hours of aging time at temperatures of 200°C and below. The material aged at 275°C showed virtually no change in eddy current response. Again there was more scatter in the responses for the air cooled material.

3.2.2 Al 2090

The eddy current responses observed on the Al 2090 alloy are shown in Figs. 3.11 and 3.12. A maximum in the eddy current response for the water quenched material was observed after six hours aging as was the case for hardness measurements. In the unaged material, the response was found to be greater in the water quenched specimen than in the air cooled material. There was an initial decrease in response for the water quenched material that was aged at 150°C.

It should be noted that the eddy current response observed during aging at 200°C of both water quenched and air cooled Al 2090 differed quite significantly from that found at the lower aging temperatures.
Fig. 3.7  Eddy current impedance change versus d.c. conductivity for the water quenched binary alloy
Aging Temperature

- O 150 C
- + 164 C
- △ 175 C
- ♦ 200 C
- ★ No Aging

Fig. 3.8 Eddy current impedance change versus d.c. conductivity for the water quenched Al 2090 alloy
Fig. 3.9 Eddy current impedance change for the water quenched binary alloy as a function of aging time and temperature.
Fig. 3.10 Eddy current impedance change for the air cooled binary alloy as a function of aging time and temperature.
Fig. 3.11 Eddy current impedance change for the water quenched Al 2090 alloy as a function of aging time and temperature
Fig. 3.12 Eddy current impedance change for the air cooled Al 2090 alloy as a function of aging time and temperature
After an initial maximum at 6 hours of aging, the eddy current response returned basically to its original unaged value after 24 hours.

3.3 Ultrasonic Attenuation and Velocity

Changes of the attenuation coefficients for the binary and the commercial alloys as a function of aging time and temperature are shown in Figs. 3.13-3.15. A decrease in attenuation was observed between the as quenched material and the aged material (on average about 1db/cm). As soon as the material is aged, the attenuation stays basically the same with appreciable scatter (about 0.5db/cm) and no obvious systematic trends, regardless of aging time and temperature. The attenuation in the air cooled binary alloy did not show any systematic trend either, including the unaged material.

The ultrasonic velocity measured on the water quenched and air cooled binary alloys and the water quenched Al 2090 alloy as a function of aging time and temperature are shown in Figs. 3.16-3.18. As may be seen, the differences were found to be small with the velocity varying from 6540m/sec to 6575m/sec for the binary alloy and 6445m/sec to 6490m/sec for Al 2090. Also, the results did not show any trends that would allow to associate the velocity with the thermal history of the material.

3.4 Yield Stress

3.4.1 Binary

The yield stress as a function of aging time and temperature for all the alloys investigated is shown in Figs. 3.19-3.22. It was found that
Fig. 3.13 Attenuation coefficients for the water quenched binary alloy as a function of aging time and temperature
Fig. 3.14 Attenuation coefficients for the air cooled binary alloy as a function of aging time and temperature
Fig. 3.15 Attenuation coefficients for the water quenched Al 2090 alloy as a function of aging time and temperature
Fig. 3.16 Acoustic velocity in the water quenched binary alloy as a function of aging time and temperature.
Fig. 3.17 Acoustic velocity in the air cooled binary alloy as a function of aging time and temperature.
Fig. 3.18 Acoustic velocity in the water quenched Al 2090 alloy as a function of aging time and temperature.
Fig. 3.19 Yield stress of the water quenched binary alloy as a function of aging time and temperature
Fig. 3.20 Yield stress of the air cooled binary alloy as a function of aging time and temperature
Fig. 3.21 Yield stress of the water quenched Al 2090 alloy as a function of aging time and temperature
Fig. 3.22 Yield stress of the air cooled Al 2090 alloy as a function of aging time and temperature.
the different cooling rates from solid solution did not have a significant effect on the yield stress of the binary. The yield stress increased dramatically over the first 3 hours of aging (except for the material aged at 275°C) with much smaller changes thereafter. The yield stress increased by a factor of 4 to 5 by aging the binary alloy for longer periods (12-33 hours) at temperatures in the range 150-200°C.

