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RELATIONSHIPS AMONG THE METALLURGICAL CONDITION,

HARDNESS, AND THE ELECTRICAL CONDUCTIVITY

OF ALUMINUM ALLOYS

BY

RICHARD A. MUELLER _ 1939-

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129521

THESIS

submitted to the faculty of

THE UNIVERSITY OF MISSOURI AT ROLLA

in partial fulfullment of the requirements for the

Degree of

MASTER OF SCIENCE IN METALLURGICAL ENGINEERING

ROLLA, MISSOURI

T2010 84P

1967

Approved by

C. 1

del Grasunas (advisor) Donald J. B.

ABSTRACT

Electrical conductivity measurements (eddy current determined) combined with indentation hardness measurements are now being used throughout the aerospace industry for nondestructive evaluation of the metallurgical condition of commercial precipitation hardenable aluminum alloys. The review of literature and experiments with two aluminum alloys, 7178 and a 5% Zn-Al binary, have shown that skilled interpretation of hardness-conductivity data depends not only upon a qualitative understanding of the modern wave mechanical theories of electron conduction, but also upon some knowledge of the precipitation reaction kinetics. In particular, the effects of "quenched-in" vacancies and retrogression upon the reaction kinetics must be considered.

Studies of conductivity vs temperature in the range of 0 to 75° F. show that the resulting conductivity changes do not result in increased interpretative information and that Matthiessen's rule and Hansen's equation both apply. Hansen's equation relates conductivity (K) of a sample to its temperature coefficient of resistance (\ll) in the form of K = B \ll + C where B and C are constants. The values of B and C depend only upon the alloy system being considered. A practical result is that the conductivity for an unknown sample can be evaluated at any known ambient temperature and then corrected to its room temperature value by calculating the sample's coefficient of resistance using Hansen's equation. The inverse calculation could also be made.

Key words: Conductivity (Eddy Current); Resistivity: Nondestructive Testing, Aluminum Alloys.

PREFACE

In recent years, eddy current determined electrical conductivity measurements have become an invaluable tool for nondestructive testing commercial precipitation hardenable aluminum alloys for their heat treat temper. Most of the related available literature contains essentially empirical data. This thesis is, in part, a qualitative study of the theoretical aspects of conductivity testing. The extensive review of literature provides the reader with a qualitative understanding of the theory which underlies the empirical conductivity data.

The experimental portion of the thesis attempts to utilize the theory to obtain more interpretive information through conductivity testing techniques. The work was only partially successful in this regard; however, a very useful concept was developed which removes the present limitation of having to conductivity test only in the vicinity of room temperature.

It is earnestly felt that conductivity testing will, eventually, rank with indentation hardness testing as a nondestructive test technique and that this work has contributed to that goal.

The author would like to thank and acknowledge the following: Dr. A. deS. Brasunas, for his constructive criticism during the preparation of this thesis; Kaiser Aluminum Corporation, for their preparation of the high purity binary alloy used in this work; and the McDonnell Company, for the use of their equipment and facilities.

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I. INTRODUCTION

A. Statement of Problem.

Electrical conductivity measurements and its reciprocal, electrical resistivity, have been used for many years to follow second phase precipitation from supersaturated solid solutions. Any physical property which is used to investigate solid state reactions can often be used as a quality control measurement. If enough of the reaction kinetics are known and if extraneous effects upon that property are eliminated or controlled, then that property has merit as a quality control measurement. Also, if that property can be measured nondestructively, it is extremely valuable as a quality control measurement.

Only recently has electrical conductivity* measurements become a nondestructive test method. Eddy current conductivity meters are now being used in the aerospace industry to inspect for proper heat treatment tempers of certain aluminum alloys.

Most of the data used for evaluation are empirically derived for each alloy and depend upon concurrent indentation hardness tests and/or some knowledge of the thermal history of the items being evaluated. This thesis is a study of the conductivities of two aluminum alloys after various heat treatments, mostly various aging cycles, to better understand the relationships among the alloys' conductivities, indentation hardnesses, and metallurgical conditions.

^{*} Hereafter referred to as "conductivity."

B. Reasons for Selection.

The data available for heat treat interpretation utilizing eddy current conductivity and indentation hardness relationships have many deviations and anomalies in actual practice. In many cases, standard heat treatment practices are not followed. Quite often precipitation hardening heat treatment (aging) cycles are missed. Quite often they are performed at the wrong temperature. This often occurs due to erroneous alloy identification.

Many of the above are covered in the literature. They can be classed as common improper heat treatments.

However, there are many uncommon improper heat treatments which cause anomalies in the R_B^* indentation hardness, # I.A.C.S.** conductivity, and metallurgical condition relationships. Due to missing a precipitation heat treatment (aging) cycle, occasionally an alloy is subjected to a subsequent thermal process which can change the conductivity. This same thermal process would not significantly affect the alloy's conductivity if it were performed after the proper aging cycle. A paint bake operation or a heated "straightening" operation are two examples of an uncommon improper heat treatment.

Therefore, in order to gain more information on various aspects of conductivity testing, a more detailed study of the relationships

* $R_{\rm B}$ is the "B" scale of a Rockwell indentation hardness tester.

** 100% I.A.C.S. is the conductivity of the international annealed copper standard.

among the metallurgical condition, hardness, and the electrical conductivity of aluminum alloys is needed.

A. Practical Applications.

Recent literature on conductivity testing (1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11) presents many techniques and data for utilizing eddy ourrent conductivity measurements for nondestructive evaluation of many aluminum alloys as to their proper heat treat temper. The information rendered is generally in the form of graphs which plot Conductivity vs Time, Conductivity vs R_B Hardness, Conductivity vs Tensile Mechanical Properties, R_B Hardness vs Time or Tensile Mechanical Properties, etc. It is interesting from a practical standpoint that in some instances the conductivity is directly proportional to the tensile yield strength of an alloy. Three typical graphs are shown in Figs. 1A. and 2.

Following the 7075 curve on Figs. 1. and 1A., it is evident that the annealed "O" temper is characterized by a high conductivity, approximately 45 %I.A.C.S., and a low hardness, approximately R_B 20. The aluminum alloy, in the annealed temper, is a multiphase mixture. Approximately 88% by volume of the alloy is comprised of the aluminum matrix which is a dilute solid solution. The remaining alloy volume is composed of particles of intermetallic compounds which are dispersed throughout the alloy. These phases can be classified into two groups. The first group contains those phases whose elements exhibit substantially increased solid solubility in the aluminum matrix with an increase in temperature. These phases will thus dissolve in the matrix if the temperature of the alloy is raised. MgZn₂ particles comprise the bulk of this group. Also, this group comprises most of



HARDNESS VS CONDUCTIVITY FOR CERTAIN COMMERCIAL ALUMINUM ALLOYS

Fig. 1 shows a composite plot of Conductivity vs R_B Hardness for various commercial aluminum alloys during various stages of heat treatment (2). Fig. 1A shows the 7075 curve of Fig. 1 in greater detail. the remaining 12% by volume of the alloy. The other group is comprised of phases whose elements are essentially insoluble in the aluminum matrix even at elevated temperature. Elevated temperatures as used here are never high enough to induce melting of any portion of the alloy, e.g., 920° F. or lower.

Figs. 1 and 1A show the basic changes which occur in an alloy's hardness and conductivity as the alloy is heat treated to the various tempers. Fig. 1A shows the 7075 curve of Fig. 1 in more detail. The tempers shown on Figs. 1 and 1A are defined as follows:

"O" - annealed - This temper applies to the softest temper of a wrought alloy. The "O" temper is usually accomplished, for 7075, by furnace cooling from the solution heat treat temperature.

"W" - Unstable condition following solution heat treatment. This designation, because of natural aging, is specific only when the period of aging is indicated.

"As quenched" - Not a specific temper, but it can be considered for aluminum alloys to be the start of the "W" temper condition. The "as quenched" condition is then the "W" temper with no aging time. It is also called the solution anneal temper.

"T4" - Solution heat treated and naturally aged (at ambient temperature) to a substantially stable condition. 7075 alloy does not have a "T4" temper, but 2024 alloy does exhibit this temper.

"T6" - Solution heat treated and then artificially aged at a temperature above room temperature. Both 7075 and 2024 have this temper designation.

"T7" - Solution heat treated and then stabilized. The "T73"

temper is a specific T7 temper of the 7075 alloy, whereby the alloy is overaged past the peak hardness condition by a controlled aging cycle which renders the alloy relatively immune to stress corrosion cracking.

The 7075 curve in Fig. 1A. shows that after quenching from the solution heat treating temperature, which is 870° F., the alloy develops a lowered conductivity of about 33 %I.A.C.S. and a slightly higher hardness of about R_B26. The alloy in the "as quenched" condition is essentially composed of the aluminum matrix alone. The matrix is now supersaturated with elements from MgZn₂ phases and other phases whose elements were soluble in the matrix at the solution heat treating temperature. The preceding statements neglect the phases whose elements are essentially insoluble in the aluminum matrix and assume that all soluble elements are trapped in solid solution by the quenching operation.

During room temperature aging and/or during the early stages of artificial aging the conductivity decreases slightly and the R_B hardness increases significantly. Further aging produces a slight increase in conductivity with greater increases of R_B hardness. When the T6 temper is reached, typical conductivity values of 32 #I.A.C.S. and hardness values of R_B 86 or greater are achieved. Overaging increases the conductivity and decreases the hardness as the T73 temper is reached. Severe overaging decreases the hardness and increases the conductivity with both approaching their respective values in the annealed temper as a limit.

The precipitates formed during aging progress from Guinier-Preston zones to transition precipitates and to the equilibrium phases

as overaging occurs. The actual sequence and occurrence of Guinier-Preston zones, etc., varies with the alloy.

It is well to note that the conductivity progresses through a minimum during the early states of aging and the hardness progresses through a maximum during the intermediate stages of aging. For 7075 aluminum alloy, the hardness maximum occurs near the T6 temper. The T6 temper has a minimum required hardness of R_B 86 for bare sheet stock, and a corresponding conductivity range of 30.5 to 34.0 %I.A.C.S. The conductivity minimum occurs after several days of room temperature aging. This statement assumes that room temperature is the lowest aging temperature being considered. The conductivity at its minimum is about 29 %I.A.C.S., with a corresponding hardness range of about 60 to 75 RB.

Fig. 2. shows the effects of delayed quenching. As the time of delay increases, the cooling rate decreases. Also, the temperature of the alloy may fall below the solvus before the sample hits the water. The slowest quench delay curve shown corresponds to an air cool. The effects of a quench delay are best shown by considering three samples, A, B, and C. Each sample was aged for the same length of time and at the same temperature subsequent to quenching. Sample A, on the fast quench curve, i.e., normal quench delay, has a high hardness of about Rg 86 and low conductivity of about 32 %I.A.C.S. Samples B and C, respectively, each with increasing quench delays, will have higher conductivities and lower hardnesses than the A sample, even though all were aged in an identical manner. Therefore, the significant trend is that samples with longer quench delays and thus slower cooling rates



Conductivity, %I.A.C.S. Fig. 2.

HARDNESS VS CONDUCTIVITY, SCHEMATIC QUENCH DELAY CURVES FOR ALCLAD 7178 ALUMINUM ALLOY.

have lower hardnesses and higher conductivities than fast quenched samples, even though all samples have equivalent aging cycles. Thus, if the alloy is known and the aging time and temperature are both known, then samples with slow or delayed quenches can be nondestructively identified.

The conductivity vs hardness relationships shown on Figs. 1. and 1A. can be used to identify the tempers of an alloy if the alloy is known. Each temper of each alloy has a definite conductivity and hardness range combination. The various ranges overlap infrequently so that nondestructive identification of mixed alloys is possible in most instances. One could use conductivity measurements alone to identify certain tempers, but R_B measurements often help to pin point the metallurgical condition of a given sample.

One could cite many instances of common improper heat treat conditions which can be identified by using curves similar to Figs. 1, 1A, and 2, but a need still exists for more information to interpret uncommon improper heat treatments. Therefore, a basic understanding of the relationships involved is needed and is developed in the following sections.

B. The Electrical Conductivity of Alloys.

1. Empirical rules.

Of the many references concerning various aspects of the origin of electrical conductivity or resistivity, Jackson and Dunleavy's work (12) provides a good starting point for this subject. Le Chatlier and Guertler's empirical rules relating electrical conductivity, temperature coefficient of resistance (in the vicinity of room temperature), and the composition in volume per cent for the various types of binary alloy systems are summarized by Jackson and Dunleavy (12) as follows:

- Rule "1. In solid solutions (except intermetallic compounds) the electrical conductivity and temperature coefficient of resistance are lowered very rapidly by the first addition of a second element. Further additions of the second element decrease these effects proportionally. In solutions with complete solid solubility, the volume composition vs conductivity or temperature coefficient of resistance curve has a catenary shape with a minimum value.
- Rule 2. Intermetallic compound formation with variable compound composition results in a maximum conductivity at the stoichiometric composition. The curve is not linear.
- Rule 3. In two phase regions, electrical conductivity and temperature coefficient of resistance vary linearly with volumes composition, following the law of mixtures."

Rule 1. can be related to the conductivity lowering with respect to the conductivity of the annealed temper caused by the increased amounts of second component elements being trapped in solid solution by the quenching operation, as shown in Fig. 1A.

Rule 2. is not applicable to this work.

Rule 3. can be related to conductivity of an aluminum alloy in

the annealed temper. It essentially states that the total observed conductivity is the sum of the conductivities of each phase present in the alloy. The conductivity contribution of each phase to the total conductivity is directly proportional to the amount of each phase present.

In commercial aluminum alloys, the precipitated phases are generally intermetallic compounds with relatively fixed compositions. In addition, the conductivities of intermetallic compounds in aluminum alloys are quite low when compared with the conductivity of the matrix solid solution. Also, the amount of the second phases present is seldom over ten volume per cent so that the second phase contribution to the observed total conductivity is quite small. Therefore, the first assumption made is that the observed conductivity of commercial aluminum alloys can be attributed to the conductivity of the aluminum matrix alone.

Another assumption can be made regarding commercial aluminum alloys. The so called "impurity elements" are held to rather close limits and are present in essentially small fixed amounts. Generally, the impurity elements are not very soluble in the aluminum matrix with Fe being the principal exception. Often they form stable intermetallic compounds. Therefore, their conductivity contributions are either negligible or essentially constant so that their conductivity contributions to the total conductivity of the alloy will be ignored. This assumption when coupled with the prior assumption indicates that only the alloying elements which exhibit appreciable increasing solid solubility with increasing temperature produce significant conduc-

tivity changes. These elements are primarily those which are the hardening elements. Some elements are added to aluminum alloys for reasons other than hardening. These elements are generally present in small fixed amounts so that their conductivity contributions can be considered a constant and will also be ignored. An example is chromium which is added to increase the corrosion resistance in 7000 series aluminum alloys (13).

2. Modern theory.

Modern theories of electrical conduction are based upon wavemechanics (14, 15). These theories state, in essence, that the electrons move through a metallic crystalline lattice not only as a particle, but also as electron waves in an electrostatic field. The electrostatic field varies in potential, which can be related to the periodicity of the lattice and to the number and type of atoms present on any given lattice site. A good treatment of the above, the Kronig-Penney solution for Schrodinger's wave equation, has been presented by Azaroff and Brophy (14).

The practical result of the modern theory and the first assumption is that the resistivity (or conductivity) of an aluminum alloy can be related to the periodicity of the aluminum matrix lattice. A lattice which is perfectly periodical, such as a pure metal having a perfect crystal lattice at absolute zero, will allow the electron wave to travel through the lattice unimpeded. A perfect lattice, in theory, would have no resistance. The converse is also important. A perturbation in the lattice, which changes the electrostatic potential, will cause an increase in resistivity. The magnitude of the increase will be proportional to the amount and type of irregularities present. The extra resistivity due to conduction electron wave scattering through lattice interaction can be described by equations which are analogous to Bragg type X-ray diffraction equations.*

* Hereafter, this effect will be referred to as "electron scattering."

- 3. Factors affecting the conductivity of an alloy.
- a) Alloying or impurity elements.

Alloying or impurity elements present in the lattice disturb the periodicity of the lattice (12). The atoms of the alloying phases generally have a different atomic radius than the parent atoms; therefore, a lattice distortion occurs which gives rise to increased electron scattering, thus decreasing the conductivity. The other main factor which decreases the conductivity due to increased electron scattering is the changed electrostatic field about an alloying atom. A different valence atom at a lattice site has a different electrostatic potential, which changes the periodicity of the lattice at that site.

The two effects above adequately explain the observed conductivity decrease of the 7075 alloy in Fig. 1A when proceeding from the annealed temper "O" to the "as quenched" condition, i.e., approximately 46 %I.A.C.S. to 32 %I.A.C.S. respectively. The effects also help explain why the delay quenched samples B and C have higher conductivities than sample A as shown in Fig. 2. The slower cooling rates trap less alloying elements in supersaturated solid solution, thus causing less electron scattering, hence higher conductivities. The effects also indicate that as second phase precipitation occurs during aging, the conductivity should rise due to the removal of solid solution atoms from the matrix.

The above effects have been utilized in other ways, e.g., Dorn and Robertson (16) have used the resistivity changes described above to calculate the valence of aluminum atoms in their lattice and have

arrived at a valence of 2.5 electrons per metallic bond.

b) Lattice defects.

Other factors which will produce electron scattering are lattice defects, i.e., vacancies, interstitial atoms and dislocations. In each of the three, the lattice period would be altered at the site of their occurrence. Three references describing lattice defects' effects upon the conductivities of alloys are Broom (17), Simmons and Balluffi (18), and DeSorbo (19). Considering these effects, Jackson and Dunleavy (12) state:

> "The magnitude of these effects is generally small compared with that observed as a result of alloying. The maximum increase in resistivity produced by extensive hardening of these types is no more than that produced by 0.1% of an alloying additive."

Broom (17) found for copper that a change of about 5×10^{12} dislocation lines/cm² corresponds to a change in resistivity equal to about 0.02 microhm-cm. Simmons and Balluffi (18) found the change in resistivity due to 10^{-3} vacancies is equal to about 0.01 to 0.001 microhm-cm in high purity aluminum. Therefore, the contributions to an aluminum alloy's conductivity due to these types of lattice defects are so small that they can be ignored. It is important to note that these types of lattice defects, per se, are not being ignored, but only their direct conductivity contributions are being ignored. This topic is discussed in Section II, C, Special Metallurgical Factors. c) Ordering.

Other factors affecting the conductivity of alloys are the effects of long range order and short range order (12). Since long range order is not involved in this work, attention will be given to

short range order effects. Clustering, a form of short range order, is closely associated with the early stages of the precipitation hardening process in aluminum alloys and affects an alloy's conductivity.