3.4.2 Al 2090

Unlike for the binary, the cooling rate affected the yield stress changes in Al 2090 quite significantly. The unaged, air cooled material had a yield stress of 24ksi, while the water quenched had a yield stress of 21ksi. The overall yield stress of the material aged at 150°C, 164°C, and 175°C was greater for the air cooled material; however, at 200°C the water quenched material had a higher yield stress. The 200°C aging temperature, which produced the highest hardness, also produced high strength. The final yield stress in the water quenched material aged at 200°C was about 3.5 times that of the material in the unaged condition.

3.5 Strain-to-Failure

3.5.1 Binary

The strain-to-failure of the binary alloy as a function of time and temperature is shown in Figs. 3.23 and 3.24. The results for both the water quenched and air cooled material were very similar. The strain-to-failure decreased rapidly after aging for 3 hours and then decreased slightly with further aging. In the unaged condition the strain-to-
Fig. 3.23 Strain-to-failure for the water quenched binary alloy as a function of aging time and temperature.
Fig. 3.24 Strain-to-failure for the air cooled binary alloy as a function of aging time and temperature
failure was very large with values about 1.7 for the water quenched and 1.4 for the air cooled material.

3.5.2 Al 2090

As seen in Figs. 3.25 and 3.26, the different cooling rates from solid solution temperature had a significant effect on the strain-to-failure values. For the water quenched material it was a factor of about 5 to 6 higher than for the air cooled material.

The strain-to-failure of water quenched material aged at 164°C, 175°C, and 200°C tended to decrease with aging time from 0.27 to a level of about 0.075 after which there was no further change. However, when aged at 150°C, the strain-to-failure, increased sharply during the first 3 hours of aging, from about 0.28 to 0.48, and decreased thereafter with further aging. The air cooled Al 2090 material showed a decrease in strain-to-failure to a fairly constant value (about 0.02) with the exception of the material aged at 200°C. At 200°C the strain-to-failure of the air cooled material increased to about 0.07 in 24-33 hours after an initial decrease to about 0.03.

3.6 Strain Hardening Exponent

In order to obtain a unique, strain independent strain hardening exponent, n, a linear relationship between log true stress and log true strain is required. In general, the results obtained on the binary alloy indicated a strain independent n, except for the material unaged or aged at low aging temperatures for short times. All values obtained are plotted as a function of aging time and temperature in Figs. 3.27 and 3.28. The n values obtained on Al 2090, in contrast, did not display
Fig. 3.25 Strain-to-failure for the water quenched Al 2090 alloy as a function of aging time and temperature
Fig. 3.26 Strain-to-failure for the air cooled Al 2090 alloy as a function of aging time and temperature
Fig. 3.27 Strain hardening exponent for the water quenched binary alloy as a function of aging time and temperature.
Fig. 3.28 Strain hardening exponent for the air cooled binary alloy as a function of aging time and temperature.
this linear strain dependency. The slope near the onset of plastic yielding and near the ultimate strength averaged ±0.32 and ±0.13, respectively (see Figs. 3.29 and 3.30).
Fig. 3.29 Log true stress versus log true strain for the water quenched Al 2090 material aged for 33 hours at 175°C
Fig. 3.30  Log true stress versus log true strain for the air cooled Al 2090 material aged for 33 hours at 175°C
4. DISCUSSION

4.1 Correlation Between NDE Responses and Microstructural Changes

As shown in Figs. 3.7 and 3.8, the impedance change, $\Delta Z$, for the eddy current measurements was found to be directly proportional to the d.c. conductivity changes. In general, the d.c. conductivity is determined principally by the "defects" in the alloy. Thus, it is strongly influenced by individual point defects or, in the present case, by Li in solid solution. Therefore, as the Li in solid solution migrates to form precipitates during the aging process, the d.c. conductivity increases very rapidly (31). The increase in $\Delta Z$, which is determined by the conductivity and permeability, is therefore equally dependent on depletion of Li from solid solution. The precipitates contribute very little to $\Delta Z$ due to their relatively large dimensions as compared to the individual Li atoms. With this mechanism in mind, the eddy current changes for the alloys are examined.