Many references covering the formation of Guinier-Preston zones* and transition precipitates (clustering is closely associated with the two) during age hardening are available. The morphology and sequence of occurrence are still somewhat controversial for many alloys, but several aspects of the above relating to resistivity changes have been investigated. If a G.P. zone is formed in such a fashion that it distorts the lattice, then increased amounts of electron scattering will occur. That such distortion occurs in a 4[#]Cu-Al age hardenable alloy was proven by Nicholson, et al, through the use of electron microscopy (20).

Matyas (21) theoretically predicted that the lattice strain and accompanying valency effect associated with the formation of G.P. zones and other coherent precipitates would cause a change in resistivity due to electron scattering. Furthermore, the size and the number of G.P. zones, and the number, valence and type of atoms in the G.P. zones all must be considered when predicting the magnitude of the resistivity changes.

Herman, et al, (22, 23) and others (24, 25, 26) bear out Matyas' theory. Asdente (27), however, disagreed. Herman (22) used Matyas' theory to calculate the critical size of spherical G.P. zones necessary for maximum electron scattering for a 5.3 at. %Zn-Al binary alloy.

^{*} Hereafter called "G.P. zones."

Theory predicted a critical size of about 8 Å and the size found through electron microscopy was 9 Å. These sizes are in good agreement and show that clustering increases the resistivity. This is the cause for the conductivity minimum shown on Fig. 1A. Herman, et al, also states that the critical size is independent of the aging temperature. Therefore, the number of G.P. zones of critical size which are present at a given aging temperature determines the magnitude of the conductivity lowering.

Referring again to Fig. 1A., it is now possible by analogy to interpret qualitatively the conductivity minimum occurring in the earlier stages of aging. Immediately after quenching, the conductivity is lowered to about 32 \$I.A.C.S. due to the change of lattice period and minor lattice distortion of the now supersaturated solid solution. As aging begins, clusters form which grow to the critical size needed for maximum electron scattering. This tends to lower the conductivity. The matrix is now becoming more periodical due to less supersaturation, which tends to increase the conductivity. The observed conductivity is the integration of these two effects. (See Herman, Cohen and Fine (22), (page 54). Obviously, since a conductivity minimum is observed, the clustering-induced conductivity lowering is the dominant tendency during the earlier stages of aging. As aging progresses, however, the decreasing supersaturation tendency becomes dominant. Usually by this time the G.P. zones of critical size are decreased in number with the remaining G.P. zones either being too large or being transformed into a transition precipitate or even into the equilibrium phase, i.e., loss of coherency.

The review of literature and references up to this point provide adequate practical and theoretical background to interpret or explain most aspects of conductivity curves of aluminum alloys. When coupled with the well known trends between hardness changes and precipitation hardening, conductivity measurements are very useful for quality control, material processing development, and failure analysis. Rummel (28) has summarized these topics for practical use, but there are some aspects of conductivity testing which are not treated sufficiently for interpretive consideration. These are retrogression and lattice imperfection effects upon reaction kinetics and will be discussed in Section II, C, Special Metallurgical Factors.

d) Temperature effects.

Another factor which will produce conduction electron scattering is an increase in temperature (12). As the temperature rises the thermally induced fluctuations of the atoms about their lattice sites change the period of the lattice. This results in increased resistivities. This also changes the temperature coefficient of resistance, since the entire electrostatic field of the lattice is changing.

Matthiessen's rule states that for dilute solid solutions the resistivity is composed of two portions. One part is due entirely to thermal effects and the other temperature independent part is attributed to all other effects. The resistivity at absolute zero is termed the residual resistivity and is considered to be independent of temperature effects. Precise work and modern theory have shown Matthiessen's rule to be correct only over small temperature ranges

and then only as a first approximation (29). The temperature coefficient of resistivity of the residual resistivity does vary with temperature.

Starr has considered portions of the above and found that there is a definite relationship between the conductivity of a solid solution alloy and its temperature coefficient of resistance as predicted by Hansen's empirical equation (30). Hansen's equation states that $K = B^{A} + C$ where K is the conductivity, \triangleleft is the temperature coefficient of resistance, and B and C are constants.

The physical basis for Hansen's equation is shown below as derived by Starr:

"The resistance of a metal or alloy is defined as: $R = \rho l/A$ where R = resistance in Λ . ρ = resistivity in $\mu \Lambda$ -cm. l = length in cm. A = cross sectional area in sq. cm.

[Note that his term for resistivity in μ -cm is inconsistent. R should either be in μ - or ρ should be in Λ -cm.]

> Differentiating with respect to temperature and rearranging terms

(Eq. 4) $\frac{1}{R} \frac{dR}{dT} = \frac{1}{P} \frac{dP}{dT} + \frac{1}{P} \frac{dP}{dT} - \frac{1}{A} \frac{dA}{dT}$

whence

 $(Eq. 5) \qquad \frac{dR}{RdT} = \alpha = K \frac{d\rho}{dT} - a$ Where α = temperature coefficient of resistance. K = electrical conductivity $1/\rho$. a = linear coefficient of expansion. T = temperature. "Rewriting Eq. 2 Hansen's equation as $(Eq. 6) \qquad \alpha = KC_1 - C_2$ Where $C_1 = 1/B$ and $C_2 = C/B$ shows the relationship to Eq. (5) when $(dP/dT) = C_1$, and $a = C_2$. That $(dP/dT) = C_1$, a constant for a given solute is to be expected for pure metals and dilute solid solutions that obey Matthiessen's rule. Matthiessen's rule states... Thus, for any one alloy system, dP/dt is independent of K, and can be considered a constant for small changes in solute concentration in dilute solid solution alloys.

The second condition of equality of Eqs. (5) and (6) shows that the constant C_2 is simply the linear coefficient of expansion, "a". While "a" will vary slightly for different concentrations of solute in a metal solvent, the change is so small that it can be considered to be a constant and equal to the mean value for all alloys of a series. For an alloy that is heat treatable, the value of "a" is again essentially independent of the degree of heat treatment and can be considered constant."

Starr covers conductivity effects of long range order and short range order. Fig. 3 is taken from Starr's work and in reference to short range order, Starr states:

> "In Fig. 2 [Fig. 3 in this thesis], where short range ordering or clustering is depicted, the equilibrium resistance increases with decreasing temperature. When true equilibrium is attained, the resistance, (and hence resistivity), change with temperature is negative. Because the time to establish equilibrium even in the case of short range ordering or clustering increases exponentially with a decrease in temperature, equilibrium is not established at low temperatures. As a result, experimental curves as shown in Fig. 2 [Fig. 3] are obtained. That the shape of the curves of d P/ dT near room temperature is caused by the previous hightemperature heat treatment is readily shown by taking a sample having a negative $d\rho/dT$ and heating it to 100°C. and holding it for a prolonged period of time. Since the equilibrium value of resistance in all cases is higher than the experimental values, the resistance should increase during an isothermal test. However, the resistance decreases continuously during heating to 100°C. and then remains constant during holding at this low temperature. Thus the change in resistance in this temperature region during the time interval under consideration is caused by electron interaction with the lattice and not atom migration ... "



Starr's last statement is somewhat confusing. He is alluding to the conductivity minimum caused by electron scattering of G.P. zones. Clustering is a diffusion controlled reaction; therefore, atom migration does occur. The total observed conductivity is the integration of the two opposing tendencies as previously stated in Section II, page 18 of this thesis. However, Starr's work is nevertheless valid.

In reference to Fig 3 and Starr's discussion, note that in the area of room temperature, dQ/dT can be both positive and negative depending upon the prior heat treatment of the sample. Note also point "M", which represents the small area where the bulk of the experimental curves merge with the theoretical curve. If point "M" is in the vicinity of room temperature, then a possibility exists that samples having equal or nearly equal room temperature conductivities, but with different prior heat treatments, could be distinguished from one another by examining their dQ/dT curves below room temperature to see if they diverge.

If point "M" is appreciably above room temperature, then one would expect that the $d\rho/dT$ curves of the two samples would have very similar slopes. Therefore, any measurable divergence of the curves would probably occur, if at all, at very low temperatures. Thus a separation of the samples would be unlikely. The above statements assume that the $d\rho/dT$ curves are straight lines, i.e., Matthiessen's Rule is applicable over the temperature range being considered.

If Matthiessen's rule does not apply, then a possible means of separation exists even if point "M" is significantly above room

temperature. This possibility exists because Matthiessen's rule is always qualified by limiting its use to dilute solid solutions. Many of the heat treatments used for hardening aluminum alloys produce different degrees of supersaturated solid solutions. Therefore, each sample's $d\rho/dT$ curve might exhibit a different temperature dependence, thus effecting the samples' separation.

If both of the previous methods for effecting a separation of the two samples do not work, then one would deduce that point "M" is appreciably above room temperature and that Matthiessen's rule is applicable. If Hansen's equation adequately describes the behavior of the aluminum samples, then it becomes possible to measure the conductivity of a 7178 sample at some known temperature T where T is other than room temperature. Then by back-calculating using Hansen's or Starr's equation, one can deduce the temperature coefficient of resistance for the sample. This assumes, of course, that Hansen's constants, B and C, for the 7178 alloy system have been previously determined. The conductivity of the unknown sample could then be corrected to its room temperature value for determination of the sample's metallurgical condition. The inverse calculation could also be made.

Therefore, a significant portion of this thesis will be devoted to resolving the following questions:

-Is point M in Fig. 3 near room temperature for 7178 aluminum alloys? And if so, can samples with different prior thermal histories be separated?

-Do 7178 aluminum alloys follow Matthiessen's rule in all heat treatment conditions? And if they don't, can samples with similar

room temperature conductivities be separated? And if they do, does Hansen's equation adequately describe their behavior?