4.1.1 Binary

The precipitation sequence for the binary alloy system is fairly straightforward as discussed earlier. The $\Delta Z$ is most likely due to the depletion of the Li in solid solution caused by the precipitation and coarsening of the $\delta'$ phase.

The material aged at 275°C did not experience any hardness changes, as seen in Fig. 3.1. There are two likely reasons for this. One is that overaging has occurred forming incoherent precipitates that do not contribute to the hardness of the material and the other is that the Li
stayed in solid solution and did not coalesce to form precipitates. The eddy current results favor the latter explanation. If incoherent precipitates did form, much of the Li in solid solution should have migrated to the precipitates. However, the material aged at 275°C revealed virtually no change in ΔZ indicating no change in the concentration of Li in solid solution from the as-quenched condition, seen in Fig. 3.9.

The lower hardness of the material aged at 200°C would indicate overaging due to the coarsening of the δ' phase. This is conceivable because at higher temperatures the diffusion process occurs at a faster rate and thus the rate of precipitation and growth is greater. Despite this, the final ΔZ for the material aged at 200°C was less than those aged at 150°C, 164°C, and 175°C.

An explanation for the ΔZ findings for the binary, water quenched material, aged at 200°C and 275°C may be found by considering the change in the solid solubility of Li in Al with temperature. Solid solubility of Li in Al increases with temperature. Therefore, there are conceivably two opposing driving forces to the formation of the second phase, the diffusion rate and the solid solubility. At low temperatures, say 175°C and below, the increased diffusion rate due to the increased temperature dominates over the increase in solid solubility. However, at higher temperatures, very likely above 175°C, the increase in solid solubility dominates and limits the amount of Li forming precipitates of the second phase.
4.1.2 Al 2090

Unlike in the binary system, the precipitation in Al 2090 is quite complex, and therefore interpretation of results is more difficult. However deductions can still be made from its $\Delta Z$ responses. As seen in Figs. 3.11 and 3.12, the water quenched material aged at 150°C for 3 hours and the air cooled material specimen without aging had a lower $\Delta Z$ than the water quenched material without any aging. Guinier-Preston (G.P.) zones are known to form early in the aging sequence for Al 2090 and are most likely present in the water quenched material aged for 3 hours at 150°C and the air cooled material without aging. Though Cu and Li atoms in solid solution precipitate out to form these G.P. zones (9), they are small enough, only $80\AA$ in diameter and $3-6\AA$ thick (21), to scatter the passing electrons in an electric current. Studies on other Al alloys have also revealed decreases in electrical conductivity at the onset of aging due to the presence of G.P. zones (9,20).

A $\Delta Z$ decrease with aging time was also noted for the material aged at 200°C. In this case, however, it is improbable that G.P. zones are involved since this $\Delta Z$ decrease occurs at much higher aging temperatures and longer times than the above situation. There are two possible explanations for a decrease in $\Delta Z$. One is an increase in alloying elements going into solid solution forming point defects, and the other is the formation of very small precipitates able to act effectively as point defects. The high hardness values produced by the 200°C aging temperature would not favor the former explanation. Most likely, the fine precipitates forming at this temperature produce high hardness, by hindering the motion of dislocations, and are effective as an electron
scatterer for an electric current as well. Transmission electron studies are definitely needed to explain this finding.

4.2 Hardness and Eddy Current Relationship

When the hardness is plotted against eddy current response for the binary alloys a linear relation becomes evident, as shown in Figs. 4.1 and 4.2. Similar plots for the Al 2090 alloy are shown in Figs. 4.3 and 4.4. The linear relation still holds for 150°C, 164°C, and 175°C aging temperatures and for 200°C at shorter aging times. At longer aging times, however, the observed values deviate strongly from this proportionality. Again, it is speculated that this effect is caused by the formation of very fine precipitates which are effective as electron scatterers and as strengthening precipitates, inhibiting dislocation motion.

4.3 Hardness and Yield Stress Relationship

Hardness and yield stress displayed a linear relationship for both alloys. Such plots are shown in Fig. 4.5-4.8. Thus hardness can be directly used to determine the yield stress of these two Al-Li alloys nondestructively. The relationship is not dependent on the thermal history of the material, meaning that the aging time and temperature do not have to be known to predict the yield stress from the hardness values.
Fig. 4.1 Hardness versus eddy current impedance change for the water quenched binary alloy.
Fig. 4.2  Hardness versus eddy current impedance change for the air cooled binary alloy
Fig. 4.3 Hardness versus eddy current impedance change for the water quenched Al 2090 alloy
Fig. 4.4 Hardness versus eddy current impedance change for the air cooled Al 2090 alloy
Fig. 4.5 Yield stress versus hardness for the water quenched binary alloy
Fig. 4.6 Yield stress versus hardness for the air cooled binary alloy
Fig. 4.7 Yield stress versus hardness for the water quenched Al 2090 alloy
Fig. 4.8 Yield stress versus hardness for the air cooled Al 2090 alloy
4.4 Hardness and Strain-to-Failure Relationship

Hardness is considered to be a property associated with the strength of a material and strain-to-failure is associated with its ductility (30). Hardness is obtained by local yielding of a material with little deformation and thus can be related to yield stress. However, strain-to-failure is calculated from a fractured specimen which of course has been subject to severe deformation. Since this property is obtained from material that has been exposed to extensive deformation, it is often difficult to predict failure on the basis of small or no deformation (example hardness or eddy current response). Despite this apparent difficulty, the hardness versus strain-to-failure relationship was investigated.

4.4.1 Binary

The hardness versus strain-to-failure plots for the binary alloys are shown in Figs. 4.9 and 4.10. For the water quenched alloy, hardness increases as the strain-to-failure decreases. As shown, the relationship between hardness and strain-to-failure is dependent on aging temperatures. The air cooled material shows a similar relationship between strain-to-failure and hardness which is basically independent of aging temperature.

4.4.2 Al 2090

Results obtained on Al 2090 are far more complex than those obtained on the binary. Both water quenched and air cooled Al 2090 specimens showed that the relationship between strain-to-failure and hardness was
Fig. 4.9 Strain-to-failure versus hardness for the water quenched binary alloy
Fig. 4.10 Strain-to-failure versus hardness for the air cooled binary alloy
dependent on aging temperature. This was particularly true of the water quenched material, with both the plots being shown on Figs. 4.11 and 4.12.

For material heated at higher aging temperatures, the strain-to-failure was lower for a given hardness. After an aging temperature of 150°C, the strain-to-failure at first increased with increasing hardness, and thereafter decreased with hardness. After 164°C and 175°C aging, the strain-to-failure decreased with hardness over the entire range investigated. After 200°C aging, the strain-to-failure decreased with hardness initially but at higher hardness began to increase.

The strain-to-failure of the air cooled Al 2090 material was significantly lower than that for the water quenched Al 2090. In spite of this, the strain-to-failure of the air cooled Al 2090 responded in a similar manner to aging at 164°C, 175°C, and 200°C as the water quenched material. The strain-to-failure decreased with hardness and the decrease was more severe with higher aging temperatures. The air cooled material, aged at 200°C, eventually showed an increase in strain-to-failure after its initial decrease. However, the 150°C aging temperature did not produce an initial increase in strain-to-failure with hardness at lower hardness as the water quenched material had done.

In general, the decrease in strain-to-failure with hardness appears to depend strongly on aging temperature. Therefore, hardness does not appear to be a suitable NDE method for the determination of strain-to-failure for Al 2090, unless the aging temperature is also known.
Fig. 4.11 Strain-to-failure versus hardness for the water quenched Al 2090 alloy
Fig. 4.12 Strain-to-failure versus hardness for the air cooled Al 2090 alloy
4.5 Estimated Fracture Toughness

4.5.1 Binary

The fracture toughness estimate obtained from Eqn. 2.8 as a function of time and temperature are shown in Figs. 4.13 and 4.14 for the binary alloys. It should be emphasized that these values are estimates and should be considered qualitative. However, they should reveal trends in the actual fracture toughness values.