C. Special Metallurgical Factors.

If the thermal history is unknown, or known but is not related to a standard temper, then the kinetics of the solid state reactions occurring during precipitation hardening are very important in relating conductivity and hardness data to metallurgical changes of state. For certain heat treatments, the kinetics can be considered to be constant parameters and thus ignored. However, one can ignore these effects only if the alloy and its prior thermal history are known and the thermal history falls under the classification of a standard heat treatment or common improper heat treatment. Vacancy effects and retrogression effects are two factors which affect second phase precipitation reaction kinetics and are discussed in the following paragraphs.

1. Vacancy effects.

Herman has studied the kinetics of 5.3 at% Zn-Al binary alloy during G.P. zone formation (22). He found that early in the aging process the kinetics of G.P. zone formation are dominated by the type and number of vacancies present in the lattice. If the vacancies aid diffusion of the Zn atoms, then the increased vacancies present increase the number of growing G.P. zones of critical size for maximum conductivity lowering early in the aging process. A higher than normal solution heat treating temperature creates the added number of vacancies needed. A lower than normal solution heat treating temperature has the opposite effect. All temperatures are above the solvus, so the effect was not one of changing supersaturation of the matrix.

Herman also found that a much higher solution heat treat temper-

ature produced a reversal in the number of growing G.P. zones. He speculated that this was caused by some vacancies going to permanent sinks, possibly a micro-pore, which effectively reduced the number of vacancies available for aiding Zn diffusion.

It has been found that 7178 bare aluminum alloys have lower conductivities both in the 75°F. four day "W" temper and the T6 temper subsequent to solution heat treating at 920°F., when compared with 7178 samples in the two tempers after solution heat treating at the normal 870°F. solution heat treat temperature (31). This is in agreement with Herman's findings. However, there is the possibility that the higher temperature for solution heat treatment of the 7178 alloy merely reflects more matrix supersaturation, since the solvus of the commercial alloy was not known. However, the "as-quenched" conductivity of samples heat treated at 920°F. is not lower than the "asquenched" conductivity of samples heat treated at 870°F., which seems to preclude different degrees of supersaturation. This effect should not be important late in the aging process, because Herman found that vacancy concentration effects were not important after over-aging. Therefore, the solution heat treat temperature is a variable to consider when interpreting hardness vs conductivity data early in the aging process because of different reaction kinetics attributable to different vacancy concentrations in the matrix.
2. Retrogression effects.

The topics of coherent nucleation and growth, coarsening and retrogression of second phase precipitates in a parent single phase matrix are all interrelated phenomena which are still the subject of much research. Cottrell (15) covers these topics quite well.

Without getting deeply involved in thermodynamic considerations, retrogression phenomena can be briefly defined or explained in the following manner: Consider an alloy which has been solution heat treated and aged at a given temperature, T_1 , for a given length of time. Assume that this heat treat cycle has produced a coherent second phase precipitate with a given mean particle size and given distribution. If this alloy is then subjected to a <u>higher</u> aging temperature, T_2 , one would expect some changes to occur with respect to the precipitate's size, shape and distribution. Since the original precipitate is coherent, true equilibrium is not established. The higher aging temperature would increase diffusion rates so that one would expect solid solution atoms to diffuse towards the precipitate causing it to grow, which would lower the free energy of the matrix. If this occurs then the precipitate would undergo coarsening.

However, in many instances the particles do not grow, but dissolve into the matrix. If the alloy is held at T_2 , the now dissolved atoms may precipitate as a coherent particle, but with a larger size and different distribution. The new precipitate may even be the noncoherent equilibrium phase. If the original precipitate dissolves, then this phenomenon is known as retrogression, restoration or reversion.

Since some second phase precipitates dissolve and others grow, i.e., retrogress and coarsen respectively, one must be able to explain why some grow and others do not. This is best explained through the concept of a critical particle size which is needed before growth can occur.

Therefore, consider a small coherent precipitate. This precipitate would have a high surface energy and a high strain energy, but a low chemical free energy, i.e., a low volume free energy. If a solid solution atom from the matrix were to attach itself to this particle, the free energy increase because of the added surface and strain energy would probably not be offset by an equal or greater free energy decrease of the matrix; therefore, an increase in the system free energy would result. Thus, the growth of the particle is not favored from a thermodynamic standpoint. In fact, thermodynamic considerations dictate that the particle should dissolve.

If the particle does dissolve, then its atoms would be free to diffuse to another portion of the alloy where a different precipitated second phase particle may exist. If this precipitate is a much larger particle, it would have more volume free energy than surface or strain energy. Therefore, if a solid solution atom were to attach itself to this new larger particle, then the volume free energy lowering would be greater than the surface and strain energy increases. This would result in a free energy decrease for the system, thus representing a thermodynamically favored chain of events.

Thus, if the size of the original precipitate was smaller than the critical size associated with T_2 , then retrogression would occur.

If the opposite were true, then coarsening would occur.

The situation is not as simple as stated since the discussion did not take into account the shape of the particle nor the quantitative degree of coherency, nor preferred nucleation sites such as grain boundaries, and many other important considerations. The net result is that if retrogression occurs and the alloy is held at T_2 so that a new precipitate is formed which has a larger mean size, and different distribution, then changes should occur in the alloy's hardness and conductivity.

For example, a larger sized and less finely distributed precipitate should offer less dislocation impediments, hence a lower hardness. The larger particle size is probably associated with a less supersaturated matrix, hence a higher conductivity should be apparent due to less electron scattering.

If the new precipitate produced higher coherency stresses in the matrix with respect to the original precipitate, a higher hardness and a changed conductivity might result. This condition often produces double or even triple hardness peaks on hardness versus aging time curves.

If the alloy were held at T_2 for the proper length of time needed for retrogression to occur, but for precipitation not to occur, then the alloy could revert back to a partial solution annealed temper.

This section's topics, if treated fully, could very well become a major subject of investigation in itself. Furthermore, any experiments in this area would require experimental techniques to separate the various conductivity contributions to the total conductivity and also

necessitate a knowledge of particle sizes, shapes and distributions. Therefore, one must recognize that retrogression effects are to be expected and should be considered when interpreting hardness vs conductivity data.

III. DISCUSSION

A. General Aspects.

It was decided to work with two alloys. 7178 aluminum alloy was chosen because it represents a commercial alloy in extensive use throughout the aerospace industry. Also, it is typical of the 7000 series alloys. Some limited work was done with a high purity 5% Zn-Al binary alloy.

The commercial 7178 alloy was obtained in 0.070 inch thick bare sheet stock and sheared into one by two inch samples. All samples were taken from the same sheet to eliminate, as far as possible, chemical differences from sample to sample. The binary alloy was contributed as sheet by the Kaiser Aluminum Corporation and was sheared into similar sized samples. A spectrochemical check analysis of each alloy is given below. Kaiser Aluminum Corporation analysis for the binary alloy is also given.

Element	Spectrochemistry (%)		Kaiser Analysis (%)	
	7178	Zn-Al Binary	Zn-Al Binary	
Si	0.12	0.01	0.003	
Mn	Trace	N.D.*	N.D.	
Cu	1.82	0.04	0.002	
Mg	2.82	N.D.	<0.01	
Zn	6.5	4.75	4.97	
Cr	0.22	N.D.	N.D.	
Fe	0.14	N.D.	0.002	
Others	0.05	N.D.	<0.001	
AL	Bal.	Bal.	Bal.	

* - Not Determined.

In order to evaluate the relationships previously discussed, it was necessary to produce several series of samples all with system-

atically varying but known heat treatments. Since quench delays and common improper heat treatments are relatively straightforward in interpretation, it was decided to concentrate on different aging cycles. Samples were heat treated as described in the following sections.

B. Basic Procedures and Equipment.

All solution heat treatments were performed in commercial aluminum heat treat nitrate salt baths operating to $\pm 10^{\circ}$ F. of the set temperature. All quenching was done into cold water with the delay quench times being about 3 seconds. All aging treatments above room temperature were performed in a recirculating air furnace with the working zone at $\pm 8^{\circ}$ F. from the set temperature. All specimens were inserted into the furnace operating at the set temperature and were air cooled after aging. A calibrated chromel-alumel thermocouple with a Rubicon potentiometer were used to substantiate all aging temperatures. All room temperature aging was at 75° F. $\pm 5^{\circ}$ F.

All aging times for samples aged above room temperature were measured from the time they were inserted into the furnace until they were removed. Aging times for room temperature samples were measured from the time of quenching until the time that a given measurement was taken. The precision of aging times was: \pm one second for times stated in seconds; \pm five seconds for times stated in minutes up to 60 minutes; and \pm two minutes for all other units of time. One sample which had a copper-constantan thermocouple imbedded in a hole drilled into the side of a sample was used to determine the length of time needed to achieve a temperature of 250°F. The sample reached 250°F. 210 seconds after insertion into the furnace. Therefore, it is estimated that each sample was at its aging temperature 255 seconds after insertion into the furnace, assuming Newton's law of heating applies.

All hardness measurements were the average of two indentations. The hardness tests were performed on a calibrated Rockwell Twin Tester.

The conductivity measurements were made using a commercial eddy current device, called a Conductivity Meter as shown on Illustration 1. It is an Fm-100 meter manufactured by the Magna-Flux Corp. The principle of operation is as follows: A probe, which is an induction coil representing one arm of an impedance bridge, is placed on the material to be measured. Actually, the coil is receased about .030" in the probe housing and the housing contacts the material. An alternating current in the inductor creates a time varying magnetic field which induces eddy currents in the material being tested. The mutual inductance between the coil and the sample establishes a response voltage in the coil. This voltage may be related to the eddy currents in the sample material by Lenz law. The net result is that the change of impedance of the test coil and the response voltage is proportional to the conductivity of the material. The changed impedance of the coil upsets the impedance bridge circuit. The bridge is rebalanced by rotating a variable capacitor. A microammeter is provided as a null point indicator. The variable capacitor is connected to a large dial which is calibrated directly in \$I.A.C.S. There are, of course, power supply circuits and other circuits which improve the stability of the system. The unit is calibrated before each use with known conductivity standards supplied by the manufacturer.

The precision of the calibration standards are $\frac{1}{2}$ 3% from N.B.S. standards. The manufacturer, however, claims that the instrument is capable of $\frac{1}{2}$ $l_2^{\frac{1}{2}}$ % of the indicated value on the dial; therefore, the total precision of the readings are doubtful on an absolute basis. However, since the data in this thesis was obtained using one machine



ILLUSTRATION 1. - CONDUCTIVITY METERS. The larger unit shown is the FM-l00 Meter. The portable unit shown is the FM l20 Meter. The probes of both units are to the right of the FM l20 Meter. and one set of calibration standards, relative measurements should be precise to about $l_2^{\frac{1}{2}}$ % of the indicated value. A conductivity of 40.0% will then be 40.0 $\stackrel{+}{-}$ 0.6% I.A.C.S. relative.

C. 7178 Heat Treatments.

All samples were solution heat treated at 870° F. and water quenched. Groups of samples were then aged at 75° , 250° , 300° , 350° , 400° , and 450° F. The hardness and conductivity of each sample were obtained. The results are shown in Tables I and II. The two tables also show the length of time each sample was aged. The samples aged at elevated temperatures were permitted to age at room temperature for at least one week before artificial aging commenced. The one week of natural aging produced a common starting point condition which is essentially reproducible for all samples subsequently artificially aged. This common starting condition is denoted on all hardness vs conductivity figures as "R.T.". The change of hardness and conductivity associated with the natural aging at room temperature, 75° F., is shown on Fig. 4.

Another group of samples was aged to the T6 temper, (24 hours of aging at 250° F. after solution heat treating), and then subsequently re-aged at 450° , 400° , and 350° F. These samples have code numbers prefixed by a TC, TB, and TA, respectively. The hardness and conductivity of each sample were obtained. The code numbers and results are shown in Table III. The re-aging times are also given.

The information contained in Tables I through III is best presented as hardness vs conductivity graphs, as shown in Figs. 5 and 6 and Figs. 9, 10, 11 and 12, with the latter figures appearing in the appendix. Fig. 5 is a composite of Figs. 4, 6, 9, 10, 11 and 12. Aging time or re-aging time is not shown on these graphs, but can be mentally superimposed on the graphs by remembering that samples having





higher conductivities have longer aging times for any given temperature. Also for any given conductivity, samples aged at lower temperatures have longer aging times.

As the samples age at room temperature, the conductivity approaches a minimum value and the hardness increases as clustering, i.e., second phase precipitation, occurs. The reasons have been covered in Section II. After about four days of room temperature aging, the rate of G.P. zone formation has slowed so that for all practical purposes it can be considered zero, (See Fig. 4). Subsequent aging then produces the curves in Figs. 6, 9, 10, 11, and 12.

The following list shows the approximate conductivity and hardness associated with the hardness peak attained at each aging temperature after direct aging from the common R.T. starting condition.

Aging	Temperature °F	Conductivity %I.A.C.S.	Peak Hardness ^R B
	250	32.0	94
	300	33.5	93
	350	35.0	90
	400	36.0	88
	450	35.0	87

The above values are taken from Fig. 5. This figure represents a family of aging curves. Note that one sample aged at 150°F. is consistent in its position with its imaginary curve of the family of curves.

Fig. 5 shows the following trends:

As the aging temperature is decreased

- the maximum hardness obtainable is increased,

- the time needed to attain the maximum hardness is increased,

- the hardness maximum generally occurs at decreased conduc-

tivities.

As the aging time and temperature increase

- conductivity approaches, as a limit, its value in the annealed temper. This value is about 46 %I.A.C.S.

Note that the 450° E. hardness peak occurs at lower conductivity values than what is consistent with other temperature hardness peaks. This is probably due to resolution of the second phase elements in the aluminum matrix at 450° F. and subsequent entrapment in solid solution during the air cool to room temperature.

Referring to the curves in Fig. 6 where the samples were aged directly at 450° F. from the starting R.T. condition and the other group of samples was re-aged from the T6 condition at 450° F., several trends are evident. When the samples are direct aged from the room temperature starting condition, the samples at first drop slightly in hardness and gain slightly in conductivity, e.g., R_B 80 to R_B 74 and 27% to 29 %I.A.C.S. Samples Dll, Dl and D2 produced this effect. This effect is also evident in Figs. 9, 10, ll and l2.

Again referring to Fig. 6, one can see that the samples aged at 450° F. from the T6 temper undergo a similar small drop in hardness with a very small change in conductivity from the values of the T6 temper, e. g., R_B 92 to R_B 90 and 30.5% to 32.8% I.A.C.S. Samples TC1, TC2, and TC11 produced the hardness decrease. This decrease on re-aging is also evident in Figs. 9 and 10. However, a slight recovery of the hardness is shown as aging progresses at the lower aging temperatures.

This behavior can be attributed to retrogression effects followed



by reprecipitation of phases which are stable at the new aging temperature as discussed in the Review of Literature. Herman demonstrates retrogression effects in his work with a 5% At. Zn-Al alloy (22). The net result is that hardness-conductivity data interpretation is very difficult if retrogression effects occur due to uncommon heat treatments. The retrogression effects associated with common improper heat treatments, however, are predictable and the data rendered interpretable in most cases.

D. dP/dT Studies.

Interpretation of hardness-conductivity data is possible if the thermal history of the item in question is known. One must always consider the effects of retrogression and varying solution heat treat temperatures. However, if one does not know the thermal history, then difficulties of interpretation develop. Notice that in many instances a given small range of hardness and conductivity could represent a sample having one of several different thermal histories. Each of the thermal histories of each sample in the rectangle on Fig. 5, which is 1 &I.A.C.S. units wide and 6 R_B units tall, could impart to the sample different mechanical properties, different corrosion resistances, or some other important engineering property. Also, one could superimpose the effects of a quench delay schematically shown in Fig. 2 on other samples to arrive at the indicated range within the rectangle. Therefore, it would be desirable to find some nondestructive means of further identifying the prior heat treatment(s).

As discussed in the Review of Literature, a study of dP/dTrelationships may provide a means of separation of the samples described above. Even if a separation is not possible, then Hansen's equation, if applicable, would provide the basis for developing a technique of conductivity testing at any outdoor temperature instead of at room temperature as is now required by many specifications, e.g., (9).

Certain samples representing several different conductivity ranges were selected for $d\rho/dT$ studies. Each range of conductivity was comprised of about five to eight samples with a total conductivity spread of about 1 %I.A.C.S. The ranges selected were around 31, 35 and 38 %I.A.C.S. Several other samples with different conductivities were also evaluated. Conductivity vs temperature data for the samples tested is listed in Table IV and was obtained in the following manner using the experimental apparatus shown in Illustration 2.

The 1 x 2 inch samples were affixed to a wooden block. A 28 gauge copper-constantan thermocouple was placed between the sample and the wooden block. The block was then immersed in a dewar containing dry ice and methyl alcohol until the temperature of the sample was -80° F. or below. Approximately five minutes were required to achieve -80° F. The block and samples were then removed from the dewar and allowed to heat to room temperature.

The temperature was monitored by using a Rubicon portable potentiometer. The temperature compensator contained in the potentiometer was used instead of an ice water bath for the reference junction. It was previously determined that the temperature error would be less than 2°F. when using the compensator. This was done by comparing the temperature of boiling distilled water using both the temperature compensator and an ice water reference junction. The temperature of the boiling distilled water was as shown below.

Reference Junction	Millivolts	Indicated Temperature
32 ⁰ F ice water	4.26	211.3°F.
75 ⁰ F Temperature Compensator	4.30	212.8°F.

As the sample warmed, a conductivity reading was taken in 10° F. increments by simply placing the probe of the conductivity meter onto the sample. The clamp acted as a heat sink which allowed the sample



ILLUSTRATION 2

d P/dt Apparatus

to warm slowly. The meter was balanced and the conductivity was read directly in %I.A.C.S.

There are important considerations with regard to precision when using this technique. The meter probe induces eddy currents into the sample. This, of course, produces resistance heating which changes the temperature of the sample as the conductivity measurement is taken. This temperature error amounts to about 5° F. for readings taken below 0° F. Readings taken above 0° F. were less affected. Since the meter detects impedance changes of the probe, which are related to the conductivity of the material being tested, another problem arises. If the probe changes temperature due to its contact with the cold sample, then its own impedance may change due to the cooling effect. If a thermal gradient existed in the sample, the actual temperature and measured temperature may not agree giving rise to small conductivity errors, particularly at lower temperatures.

Therefore, to minimize these errors, the following technique was used. Several samples were tested to obtain a "feel" for the approximate conductivities which would be encountered at each temperature. Then during the actual runs, the meter dial was set to the anticipated conductivity value for each sample at each temperature so that the length of time needed to balance the bridge was thus held to a minimum, less than one second, thus reducing the heating effects upon the sample and the cooling effects upon the probe. Also, the thermocouple was placed under the area of probe contact to minimize thermal gradient errors.