As shown the estimated fracture toughness values quickly decreased to a level where little change had seems to occur as a function of aging time. The asymptotic value reached is about $30 \pm 10$ ksi-in. There was not much difference between the water quenched material and the air cooled material except that the unaged water quenched material and those aged for 3 and 6 hours had a higher fracture toughness estimation.

4.5.2 Al 2090

As noted in Section 3.6, the strain hardening exponent is strain dependent. Since the fracture toughness estimation is strongly dependent on the strain hardening exponent, this quantity was not calculated for the Al 2090 alloy.

4.6 Hardness and Estimated Fracture Toughness Relationship

4.6.1 Binary

There seemed to be a decreasing trend between the hardness and the estimated fracture toughness values. Plots of the hardness vs. fracture
Fig. 4.13 Estimated fracture toughness of the water quenched binary alloy as a function of aging time and temperature.
Fig. 4.14 Estimated fracture toughness of the air cooled binary alloy as a function of aging time and temperature
toughness estimations are shown in Figs. 4.15 and 4.16.

An approximate fracture toughness may be determined for the binary alloys from the hardness data. A correlation can be seen between hardness and the estimated fracture toughness which would most likely improve with actual fracture toughness values.

4.7 Ultrasonic Measurements

The observed changes in attenuation with aging time and temperature were quite small and therefore no conclusive correlations between attenuation and other mechanical properties of the materials can be deduced in the present case. The small attenuation changes may be due to the relatively long wave length, \( \lambda \), of the acoustic waves compared with the small size of the precipitates. For such a case the wave scattering of the precipitates, and thus the attenuation, is negligible. \( \lambda \) was estimated from

\[
\lambda = \frac{c}{f},
\]

where \( c \) = the acoustic velocity in the Al-Li alloy (~6500m/sec) and \( f \) = the frequency of the transducer (10MHz), to be ~0.65mm. The maximum size of the \( \delta' \) precipitates is roughly \( 0.3 \times 10^{-3} \)mm (32).

Attenuation can also be affected by the ability of point defects to pin the motion of dislocations. Their ability to do so lowers the attenuation by not allowing energy to be lost due to the friction of planes of atoms sliding on one another (33). This would explain why the water quenched material without aging, both for the binary and Al 2090, had a higher attenuation than the rest of the aged material and the air cooled material without aging, seen in Figs. 3.13-3.15. We assume that
Fig. 4.15 Estimated fracture toughness versus hardness for the water quenched binary alloy
Fig. 4.16  Estimated fracture toughness versus hardness for the air cooled binary alloy
the motion of the dislocations is relatively easy for the material in the as-quenched state due to its high attenuation. Point defects, such as Li in solid solution, are known to be attracted to dislocations (34) and dislocations can act as nucleation sites for the \( \delta' \) precipitates (32). Once the Al-Li alloy has been solution heat treated, aging will permit the migration of the Li in solid solution to the dislocations as well as precipitation of \( \delta' \) at the dislocations. Thus, dislocation motion is limited by the presence of precipitates giving rise to a lower attenuation coefficient.

No systematic changes in ultrasonic velocity were observed as one would expect due to the small volume fraction of the second phase in these Al-Li alloys. Ultrasonic velocity is known to be a function of the density and elastic modulus of the material (35). As noted in Section 2.3.7, the density change is negligible. The elastic modulus of a precipitation hardened alloy would change if a large amount of the second phase precipitated out and its elastic modulus was significantly different from that of the matrix. This is obviously not the case in the present materials.

4.8 Recommendations

The above discussions contain a fair amount of speculation on the microstructural effect in the Al-Li alloys on some of the NDE and mechanical properties determined in this study. Transmission electron microscopy is clearly needed to determine the type and volume fraction of the precipitate phases present and as they are affected by heat treatment. Of particular interest here is to determine the cause for the
increasing hardness and decreasing eddy current impedance change for Al 2090 aged at 200°C for longer times (12-33 hours) since these properties change in an unusual fashion.