It was readily apparent after plotting the dA/dT curves that one

of the above effects did influence the data in most instances, particularly below 0^OF. The curves show slightly higher conductivities at low temperatures than what is consistent with data taken at higher temperatures. Some curves do not show these anomalies.

Since the deviations produce higher conductivities, the resistance heating effect of the induced eddy currents can be discounted as the cause since this would produce lower conductivity contributions. Therefore, it was deduced that either the probe cooling effect or thermal gradients caused the deviations.

In a separate experiment, the meter was standardized on a 61 #I.A.C.S. standard. The probe was cooled by placing it on a chilled brass block and the conductivity of the 61# standard was remeasured as 60 #I.A.C.S.; therefore, indicating that the probe cooling effect would produce a decreased conductivity error. The probe cooling effect was discounted as the cause for the deviations leaving small thermal gradients as the suspected cause. This effect was neglible at temperatures above $0^{\circ}F$. Therefore, the practical lower temperature limit of the apparatus appeared to be about $0^{\circ}F$.

As the data in Table IV was plotted, it became apparent that the samples with very similar room temperature conductivities had very similar $d\rho/dT$ curves so that no further interpretation, i.e., separation of similar samples, could be accomplished. Fig. 7 shows typical examples of the $d\rho/dT$ curves.

The linearity of the $d\rho/dT$ curves indicates that over the temperature range considered, Matthiessen's rule applies. If very precise instrumentation could be used to effect a separation of samples having



the same or nearly the same conductivities, the separation most likely would be based upon deviations from Matthiessen's rule. The residual resistivity in Matthiessen's rule can be considered to be the summation of all conditions producing the residual resistivity. The two major resistivity contributions are those caused by the matrix solid solution and those caused by the coherency stresses. If they are designated X_1 and Y_1 for sample 1 and X_2 and Y_2 for sample 2, with each having the same total residual resistivity, then

$$X_1 + Y_1 = X_2 + Y_2$$

This does not mean that $X_1 = X_2$ nor that $Y_1 = Y_2$, but only that their sums are equal. This could be the reason why two samples with different heat treatments can develop the same conductivity at a given temperature. This hypothesis would also explain why samples with identical conductivities can have different hardnesses and, very likely, different mechanical properties. Therefore, any separation based mostly on conductivity measurements would depend upon the relative temperature dependence of X and Y in each sample as precisely measured over a larger temperature range.

The dP/dT curves indicate that point "M" in Starr's graph, (Fig. 3), is well above room temperature. That point "M" is well above room temperature is better illustrated in Fig. 7 which shows the same typical curves as Figs. 13, 14 and 15, which are contained in the appendix. Fig. 7 shows that the curves have essentially equivalent slopes and, therefore, would not converge unless extrapolated well above room temperature.

The data for several typical samples shown in Fig. 7 were

analyzed to determine whether Hansen's equation fits the data. Table V. shows, in tabular form, the necessary calculations as developed by Starr, to plot Fig. 8. The linear coefficient of expansion was taken as an average value of 23.4 x 10^{-6} cm/cm/°C. It is also assumed that df/dT is equivalent to $\Delta f'/\Delta T$. Fig. 8 shows that the conductivity vs temperature coefficient of resistance follows Hansen's equation.

The total equation for the 7178 alloy system is shown below:

$$K = B \propto + C$$

 $K = 0.00621 \propto + 0.0819$

when K is given in reciprocal microhm-cm and \propto is given $\Omega/\Omega/^{\circ}C. \times 10^{4}$.

For K given in reciprocal Ω -cm and α given in $\Omega / \Omega / C$, then B = 0.00621 x 10¹⁰ and C = 0.0819 x 10⁶ or K = 0.00621 x 10¹⁰ α + 0.0819 x 10⁶.

Using the above equations or Fig. 8, one only has to measure the conductivity at any known temperature, calculate or find its corresponding α , and correct the conductivity to its room temperature value. The corrected value thus determined can then be used for conductivity-hardness information. Three samples, TA3, D3 and E4 were used to test the equation. The conductivities as measured at 20°F., taken from Table IV., were used as the initial ambient values. The results are listed below:

Sample Code #	K (20 ⁰ F.) %I.A.C.S.	≪ ∿/∿°Cx10+ ⁴	K (75 ⁰ F.) %I.A.C.S. Calculated	K (75 ⁰ F.) %I.A.C.S. Measured	Error %I.A.C.S.
TA 3	32.6	17.7	30.9	31.0	0.1
D 3	37.5	22.1	35.2	35.2	0.1
E 4	40.3	25.0	37.3	37.9	0.6





Note that the errors are 0.1, 0 and 0.6 %I.A.C.S. for samples T3, D3 and E4, respectively. These errors are within the tolerances of the conductivity meter itself.

E. 5% Zn-Al Heat Treatments.

It was attempted to obtain similar data for the 5% Zn-Al binary alloy, but unfortunately the alloy was too low in Zn content to achieve significant hardening, even though the Zn content was approximately the same per cent by weight as the Zn content of 7075 alloy. The 7000 series alloys, however, contain Cu and Mg. These atoms form intermetallic compounds such as MgZn₂ which can be coherent phases. These phases evidently are needed to develop the high coherency stresses to produce the marked change of hardness exhibited in these alloys.

However, $d\rho/dT$ curves were obtained to provide some limited data. The results of the $d\rho/dT$ tests are shown on Table VI. The procedure for the conductivity changes with temperature was similar to those used for the 7178 alloy. The binary alloy samples were annealed for six hours at 300°C. and furnace cooled to promote homogeneity in the samples. The samples were then solution heat treated at 250°C. for one hour before water quenching. The solvus for the 5% by weight Zn-Al binary alloy is approximately 125°C. Herman, Cohen and Fine (22) in their work with a 5.3 at% Zn-Al binary alloy used solution heat treating temperatures of both 250°C. and 300°C. for a length of time of one hour. After quenching, the samples were affixed to the wooden block for subsequent $d\rho/dT$ measurements after aging for the times indicated in Table VI.

The $d\rho/dT$ curves for the binary alloy samples formed a band. Each individual sample's $d\rho/dT$ curve could not be separated from its closest neighboring curve; therefore, an average curve is depicted on Fig. 7 and averaged data is used on Table V to produce the indicated point on Fig. 8. Samples with higher Zn content would have to be used to develop data similar to that developed for the 7178 alloy samples. That similar data might be developed is based on the fact that the dP/dT curves are linear over the range tested which indicates that Matthiessen's rule applies; thus, indicating that Hansen's equation might apply.

IV. CONCLUSIONS

The literature survey has shown that modern wave-mechanical electron conduction theory provides a basis for the interpretation of conductivity changes caused by chemical changes in the composition of the matrix, which takes place during the heat treatment of aluminum alloys. Rummel's theory for conductivity-hardness interpretation is adequate for most practical situations. However, one must consider the possible occurrence of retrogression effects upon hardness-conductivity relationships and the effects of different solution heat treating temperatures in conjunction with Rummel's theory for accurate, logical interpretation of hardness-conductivity data.

The conductivity behavior of the 7178 alloy system can be described by Matthiessen's rule and Hansen's empirical equation,

$K = B\alpha + C$

A practical result is that the conductivity of any 7178 sample can be measured at any known temperature and then corrected to its room temperature value for interpretation of the sample's metallurgical condition. The inverse calculation could also be performed. Inverse calculations could be used to construct a table giving the conductivity ranges corresponding to the standard tempers of the 7178 alloy at any desired temperature.

Point "M" which is the area of convergence shown on Fig. 3 seems to be well above room temperature. This suggests that the resistivity due to clustering is not close to its theoretical value. However, this may also mean that the clustering induced resistivity is only a

very small portion of the total observed resistivity. One would have to be able to separate clustering resistivity effects from the matrix resistivity effects to truly determine the temperature value of point "M" with respect to room temperature.

No effective separation of samples having similar room temperature conductivities but different thermal histories could be achieved through $d\beta/dT$ studies. Very precise conductivity measurements over a larger temperature range may produce $d\beta/dT$ curves which show deviations from Matthiessen's rule, thus effecting the samples separation. The deviations would depend upon the relative temperature dependence of the two major residual resistivity components, i.e., the clusteringinduced resistivity and the matrix resistivity. However, more precise instrumentation than what was available might also separate samples having similar room temperature conductivities which appear to be similar due to the inability of the conductivity meter to distinguish slight conductivity differences.

It would be interesting if other workers could use the concepts described in this thesis to evaluate other aluminum alloys, or other nonaluminum base precipitation hardenable alloys. The concepts of conductivity testing in general could very well be extended to any coherent second phase which is developed in a parent lattice. Suitable allowances would have to be made for the various kinetic effects of various second phase reactions, e.g., tempering would be a factor in a transformation hardening alloy system.

Some work has been accomplished in the above area. Conductivity measurements have been used to follow the omega phase reaction in an

8% Mn-Ti alloy, (32). In this particular application, conductivity measurements separated samples having too much omega phase from those with little or none present. The omega phase severely embrittles the alloy if it is present in quantities above a certain threshold amount.

Other related work, (32), has shown that conductivity testing concepts, utilizing commercially available eddy current conductivity meters, did not produce usable results for other titanium alloys. All of the other titanium alloys tested had phases which were saturated and/or supersaturated solid solutions at all times, no matter what the heat treat condition. Therefore, it is speculated that any resistivity changes caused by the second phase's coherency stresses are masked by the essentially constant matrix resistivities. No real effort has been made to date to define the necessary and sufficient conditions needed for conductivity testing in general; therefore, this area would constitute another interesting subject for someone to explore.