This study has revealed some significant NDE and mechanical property changes as well as correlations among them. These changes occur due to varying heat treatments of two Al-Li alloys showing some undesirable scatter. In order to reduce the scatter in the results obtained, it is recommended to improve the heat treatment facilities as well as to increase the number of specimens used and also to increase the number of heat treatment conditions to obtain better statistics.
5. PRELIMINARY DATA ANALYSIS BASED ON $\Delta Z$ MEASUREMENTS

A preliminary data analysis of the eddy current impedance change, $\Delta Z$, and the d.c. conductivity was performed expanding on the discussion of Section 4.1. In this section, it was deduced that $\Delta Z$ was basically a function of the concentration of Li in solid solution. This arose through the observation that $\Delta Z$ was linearly proportional to d.c. conductivity, Figs. 3.7 and 3.8, which is known to be strongly determined by the amount of point defects, such as Li in solid solution, in the material (31). The emergence of Li into the form of precipitates has little effect on either measurement. Therefore, one can assume that this is a "diffusion-limited" precipitation process (36) in which case the precipitates are homogeneously distributed throughout the matrix. The bulk concentration decreases with time as (36)

$$\bar{c} = c_0 \exp[-(t/\tau)^n]$$  \hspace{1cm} 5.1

where $\bar{c}$ $\equiv$ the average concentration at time $t$, $c_0$ $\equiv$ the initial concentration, and $\tau$ $\equiv$ a "relaxation time", determined by the diffusion coefficient. Ham (36) found that for short times $n=1.5$ and for long times $n=1.0$. Converting the concentration $\bar{c}$ to $\Delta Z$ values, Eqn. (5.1) yields

$$\ln \frac{\Delta Z - \Delta Z_0}{\Delta Z_\infty - \Delta Z_0} \propto -(t/\tau)^n$$  \hspace{1cm} 5.2

where $\Delta Z_\infty$ $\equiv$ the impedance change when basically all Li has stopped precipitating out (the solid solubility limit has been reached), $\Delta Z_0$ $\equiv$ the initial impedance change with all Li in solid solution, and $\Delta Z$ is
impedance change at time $t$. Fig. 5.1 shows such a plot for $n=1.5$, demonstrating the expected short time diffusion behavior. Fig. 5.2, on the other hand shows a plot for $n=1.0$, demonstrating the expected long time diffusion behavior. The data used for these plots were from measurements taken early in the study. This data differ slightly from recent measurements for unknown reasons. As can be seen from Fig. 5.2, the slope at longer times is clearly temperature dependent. This temperature dependence is caused by the relaxation time $\tau$ in Eqn. 5.1, given by

$$\tau = \tau_0 \exp\left(\frac{E}{kT}\right) \quad 5.3$$

where $E$ is the activation energy of migration for Li in Al. The slopes of the temperature dependent region in Fig. 5.2 are equal to $-\tau$ for each temperature. $E$ should be able to be estimated by using Eqn. 5.3, as demonstrated by Fig. 5.3. The slope should equal $E/k$. Unfortunately, the calculated $E$, 0.25eV, is much less than the $E$ (1.23eV) found in other studies (37). Therefore, more analysis is needed to determine if eddy current impedance change is a viable method to determine the amount of Li in solid solution.

Ideally, the modeling of the impedance change for the concentration of Li in solid solution, along with the modeling of hardness for precipitate information, will reveal volume fraction and size of precipitates. If this can be achieved, it could be a direct indicator of yield stress for yield stress is known to be a function of precipitate size and volume fraction (38).
Fig. 5.1 Log of eddy current change variation versus time with $n=1.5$, representing short time diffusion behavior, for the water quenched binary alloy.
Fig. 5.2 Log of eddy current change variation versus time with $n=1.0$, representing long time diffusion behavior, for the water quenched binary alloy.
Fig. 5.3  Log of the relaxation time versus 1000/aging temperature
6. SUMMARY

The purpose of this study was to investigate nondestructive methods useful to determine the thermal history of Al-Li alloys and to correlate nondestructive properties with mechanical properties. Two alloys were studied. One was a binary alloy of Al-8at% Li and the other was Alcoa's Al 2090 alloy. The alloys were subjected to two quench rates (water quenched and air cooled), five aging temperatures (150°C, 164°C, 175°C, 200°C, and occasionally 275°C), six aging times (0, 3, 6, 12, 24, and 33 hours). The nondestructive measurements included eddy current impedance change, d.c. conductivity, microhardness, ultrasonic velocity, and ultrasonic attenuation. These responses were compared to the mechanical properties of yield stress and an estimate of the fracture toughness.

Eddy current impedance change, \(\Delta Z\), and hardness were the NDE measurements most sensitive to microstructural changes. Ultrasonic attenuation and velocity did not produce systematic changes that could be correlated the the microstructure.

\(\Delta Z\) was primarily a function of the amount of Li in solid solution since it had a linear relationship with d.c. conductivity. D.C. conductivity is known to be dependent on the amount of point sources in the material, such as Li in solid solution, which act as electron scatters. However, the Al 2090 material aged at 200°C for longer periods of time did show a decreasing trend in \(\Delta Z\) and d.c. conductivity which could not be attributed to a decrease of Li in solid solution.

The hardness had a linear relationship with \(\Delta Z\) except for Al 2090 aged at 200°C for longer periods of time. There was an increasing
hardness and decreasing ΔZ trend for the material aged for these conditions.

The hardness is a good indicator of yield stress due to their linear relationship. However, hardness is not able to predict strain-to-failure. Both of these observations might be expected since hardness and yield stress are determined from a small amount of deformation and strain-to-failure is calculated from material severely deformed, in fact fractured.

An asymptotic relationship was observed between the fracture toughness estimate and the hardness. The result contained a lot of scatter, however; true fracture toughness values are needed determine a true correlation.

There was a noticeable decrease in ultrasonic attenuation between the water quenched alloys in the as-quenched state and other heat treated material. This was most likely due to Li migrating to dislocations and precipitation of \( \delta' \) at the dislocations during the slow cool from solution heat treatment and during aging causing the attenuation to decrease. Besides this change in attenuation, there was no other systematic change that could be related to microstructural changes.

There was no systematic change in ultrasonic velocity. This was likely due to low volume concentration changes in the heat treated specimens.

Specific conclusions are as follows:

- A linear proportionality exists between d.c. conductivity and the eddy current impedance change, ΔZ.
- Eddy current impedance change is linearly proportional to hardness except for the Al 2090 alloy aged at 200°C for 12 hours and longer.
- Hardness is a strong indicator of yield stress due to their linear relationship.
- There is an asymptotic relationship between hardness and fracture toughness estimates for the binary alloy.
- Ultrasonic attenuation and velocity are not a good determination of thermal history.

A fair amount of speculation has been made on the microstructural effects in the Al-Li alloys on some of the NDE and mechanical properties determined in this study. Transmission electron microscopy is needed to determine the type and volume fraction of the precipitate phases present and as they are affected by heat treatment.
7. BIBLIOGRAPHY


7. ACKNOWLEDGMENTS

I would like to express my sincere appreciation to my major professor, Dr. Otto Buck, for his help and guidance during the course of this research project. Dr. David Jiles also has my utmost gratitude for his guidance and stimulating conversation. Thanks also go out to Dr. Poornandam Garikepati for his help. All of the technical staff were ever so helpful by sharing their knowledge and they have my gratitude. Thanks also to all of the other Ames Laboratory personnel and graduate students who made my time at Ames Laboratory so enjoyable.

My special appreciation goes to my parents, Reno and Carol, and my sister and brother, Eileen and Steve. Their love and support made it possible for me to be successful in my pursuit of a higher education. The encouragement they gave me when times seemed so unbearable gave me the strength I needed to carry on.