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<u>APPENDIX</u>
















	TAE	BLE I	
AGING TIME, CO 7178 Alloy (Code #)	ONDUCTIVITY & HA Aging Time (m-min.) (h-hours) (d-days)	RDNESS FOR DIRECT Conductivity (% I.A.C.S.)	AGED SAMPLES Hardness (R _B)
Direct Age at	450°F.		
D 11 D 1 D 2 D 3 D 4 D 5 D 6 D 7 D 8 D 9 D 10	0 1.0 m 3.0 m 5.0 m 7.0 m 10.0 m 15.0 m 20.0 m 25.0 m 30.0 m	26.8 29.0 32.2 35.2 36.8 37.8 38.7 39.3 39.6 40.2 41.6	82.0 74.5 84.5 86.0 86.0 83.0 81.0 79.5 77.5 76.5 68.5
Direct Age at	400°F.		
E 11 E 8 E 7 E 9 E 10 E 2 E 2 E 5 6	0 2.5 m 5.0 m 7.5 m 10.0 m 12.5 m 20.0 m 30.0 m 40.0 m 50.0 m	27.2 31.4 33.6 33.7 35.2 35.8 36.9 37.5 37.9 38.6 39.0	80.5 82.0 85.0 87.0 88.0 88.0 87.5 88.0 86.0 85.0 85.0 83.0
Direct Age at	350°F.		
C 11 C 7 C 5 C 1 C 2 C 3 C 4 C 8 C 9 C 6 C 10	0 2.5 m 5.0 m 15.0 m 30.0 m 48.0 m 1.0 h 1.5 h 2.0 h 3.0 h	27.0 26.9 29.6 31.0 32.8 33.3 33.8 34.5 35.6 35.5 37.0	81.5 81.0 75.0 83.0 88.0 89.5 90.0 88.0 85.0 88.0

	TAB	LEI	
AGING TIME, C 7178 Alloy (Code #)	ONDUCTIVITY & HAD Aging Time (m-min.) (h-hours) (d-days)	RDNESS FOR DIRECT Conductivity (% I.A.C.S.)	AGED SAMPLES Hardness (R _B)
Direct Age at	300 ⁰ f.		
B 11 B 9 B 10 B 1 B 2 B 3 B 4 B 5 B 6 B 7 B 8	0 5.0 m 7.5 m 10.0 m 2.0 h 8.0 h 16.0 h 24.0 h 32.0 h 40.0 h 53.0 h	26.9 29.7 29.9 31.5 33.0 33.9 34.8 35.2 36.4 37.1	81.0 78.0 77.0 82.5 91.0 93.0 91.5 92.0 90.5 90.5
Direct Age at	250°F. (24 Ho	urs Produces T6 T	emper)
A 7 A 8 A 9 A 10 A 11 A 12 A 1 A 2 A 1 A 2 A 3 A 4 A 5 A 6	$\begin{array}{c} 15.0 \text{ m} \\ 30.0 \text{ m} \\ 60.0 \text{ m} \\ 2.0 \text{ h} \\ 3.5 \text{ h} \\ 5.5 \text{ h} \\ 24.0 \text{ h} \\ 2.0 \text{ d} \\ 3.0 \text{ d} \\ 4.0 \text{ d} \\ 5.0 \text{ d} \\ 6.0 \text{ d} \end{array}$	28.6 28.9 29.3 29.5 29.8 30.1 31.1 31.5 31.9 32.1 32.4 32.5	84.0 85.0 87.0 89.5 91.0 92.0 93.0 93.5 96.0 94.0 93.0 93.0
Direct Age at	140-165 ⁰ F.		
A ll	13.0 d plus 1.0 h	29.1	93.0

All times in Table I are measured from insertion into the aging oven. A "O" time indicates at least one week of room temperature aging at $75^{\circ}F$.

TABLE II

AGING	TIME,	CONDUCT	IVITY	& ł	HARDNESS	FOR	ROOM	TEMPERATURE	AGED	SAMPLES
	7178 A (Code	lloy #)	Agin (m- (h- (d-	ng ' -mi) -hoi -daj	Time n.) urs) ys)	Co (%	nduct	ivity H C.S.)	lardne (R _B)	SS
	Direct	Age at	75°F.		(Room Te	mper	ature)		
	R.T	•	3(0.0	m		31.	2	43.5	
	R.T		60	0.0	n		30.	7	49.0	
	R.T		2	2.0	h		29.	3	58.0	
	R.T		2	2.5	h		29.	Ō	59.0	
	R.T			3.0	h		28.	6	63.0	
	R.T		Ī		h		28.	1	64.5	
	R.T	•		5.3	h		27.	8	66.5	
	Я. Т	-	ē	5.0	h		27.	6	66.5	
	R.T		21	+.0	h		26.	9	75.0	
	R.T	•		7	d		26.	9	80.0	

All times in Table II are measured from the time of water quenching.

AGI	NG TIME, CC 7178 Allo (Code #)	TABLE DNDUCTIVITY & HARDNESS y Aging Time (m-min.) (h-hours) (d-days)	III FOR SAMPLES AGED Conductivity (% I.A.C.S.)	FROM T6 TEMPER Hardness (R _B)
	Re-Age at	450°F. from T6 Temper	`•	
	TC 1 TC 2 TC 11 TC 3 TC 10 TC 4 TC 4 TC 5 TC 6 TC 7 TC 8 TC 9	1.0 m 2.5 m 3.5 m 5.0 m 7.5 m 10.0 m 15.0 m 20.0 m 25.0 m 30.0 m	30.5 30.9 32.8 34.8 37.5 38.9 39.5 39.9 40.4 40.8 41.7	93.0 90.5 90.0 90.0 86.0 81.0 79.0 77.5 70.5 73.0 66.0
	Re-Age at	400°F. from T6 Temper	•	
	TB 1 TB 2 TB 9 TB 10 TB 3 TB 4 TB 5 TB 6 TB 7 TB 8	2.0 m 5.0 m 7.5 m 8.5 m 10.0 m 20.0 m 30.0 m 60.0 m 1.5 h 2.1 h	30.6 31.6 33.9 34.2 35.0 36.5 37.0 38.5 39.7 40.1	92.0 89.5 91.5 91.5 92.0 90.5 89.5 86.0 83.0 80.5
	Re-Age at	350°F. from T6 Temper	` #	
	TA 1 TA 2 TA 3 TA 4 TA 5 TA 6 TA 7 TA 6 TA 7 TA 8 TA 9 TA 10 TA 11	1.0 m 2.5 m 5.0 m 10.0 m 21.0 m 38.0 m 62.0 m 1.5 h 2.0 h 3.0 h 5.0 h	30.6 30.4 31.0 30.9 31.4 32.3 33.5 34.1 35.2 36.5 37.5	92.0 92.5 91.0 91.0 92.0 92.0 91.0 93.5 91.5 91.0 89.0
	T 6	Average Values	30.5	93.0

* This sample had a low hardness before re-aging.

d 9/dT DAT.	A FOR 717	8 ALLOY	SAMPLES	5	
Temperature °F.	Conduc (% I.A	tivity .C.S.)		Condu (% I.	ctivity A.C.S.)
	Run 1	Run 2		Run 1	Run 2
Code #	D	3		D	5
-60 -50 -40 -30 -20 -10 0 10 20 32 40 50 60 75	40.9 40.4 39.9 39.6 39.3 38.7 38.3 38.0 37.5 37.0 36.6 36.3 35.8 35.8	40.9 40.6 40.1 39.7 39.4 39.0 38.4 38.0 37.5 37.0 36.6 36.4 35.8		45.6 45.1 44.0 43.0 42.7 42.0 41.5 41.1 40.6 40.0 39.6 39.0 38.6 37	45.7 44.6 43.0 42.7 42.0 41.4 41.0 40.5 39.9 39.5 38.9 38.4 .8
	E	3		E	4
-60 -50 -40 -30 -20 -10 0 10 20 32 40 50 60 75	45.1 43.5 43.0 42.4 41.5 41.0 40.6 40.0 39.4 39.0 38.5 38.1 37.	45.0 44.1 42.2 41.9 41.4 40.5 39.8 39.0 38.1 5		45.7 44.1 43.4 42.6 41.9 41.5 41.0 39.8 39.4 38.9 38.4 38.9 38.4 37	46.1 45.0 44.3 43.3 42.6 42.0 41.4 40.8 40.4 39.7 39.4 38.9 38.4 .9

TABLE	IV

	DATA POIL /			
Temperature °F.	Conduc (% I.A	tivity .C.S.)	Conduc (% I.A	tivity .C.S.)
	Run 1	Run 2	Run 1	Run 2
Code #	E	8	C	1
-60 -50 -40 -30 -20 -10 0 10 20 32 40 50 60 75	34.5 34.0 33.6 33.2 33.0 32.5 32.3 31.9 31.6 31.3 31.0 30.7 31.0	34.6 34.2 33.7 33.6 33.2 32.8 32.5 32.1 31.8 31.5 31.3 31.0 30.7	34.7 34.4 34.0 33.7 32.9 32.6 32.3 32.0 31.7 31.4 31.0 30.8 31.0	35.0 34.3 34.2 33.8 33.2 32.9 32.7 32.4 32.0 31.8 31.3 31.1 30.7
	C	10	В	1
-60 -50 -40 -30 -20 -10 0 10 20 32 .40 50 60 75	43.5 42.6 42.2 41.8 41.2 40.5 40.1 39.7 39.0 38.6 38.1 37.8 37.4 37.4	43.7 42.7 42.2 41.7 41.0 40.5 40.1 39.7 39.1 38.6 38.3 37.9 37.5	32.8 32.3 32.0 31.8 31.5 31.3 30.9 30.6 30.2 30.1 29.8 29.5 29.8	32.8 32.2 31.9 31.7 31.4 31.2 31.0 30.3 30.1 29.9 29.5

TABLE IV de/dt data for 7178 Alloy Samples

d e /dT Temperature °F.	DATA FOR 71 Conduct (% I.A	78 ALLOY tivity .C.S.)	SAMPLES Condu (% I.	A.C.S.)
	Run 1	Run 2	Run 1	Run 2
Code #	В	2	E	3 5
-60 -50 -40 -30 -20 -10 0 10 20 32 40 50 60 75	36.4 35.9 35.0 34.6 34.2 33.9 33.5 33.1 32.8 32.5 32.2 31.9 31.9	36.3 35.8 35.3 34.8 34.6 34.3 33.5 33.1 32.8 32.5 32.1 31.9 5	40.0 39.8 39.3 38.6 38.4 37.8 37.5 37.1 36.7 36.4 36.0 35.4 35.4	40.4 39.7 39.1 38.7 38.4 37.6 37.1 36.5 36.2 35.9 35.5
	В	6	В	8
-60 -50 -40 -30 -20 -10 0 10 20 32 40 50 60 75	41.5 40.5 40.0 39.5 38.5 38.0 37.6 37.2 37.0 36.5 36.2 35.2	41.2 40.4 39.5 39.0 38.6 38.3 37.8 37.4 37.0 36.5 36.2	44.2 43.2 42.6 42.0 41.6 41.1 40.5 40.0 39.5 39.1 38.5 38.0 37.6 37	44.1 43.2 42.7 42.0 41.5 41.1 40.6 40.0 39.6 39.1 38.6 38.1 37.7

TABLE TV

d e/dT Temperature °F.	TABLE DATA FOR 71 Conduc (% I.A	IV 78 ALLOY S tivity .C.S.)	Conduc (% I.A	IPLES Conductivity (% I.A.C.S.)		
	Run 1	Run 2	Run 1	Run 2		
Code #	Anne	aled	B	11		
-60 -50 -40 -30 -20 -10 0 10 20 32 40 50 60 75	59.0 56.8 54.5 53.5 52.5 51.0 50.2 49.6 48.5 47.5 46.	59.0 56.2 54.0 53.4 52.4 51.1 49.9 49.2 48.5 47.5	30.0 29.6 29.2 28.9 28.7 28.5 28.2 28.0 27.7 27.5 27.3 26.5	30.1 29.8 29.5 29.2 28.9 28.6 28.5 28.2 27.9 27.7 27.5 27.3		
	TC	2	А	1		
-60 -50 -40 -30 -20 -10 0 10 20 32 40 50 60 75	35.0 34.8 34.1 34.1 33.5 33.3 33.0 32.5 32.3 32.0 31.7 31.3 30.1	35.1 34.7 34.5 34.0 33.5 32.6 32.6 32.6 32.6 32.5 31.3 9	35.6 34.7 34.5 34.0 33.4 33.2 32.9 32.6 32.2 32.0 31.5 31.3 31.3	35.5 35.0 34.7 34.2 34.0 33.5 33.1 32.9 32.5 32.2 32.0 31.6 31.2 1		

d P/dI Temperature F.	TABLE DATA FOR 71 Conduc (% I.A	IV 78 ALLOY S tivity .C.S.)	SAMPLES Conductivity (% I.A.C.S.)		
	Run 1	Run 2	Run 1	Run 2	
Code #	TC	10	TB	2	
-60 -50 -40 -30 -20 -10 0 10 20 32 40 50 60 75	45.1 44.4 43.9 43.2 42.5 41.8 41.4 40.7 40.1 39.7 39.3 38.8 38.3 37.	45.6 44.6 43.5 42.7 42.3 41.9 41.5 40.4 39.8 39.3 39.0 38.5	36.4 35.6 35.3 35.0 34.6 34.2 33.8 33.5 33.1 32.8 32.5 32.2 31.9 31.	35.7 35.2 35.0 34.5 34.2 33.8 33.5 33.2 32.7 32.5 32.1 32.0	
	TB	5	TA	3	
-60 -50 -40 -30 -20 -10 0 10 20 32 40 50 60 75	44.4 43.4 43.0 42.2 41.6 41.0 40.5 40.0 39.4 39.0 38.5 38.0 37.6 37.6	44.4 43.6 42.6 42.1 41.5 40.9 40.3 40.0 39.5 39.1 38.6 38.1 37.7	35.3 35.0 34.6 34.4 33.6 33.3 33.0 32.7 32.2 31.9 31.7 31.4 31.	35.7 35.1 34.8 34.4 34.0 33.2 33.0 32.6 32.2 31.9 31.7 31.3	

de/dT Temperature	TABLE DATA FOR 7 Conduc	IV 278 ALLOY tivity	SAMPLES Conduct	ivity	
°F.	(% I.A	c.s.)	(% I.A.	.C.S.)	
	Run 1	Run 2	Run 1	Run 2	
Code #	TA	5	TA	11	
-60	36.6	36.4	45.4		
-50	35.6	35.8	44.0	44.0	
-40	35.5	35.6	43.4	43.5	
-30	35.2	35.0	42.6	43.0	
-20	34.6	34.6	42.0		
-10	34.4	34.3	41.6	41.8	
0	33.9	34.0	41.0	41.3	
1.0	33.5	33.6	40.5	40.7	
20	33.3	33.2	40.1	40.2	
32	33.0	32.9	39.7	39.6	
40	32.7	32.5	39•5	39.3	
50	32.3	32.2	39.0	39.0	
60	32.0	32.0	38.6	38.4	
75	31.	4	37.5	5	• •

TABLE V

ELECTRICAL PROPERTIES AT 75°F OF SAMPLES INDICATED

Code #	К	P	К	<u>dP</u> dT	K d P	\prec
	% I.A.C.5.	p_ncm	$\frac{1}{\rho - cm}$	h-vcm	~~ /~/ °C	r/r/°C
B 11	26.9	6.41	0.156	0.00965	.001505	.001482
B l	29.8	5.78	0.173	0.00673	.001164	.001141
TC 2	30.9	5.57	0,180	0.00878	.001580	.001.557
TA 5	31.4	5.49	0.182	0.01044	.001900	.001877
B 2	31.5	5.47	0.183	0.00900	.001647	.001624
в б	35.2	4.90	0.204	0.01020	.002081	.002058
D 5	37.8	4.56	0.219	0.00965	.002113	.002090
ANN	46.4	3.72	0.269	0.01081	.002908	.002885
5% Zn-A	1 55.5	3.11	0.322	0.00965	.003107	.003184

 $\frac{d\rho}{dT} = \frac{\Delta \rho}{\Delta T} \qquad \alpha = K \frac{d\rho}{dT} = a a = 23.4 \times 10^{-6} \text{ cm/cm/°C}$

TABLE VI d?/dT DATA FOR 5% Zn-Al Alloy AGED AT ROOM TEMPERATURE

Temperature ^o F.	Conductivity (% I.A.C.S.)	Conductivity (% I.A.C.S.)
Aging Time (Minutes)	l	2
Code #	КІ	КЭ
-60 -50 -40 -30 -20 -10 0 10 20 32 40 50 60 75	73.1 70.7 69.8 68.1 67.0 65.3 64.0 62.8 61.5 60.1 59.0 58.2 56.8 55.5	72.4 72.4 69.5 68.0 67.0 65.1 64.0 63.1 61.1 59.5 58.9 57.8 56.4 55.3
Aging Time (Minutes)	5	10
	K 5	К 9
-60 -50 -40 -30 -20 -10 0 10 20 32 40 50 60 75	73.0 72.0 70.1 68.5 67.2 65.9 62.9 61.0 60.2 59.3 57.8 56.1	73.5 72.0 71.5 69.6 66.1 65.0 63.5 62.6 61.0 60.0 58.8 57.5 56.0

TABLE VI

dC/dT DATA FOR 5% Zn-Al Alloy AGED AT ROOM TEMPERATURE

Temperature ^O F.	Conductivity (% I.A.C.S.)	Conductivity (% I.A.C.S.)	Conductivity (% I.A.C.S.)
Aging Time (Minutes)	15	25	60
Code #	K 11	К 7	к 13
-60	74.5	74.3	
- 50	73.1	71.3	72.0
-40	70.5	70.1	69.6
-30	68.9	68.1	67.4
-20	67.3	68.2	66.5
-10	66.5	65.9	65.2
0	64.9	64.1	63.6
10	63.6	63.0	62.5
20	62.6	61.4	61.4
32	60.9	60.1	59.9
40	60.0	58.8	58.8
50	58.8	57.7	57.5
60	57.6	56.6	57.0
75	56.0	55.4	55.2

The author was born on October 9, 1939, in St. Louis, Missouri, where he received his primary and secondary education. He received a Bachelor of Science Degree in Metallurgical Engineering-Nuclear Option from the University of Missouri School of Mines and Metallurgy in June, 1962.

He has been enrolled in the part time Graduate School of the University of Missouri at Rolla - St. Louis Graduate Extension Center since its inception in September, 1964. He previously was enrolled in the Graduate School of Washington University, St. Louis, Missouri.

He is presently employed by the McDonnell Company, St. Louis, Missouri.

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VITA